PATENT SPECIFICATION

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COMPLETE SPECIFICATION

Process for the removal of Wax Contaminants from Finely Divided Catalysts Used in Hydrocarbon Synthesis Processes

We, STANDARD OIL DEVELOPMENT COMPANY, a corporation duly organised and existing under the laws of the State of Delaware, United States of America, having an office at Linden, New Jersey, United States of America, do hereby declare the nature of this invention, and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

The present invention relates to the removal of soluble contaminants from finely divided solids.

The main object of the invention is to provide a method and suitable apparatus therefor for washing the wax deposit from the catalyst employed in a hydrocarbon synthesis.

The improvements relate to the so-called "fluid-solids" technique wherein during the synthesis of the hydrocarbons from CO and hydrogen, the catalyst which is in the form of powder, is maintained in a dense suspension in the reaction zone intermixed with the reactants. This is accomplished by causing the reactants to flow upwardly in the reaction zone at a velocity which is sufficient to maintain the catalyst in a turbulent condition within the dense suspension previously referred to. In this type of operation the products obtained in the synthesis together with any unreacted materials and a small amount of entrained catalyst issue from a point at the top of the reactor after having passed through several centrifugal separators or "cyclones" which serve to separate most of the catalyst from the gasiform material by centrifugal action. The catalyst separated by the cyclones is returned to the reaction zone.

In the course of the synthesis within the dense suspension referred to in the above, hydrocarbons of a wide range of molecular weight may be formed. The molecular weight varies from that of methane to high boiling materials some of which, at room temperature, exist in the solid form and hence are commonly known as waxes. The operating conditions of temperature and pressure in the reaction zone set the amount of the heavier hydrocarbons which may be removed as gas with the other gasiform materials leaving the reactor. Under certain conditions of operation the rate of production of the heavier hydrocarbons exceeds the rate of removal with the materials leaving the reactor. Consequently, condensation occurs and these heavier hydrocarbons are then contained on the catalyst in the form of liquids. If the quantity of liquid contained on the catalyst is allowed to build up, a condition is reached at which the catalyst particles begin to stick together, thereby losing true fluid characteristics.

Previous reference has been made to the fact that a small amount of entrained catalyst leaves the reactor with the gasiform materials after passing through cyclones for separating the main portion of catalyst from the gas. The entrained catalyst may also contain liquid materials if such material has been deposited on the catalyst within the reactor. The amount of catalyst plus contained liquid removed in this fashion may still be insufficient to prevent further accumulations of liquid on catalyst within the reactor without reaching the "sticking," non fluid condition, referred to above. Hence it is necessary to provide for direct removal of catalyst from the reactor, with subsequent removal of the heavy liquids or waxes contained thereon. This is termed "dewaxing of the catalyst," and is followed by re-introduction of the dewaxed catalyst to the reactor.

It has already been proposed to dewax catalyst in a process of this type by washing it in an extraction zone with a suitable solvent, the dewaxed catalyst then being returned to the reaction zone. The process of the present invention provides a continuous method for achieving the above result.

The present invention comprises a process for the removal of soluble contaminants from finely divided catalysts, which comprises passing the catalyst material in the form of a slurry, in countercurrent flow to a stream of solvent for the contaminants, such a solvent being also used for slurrying the catalyst; this procedure may be carried out continuously as part of a cyclic reaction system.

In the accompanying drawings, in Figure I, there is shown diagrammatically a single stage countercurrent washing system; in Figure II, a two-stage countercurrent washing process; in Figure III, in enlarged view a catalyst-solvent mixer and washer, and in Figure IV, is shown a fragmentary view of a tube in enlarged scale.

Referring to Figure I, the synthetic products and unreacted materials from the reaction zone, containing entrained catalyst, enter the washing system through line (1) and first pass into a scrubbing zone (2) where they are treated with a high boiling hydrocarbon oil admitted through line (3) after having been heated to the desired temperature by the heater (4). The hydrocarbon oil introduced by (3) passes countercurrently downward against the upflowing gases and washes the entrained catalyst, collecting as a slurry in the bottom of scrubber (2) and the gaseous reaction products then pass overhead through line (5) to a condensation and recovery system not shown. The hydrocarbon oil used for washing entrained catalyst in the above system will consist of the heavier fractions of the synthetic product and consequently very little vaporization of this oil will take place. The operation will be conducted at a temperature and pressure approximately the same as those existing in the reactor. Instead of using heater (4) the heavy oil obtained as bottoms may be used (with only a moderate amount of cooling) from the solvent-oil fractionation system (not shown) which supplies the solvent used in the washing system. The amount of heavy oil entering (2) by way of line (3) will be adjusted so as to provide a catalyst-oil slurry in the bottom of (2) containing no more than 4 lbs. of catalyst per gallon of slurry. The preferred operation involves the use of enough oil to provide a slurry containing 1—4 lbs. per gallon.

The slurry of oil and catalyst is withdrawn from the bottom of the scrubber (2) via line (6) the rate of removal being controlled by means of valve (7) responsive to a liquid level device (8) which serves to maintain a desired level of slurry (L) at all times within the scrubber (2). Excess slurry may be withdrawn, if necessary, through line 6A. The slurry then passes through line (9) to a mixer (10). The mixer may be considered to be merely a mechanically agitated vessel.

The main portion of catalyst to be dewaxed is removed from the reactor (not shown) passing through line (11) and control valve (12) to the scrubber (14) by means of line (13). Gases occluded with the catalyst taken from the reactor together with any extraneous gas used to effect the transfer from the reactor to the scrubber (14) are released from the catalyst and pass up through the scrubber, out through line (17) to the flow controller (18) thence by way of line (19) to the pressure controller (20) and finally by line (21) to a recovery system (not shown) which may be the same as that used for the main gas stream leaving through line (5). The pressure controller (20) controls the pressure of the wash system at a value somewhat lower than the reactor system pressure so that transfer of catalyst and/or catalyst and slurry to the wash system from the reactor system may be readily effected. Additional inert gas may be introduced, if desired, through line 19A. The flow controller (18) operates valve (12) which adjusts the rate of removal of catalyst from the reactor. The gas passing up through scrubber (14) is scrubbed with a metered amount of fresh solvent which enters the system through lines (22) and (23) is metered and controlled by the flow control meter (24) and valve (25) and passes through line (26) to heater (16) wherein it is raised to the desired operating temperature and then passes to scrubber (14) by way of line (15). The solvent used in the system and shown entering through line (22) will be a portion of the synthetic product boiling within the range of about 250° to 450° F. In passing through the heater (16) it will be heated to 150°—300° F. preferably in the range of 200°—250° F. The quantity will be adjusted to provide a solvent-catalyst slurry in the bottom of scrubber (14) which contains no more than 4 lbs. of catalyst per gallon of slurry and preferably between 1 and 4 lbs per gallon.

The solvent-catalyst slurry collects in the bottom of scrubber (14) and a level of the material is maintained by the level device (27) which controls valve (28) thereby permitting the thoroughly mixed catalyst-oil-solvent slurry maintained in suspension in mixer (10) to flow by way of line (29) valve (28) and line (30) into the countercurrent washer (31). Slurry from scrubber (14) flows through mixer (10) by way of line (32) as demanded by the operation of the level control device (27) and the control valve (28).

The main portion of the solvent enters the bottom of the washer (31) through lines (22) and (33), is controlled in quantity by flow controller (34) which operates valve (35), is heated to 150° F.—300° F. preferably 200° F.—250° F. in heater (36) and enters the washer (31) through line (37). Sufficient solvent is introduced through line (37) to not only provide a slowly moving stream moving upward in washer (31) against the downward flowing catalyst which has been introduced as a slurry through line (30) so as to achieve countercurrent washing of the catalyst but also to provide a pumpable catalyst-solvent slurry containing between 1/2 and 3 lbs. of catalyst per gallon which collects in the bottom of washer (31) and is removed through lines (38) and then to pump (39) from which it passes to a solvent vaporizer not shown. Following the vaporizer a " cyclone " is used to separate solvent vapor from catalyst and the catalyst allowed to pass into an aerated standpipe from which it is returned to the reactor by means of a stream of synthesis gas. Instead of using a solvent vaporizer a filter may be used to remove solvent from the catalyst, together with a screw to recharge the damp catalyst to the reactor. In some cases pump (39) may be used to charge the catalyst-solvent slurry directly back to the reactor.

The interior of the washer may or may not, depending upon the diameter of the washer, be supplied with baffles at various levels to prevent stratification of the downward flowing catalyst and the upward flowing solvent. At the top of the washer (31) the clarified solvent-oil mixture flows over a circular weir (40), collects in the annular space afforded by the weir and flows out of the washer through pipe (41) to a collecting drum (42). To equalize pressures, the collecting drum is vented by line (43) to line (19) which in turn connects into (14) by means of line (17). A level of solvent-oil is maintained in drum (42) by the level control device (44) operating valve (45). The solution is then allowed to flow out of drum (42) by way of line (46) valve (45) and line (47) to a solvent-oil fractionating system (not shown). In this system the solvent is taken as an overhead cut and returns to the wash system described by way of line (22). Some of the heavy oil is withdrawn as product and the remainder is used as the scrubbing medium in the scrubber (2) and enters this system through line (48) heater (4) and line (3).

There has been described a system for carrying out the invention in the most simple manner. There will now be described a system involving the use of two countercurrent washing stages wherein the catalyst entrained in the synthesis gas leaving the top of the reactor is washed continuously in a first countercurrent washer before joining the main portion of catalyst removed directly from the reactor for dewaxing a second countercurrent washer. The washing medium for the entrained portion of the catalyst is obtained from the second countercurrent washer used for the total catalyst and consequently contains the oil or wax removed in the second washer. Important economies in the use of solvent are effected by means of the two stage arrangement and in referring to a first and second washer limitation to two individual countercurrent washers is not implied. Depending upon the size of the plant it may be desired to use washers both in series and in parallel in the general stages referred to as the first and second.

First, in referring in detail to Figure II, the synthetic products and unreacted materials from the reaction zone, containing entrained catalyst, enter the washing system through line (101) and first pass into a scrubbing zone (103) where they are treated with a high boiling hydrocarbon oil admitted through line (104). The quantity and characteristics of the oil admitted through (104) have been discussed in detail in the previous illustration. The hydrocarbon oil introduced at (104) passes countercurrently downward against the upflowing gases and washes out the entrained catalyst collecting as a slurry at the bottom of the scrubber (103). The reaction products pass overhead through line (105) to a condensation and recovery system (not shown). The temperature and pressure used in (103) are approximately the same as those existing in the reactor.

The slurry of oil and catalyst is withdrawn from the bottom of the scrubber (103) via line (108), the rate of withdrawal being controlled by valve (109) responsive to a liquid level device (110) which serves to maintain a desired level of slurry (L) at all times within the scrubber (103). The slurry then passes into mixer (112) which is located at the top of the countercurrent washer (115). The mixer and washer will be described in detail later. There is also discharged into the mixer (112) via line (113) a portion of the hot solvent-oil mixture obtained from washer (140). As previously stated the solvent will be obtained from the synthetic product and will be a fraction boiling in the range of about 250° F.—450° F. It will be used in the wash system at a temperature of 150° F.—300° F., preferably 200° F.—250° F. The solvent-oil-catalyst slurry is thoroughly mixed in (112) and then passes downwardly in the first washing stage (115). Meanwhile the main portion of the solvent-oil mixture obtained from the second stage (140) enters the bottom of the washer through line (116), is metered and controlled by the flow controller (157) operating valve (158) and passes upward against the downward settling catalyst so as to achieve countercurrent washing in the first washer (115). The quantity of wash solvent entering the bottom of the washer through line (116) is sufficient in quantity to supply that necessary for washing plus that necessary to provide a catalyst slurry in the mixer (141) which contains no more than 4 lbs. of catalyst per gallon of slurry and preferably 1—4 lbs. per gallon. The clarified solvent-oil mixture overflows a weir located in the top of the washer (115) and is withdrawn through line (120) and discharged into receiving vessel (121) and finally withdrawn from the system through line (122) its rate of withdrawal being controlled by valve (123) which, in turn, is responsive to the liquid level device (124) designed to maintain in the receiver (121) a fixed level of liquid. Line (174) is used to establish the pressure on the system and may be connected to the condensation and recovery system served by line (105) either before or after the pressure control valve used to adjust the system pressure. This insures that the entire dewaxing system will operate at a pressure which is lower than the main synthesis system thereby making for more ready transfer of materials from the synthesis system to the dewaxing system.

The slurry is withdrawn from the bottom of washer (115) through line (128) its rate of withdrawal being controlled by the flow meter (159) operating valve (160) and passes into a scrubbing tower (129). The difference between the flow rates on meters (157) and (159) set the rate at which the solvent-oil mixture rises in the washer (115).

The main portion of catalyst to be dewaxed enters the system from the reactor (not shown) through line (161) which submerges below the liquid level maintained in mixer (141). Any occluded gas or gas used for transferring the catalyst from the reactor is disengaged and passes up through the scrubbing tower (129) and out through line (130). It then passes through a cooler (131) which serves to condense out any solvent constituents which might have vaporized in (129). The trap (132) collects the condensate and returns it to (129) via line (133). The gas is removed via line (135) and (162) and pressure controller (163) and finally joins the main recovery system (not shown) via line (164).

As previously stated the slurry is withdrawn from the bottom of (115). It enters scrubber (129) via line (165) and serves to wash catalyst out of the gas rising up through the scrubber. The slurry entering (129) through line (165) combines with the catalyst withdrawn from the reactor through line (161) and the total catalyst-oil-solvent slurry is thoroughly and continuously mixed in (141) and enters the main washing zone of (140) by way of a large number of tubes located at the bottom of the mixer. The level of slurry within the mixer is set by the weir (166) which allows the clarified solvent-oil mixture to flow into receiver (167) by way of line (168). A level (L) is maintained at all times in receiver (167) by the level control device operating pump (170). Thus the solvent-oil mixture in (167) is made available for washer (115) by way of line (171) pump (170) and line (172). Gas in the upper portion (138) is led by line (136) to the vent line (162).

Fresh hot solvent (150° F.—300° F., and preferably 200° F—250° F.) is introduced at the bottom of washer (140) through line (144) and control meter (145) operating valve (173). However, it may be desired to use additional fresh solvent in (115).

The catalyst-slurry flows from the mixer (141) into the main washing zone of (140) and is uniformly dispersed over the cross section of the vessel. Catalyst settles to the bottom of (140) through a slowly rising stream of solvent and is continuously and countercurrently washed.

The slurry from the bottom of (140) is then withdrawn through line (150) and is pumped by means of the pump (151) to a pressure settler (152) to effect further washing and concentration. Slurry from the bottom of settler (152) containing up to 4 lbs. of catalyst per gallon is withdrawn through line (135) passes through the flash coil (156) and then to the top of an aerated standpipe (not shown). Cyclones at the top of the standpipe separate the solvent vapors from the catalyst and the vapors are led to a condensing system (not shown) and are finally accumulated for reuse. The catalyst passes down through the standpipe and is brought back to the reactor by means of gas preferably fresh synthesis gas, or unreacted synthesis gas separated from the reacted products and recycled.

A clarified part of the solvent leaves the top of settler (152) via line (154) and back pressure controller (175) passes through line (176) and enters the washer at two places through line (177) and line (178). Control meter (179) which operates valve (181) is set at the same rate as meter (145) operating valve (173). An orifice restriction (180) provides the necessary pressure drop to allow (179) to function properly.

The use of the pressure settler affords an effective method for reducing the amount of solvent to be vaporized in the flash coil (156) since the concentration of catalyst in the slurry is increased beyond the point at which it can be effectively handled by pumps. As previously stated, however, a filter may be used to remove solvent contained in the slurry collected at the bottom of (140) together with a screw to recharge the filtered catalyst back to the reactor. If desired, pump (151) may be used to charge the catalyst-solvent slurry directly back to the reactor.

The apparatus and operating conditions for conducting the countercurrent washing of the catalyst will now be described more fully. Referring to Figure III, there is shown in enlarged view the internal construction of the washers (115) and (140) of Figure II which carry a large number of parallel tubes in which the catalyst settles through a slowly rising stream of liquid washing agent. It has been found that tubes provide an easy way of obtaining a desirable ratio of the length of the washing path to the diameter, which might range between 10 and 100. Tubes also provide an easy means for installing inclined baffles, which may be frusto-conical, cylindrical or even fins, as shown in Figure IV whose function will be described later. Obviously, there are other ways of subdividing the washer into smaller elements than by the use of tubes.

Thus in Figure III, the catalyst to be washed together with oil and solvent enters the mixer (302) through line (301). The mixer may be mechanically operated by, for example, a motor (303) driving a set of paddles (304). Other means for obtaining a well agitated slurry of uniform composition ranging up to 4 lbs. catalyst per gallon but preferably 2 lbs. per gallon may of course be used. Since a certain amount of washing takes place within the mixer it is desirable to provide a normal retention time ranging from 0.25 to 2 hours depending upon the type of catalyst to be washed. It is preferred to operate the mixer at a temperature of 150° F.—300° F., preferably 200° F.—250° F.

At the bottom of the mixer (302) a number of tubes (305) allow the slurry to flow into the tube wash elements (306) of which the washer is composed. Each inlet tube (305) serves one tube wash element (306) wherein the catalyst settles through the washing medium introduced at the bottom through line (307). The clarified solvent-oil mixture rises in the zone (308) around the outside of the mixer and overflows a weir (309), is collected by the annular space (310) formed by the weir and leaves the washer through (311).

As previously stated the washing medium enters at (307) and enough is provided at this point to provide the countercurrent wash action in the tube elements (306) and also the provide a slurry of suitable catalyst concentration to be removed through line (312). To obtain good washing conditions by the upward flowing liquid from 0.01 to 1.0 gallon of solvent are used per pound of slurried catalyst and the upward average liquid velocity ranges from 0.1 to 1.5 inches per minute. The length of the tube elements provides a washing time which may range between 0.5 hours and 6 hours.

In this type of solids washing it is very essential that the velocity of the washing medium is lower than the settling rate of the slowest settling particle in order that a clarified solution may be withdrawn. It is also essential that stratification of the solid and the washing medium be minimized to obtain a maximum degree of washing. In some cases these requirements are difficult to realize and it is expedient to use internal baffles (401) within the tube washing elements (306) as shown in Figure IV. These baffles become particularly effective when the upward flowing solvent is not introduced in a uniform fashion but rather by definite surges. The easiest way of attaining the surge is for the solvent to be introduced by an intermittently operating reciprocating pump. At each strokes of the pump the velocity conditions within the washing element are much higher than average and each baffle causes a slight turbulence in its immediate vicinity thereby mixing the catalyst in the zone and causing redistribution thereby eliminating any tendency for stratification. During the period when the pump is not operating the catalyst settles in entirely quiescent liquid. Regulation of the quantity and rate, at which surges are imposed, establishes another means for controlling the concentration of catalyst within the washing elements and hence the time of settling and the degree of washing. Of course other methods could be used to impose the surge condition instead of by means of a reciprocating pump. For example by varying the pressure in a gas (either air or an added gas) contained in the spaces between the outsides of the washing tubes, the vessel proper, the upper tube sheet and the level of liquid surrounding the tubes. The gas (which is above the liquid level in the spaces surrounding the tubes), may be in communication with an outside reservoir by means of which (and proper piping) the gas pressure may be varied. It will be noted that the spaces around the tubes and the tube interiors are in communication, there being no bottom tube sheet.

Having now particularly described and ascertained the nature of our said invention, and in what manner the same is to be performed, we declare that what we claim is:—

1. A process for removing heavy hydrocarbons and waxes deposited on finely-divided catalysts during the catalytic production of hydrocarbons from mixtures of hydrogen and carbon monoxide, comprising treating the catalyst in the form of a slurry, in countercurrent flow with a stream of solvent for the heavy hydrocarbons and waxes, such a solvent being also used for slurrying the catalyst.

2. A process according to Claim 1, in which the extraction is carried out in an extraction zone at one end of which the slurry of contaminated catalyst is introduced and the solvent extract removed and at the other end of which the solvent is introduced and a slurry of extracted catalyst in the solvent removed.

3. A process according to Claims 1 and 2, in which the solvent is a hydrocarbon oil, preferably an oil fraction derived from the products of the reaction for the synthesis of hydrocarbons from mixtures of hydrogen and carbon monoxide, in which the catalyst was used.

4. A process according to any of the preceding claims, wherein the slurry flows downwardly through the extraction zone, and the solvent flows upwardly through the extraction zone.

5. A process according to Claim 4, wherein the upward velocity of the solvent is so controlled relative to the particle size of the solid that a substantially clear solution of contaminants in solvent is withdrawn from the upper part of the extraction zone, and a slurry of decontaminated catalyst is withdrawn from the lower part of the extraction zone.

6. A process according to any of the preceding claims wherein the solids are slurried and passed downwardly through a first extraction zone in countercurrent flow to solvent passing upwardly through the first extraction zone, a slurry of washed solids being withdrawn from the bottom of the first extraction zone and mixed with fresh contaminated solid slurry, the mixed slurry being passed downwardly through a second extraction zone in countercurrent flow to solvent passing upwardly through the second extraction zone, washed solid slurry being withdrawn from the bottom of the second extraction zone, and clear contaminant-solvent solution being withdrawn from the top of the second extraction zone, and this clear contaminant-solvent solution being employed for the slurrying of the solids introduced to the first extraction zone, and as the solvent introduced into the lower part of the first extraction zone.

7. A process according to Claim 6, wherein one or both of the extraction zones comprises a series of conduits in parallel with or without baffle means, solid slurry being separately introduced into the top of each conduit and solvent flowing upwardly through all the conduits, the streams of solvent emerging from the top of the conduits being combined and withdrawn, and the streams of solid emerging from the bottom of the conduits being combined and withdrawn.

8. A process according to Claim 6 or Claim 7, wherein the solvent flow is intermittent, whereby surges are caused, thus enhancing slurry-solvent mixing.

9. A process according to any of Claims 6 to 8, wherein from 0.01 to 1 gallon of solvent is used per pound of slurried catalyst.

10. A process according to any of Claims 6 to 9, wherein the average upward velocity of the solvent is from 0.1 to 1.5 inches per minute.

11. A process according to any of Claims 6 to 10, wherein the washing time in the extraction zones is from 0.5 to 6 hours.

12. A process according to any of the preceding claims, wherein the slurry comprises 1 to 4 and preferably 2 lbs. of catalysct per gallon of solvent.

13. Apparatus for carrying out the process of any of Claims 2 to 12, comprising an extraction chamber and a mixing chamber, conduit means for the separate introduction of finely divided catalyst and solvent to the mixing chamber, conduit means for the introduction of slurried solids into the upper part of the extraction chamber, conduit means for the introduction of solvent into the lower part of the extraction chamber, and withdrawal conduits at the upper and lower ends of the extraction chamber for the withdrawal of contaminated solvent, and purified solid slurry respectively.

14. Apparatus according to Claim 13,

wherein the extraction chamber comprises a plurality of open-ended tubes in parallel, separate conduit means leading from the mixing chamber to the upper part of each tube.

15. Apparatus according to Claim 13 or Claim 14, wherein the said tubes are provided with baffle means.

16. Apparatus according to any of Claims 13 to 15, wherein there is provided a reciprocating pump for the introduction of the solvent into the extraction chamber in surging flow.

17. Apparatus according to any of Claims 13 to 16, comprising two such systems in series, conduit means being provided for the introduction of purified slurry from the first extraction chamber to the second mixing chamber, and conduit means being provided for the introduction of the contaminated solvent withdrawn from the second extraction chamber partly into the first mixing chamber and partly into the lower part of the first extraction chamber.

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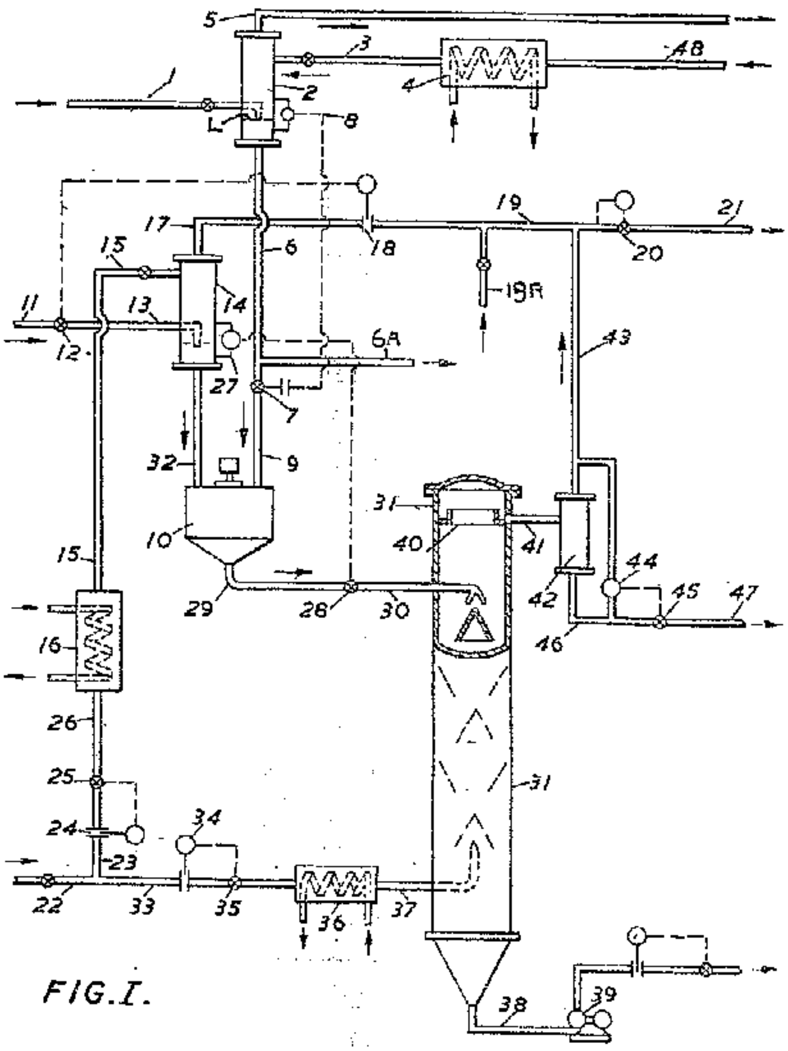

FIG. I.


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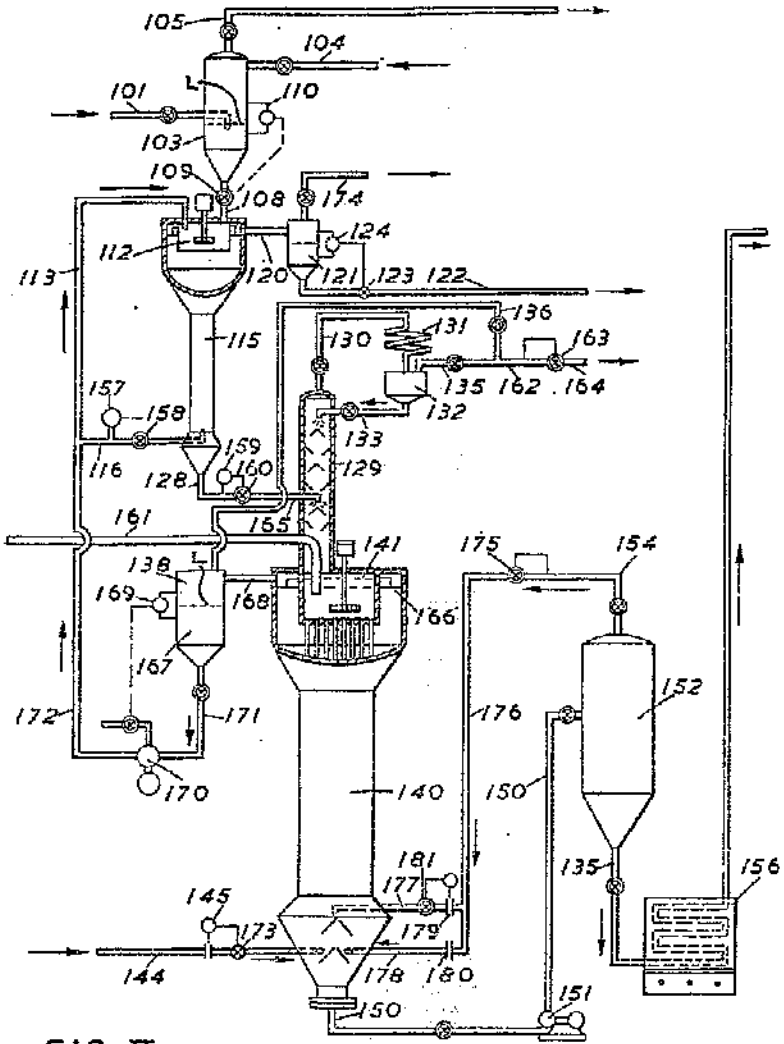
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FIG. II.

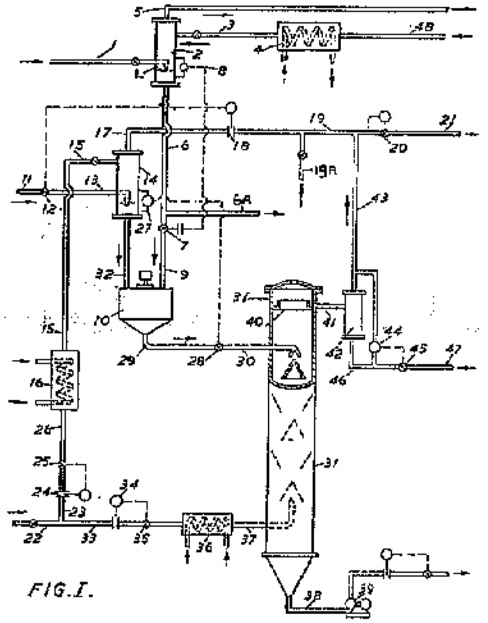

FIG. I.

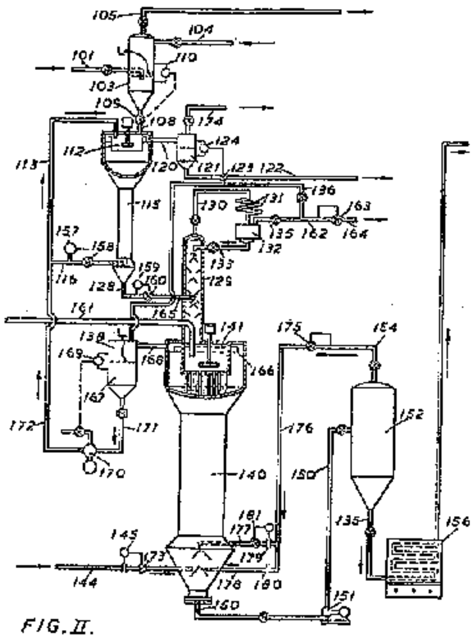

FIG. II.

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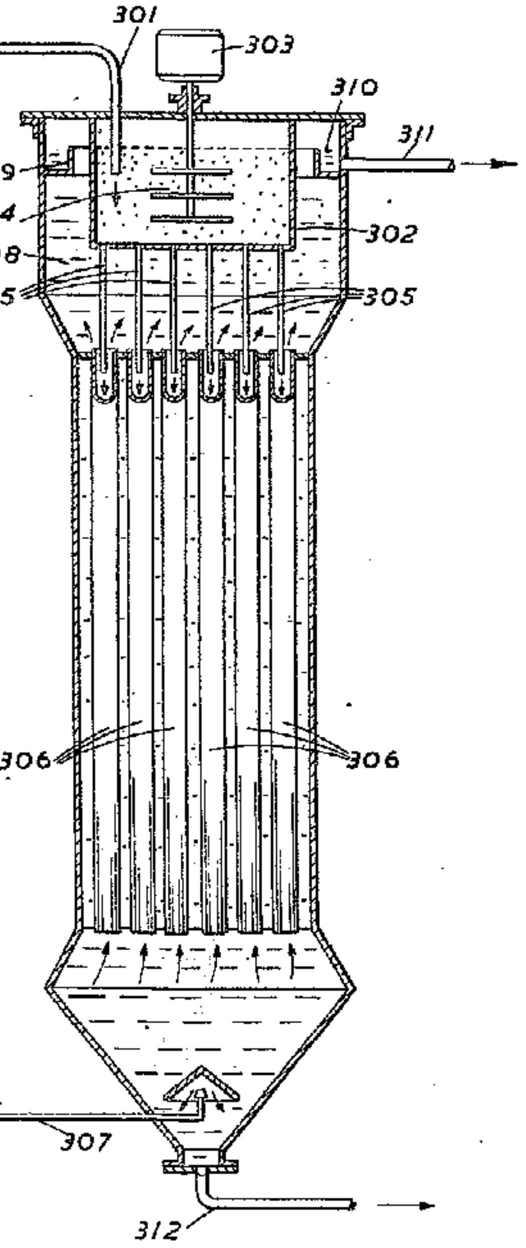

FIG. III.

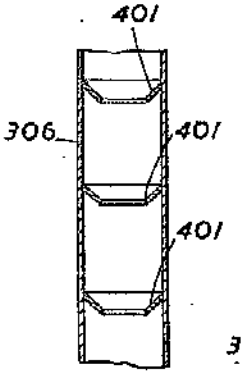

FIG. IV.