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(54) APPARATUS FOR THE MECHANICAL SEPARATION OF OIL-WATER EMULSIONS AND THE RECOVERY OF THEIR CONSTITUENTS

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 organized under the laws of Hungary, do
 5 hereby declare the invention for which we
 pray that a patent may be granted to us, and
 the method by which it is to be performed,
 to be particularly described in and by the
 following statement:—

10 The invention concerns apparatus for the
 mechanical separation of oil-water emul-
 sions and the recovery of their constituents.

It is known that in industrial plants, par-
 ticularly in industrial degreasing plants, the
 15 concentrated emulsifying chemicals used
 generate oil-water emulsion liquids which
 must periodically or continuously be
 removed from the degreasing plant. The
 effluent containing emulsifying chemicals
 20 and oil in a high concentration would pol-
 lute and contaminate the biological envi-
 ronment. Before being discharged into liv-
 ing waters the effluent must, therefore, be
 de-oiled and decontaminated to meet the
 25 requirements of an effective ecological pro-
 tection of the living environment.

The generally known method of oil flota-
 tion based on the difference in specific
 gravities is not a practical proposition for
 30 this purpose due to dissolved surface-active
 substances contained in effluent, and due to
 the low concentration of oil. Such emulsions
 are, therefore, mechanically separated by
 macro-filtration, a term explained below. In
 35 this process the emulsion to be treated is
 collected in a storage tank from where the
 liquid, after being pumped through a so-
 called macrofilter unit, is recirculated into
 the collecting/storage tank. This process is
 40 repeated until the oil content of the emul-
 sion increases to 40-50%. Thereafter the
 liquid is pumped into a sedimentation tank
 where it is left to settle for 6-8 hours. Subse-
 45 quently the oil is drawn off from the surface
 of the liquid contained in the sedimentation

tank. The residual aqueous phase is then
 pumped back into the storage tank where it
 is filled up again with emulsion to be treated
 and the cycle is then repeated. The separa-
 tion of the chemically strongly bonded 50
 emulsions by this method is, however, a very
 protracted and intermittently operated pro-
 cess and the functioning of the separation
 plant requires permanent supervision. Another
 drawback is that the separated oil 55
 contains large quantities of mechanical im-
 purities; it needs, therefore, further purifi-
 cation before it can be used again or, if the
 oil is to be used as a fuel, it requires
 special burners for combustion. 60

An aim of the invention is to eliminate or
 reduce the above-described shortcomings
 and to provide relatively simple and inex-
 pensive apparatus in which the separation of
 the strongly bonded oil-water emulsions 65
 formed in industrial degreasing plants can
 be carried out mechanically in a continuous
 operation in a fully automated manner, and
 in which the oil-water emulsions are not
 only separated into their constituent parts 70
 but also the degreased solution is rendered
 re-usable while the separated oil is purified
 to such an extent that for less demanding
 purposes it can be used without after treat-
 ment. 75

Thus the invention concerns apparatus for
 continuously mechanically separating oil-
 water emulsions and for recovering their
 constituent parts, comprising a collect-
 ing/storage tank for containing the emulsion 80
 and connected to an ultra-filter unit includ-
 ing a membrane which allows passage of
 water and surfactants from the emulsion
 whilst not allowing passage of oil, an after-
 sedimentation tank with an overflow outlet 85
 for separated oil, a mechanical separation
 column the lower part of which is formed as
 a hydrocyclone provided with tangentially
 arranged inlet apertures and a discharge
 pipe for heavier liquids separated in the 90

hydrocyclone while its upper part has a flow-connection with the hydrocyclone for lighter liquids separated in the hydrocyclone, the said upper part being also connected to the said after-sedimentation tank, the ultrafilter unit having an outlet on the filtrate side thereof and being by-passably connected in a recirculation ducting that is connected between the said discharge pipe and the said inlet apertures of the hydrocyclone, and a circulation pump connected, by appropriately setting a valve, to the collecting/storage tank, to the discharge pipe of said separation column or to both said collecting/storage tank and said discharge pipe of said separation column, the pump circulating the liquid from the discharge pipe and/or the collecting/storage tank through the recirculation ducting.

According to a preferred embodiment of the invention, the upper part of the separation column is constructed as a recirculating sedimenting device, the hydrocyclone and the recirculating sedimenting device being separated by an upstanding frustoconical funnel in the rim of which suction apertures are provided, while under the base plane of the funnel and on the axis of the separation column an inlet pipe for the introduction of compressed air is arranged.

In use, the apparatus according to the invention separates with the aid of the membrane of the ultrafilter unit, the water and a large portion of the surface-active materials (surfactants) from the oil particles bound into an aqueous emulsion by the surfactants. Consequently, the emulsifying effect of the chemicals is reduced thus loosening the emulsive bonding of the constituents of the emulsion. Then from this loosened emulsion the separated oil particles are floated to the recirculating-type sedimentator arranged in the upper part of the separated column, with the aid of air bubbles formed by introducing compressed air through an inlet pipe arranged in the line of axis of the funnel and by the effect of the centrifugal force prevailing in the upper part of the hydrocyclone. In the sedimenting device, the un-bonded oil particles float to the surface while those remaining bonded settle to the bottom of the recirculation-type sedimentating device, from where they are sucked back into the cyclone-space by the recirculating stream entering thereto through the suction orifices arranged over the inlet apertures of the hydrocyclone. The mechanical impurities of high specific gravity move along the lower conical surface of the hydrocyclone, arrive at the base of the cone and from there into the storage tank. The liquid of high concentration of oil passes via the overflow at the upper part of the column or tower to the after-sedimentation tank where it is further broken down. Here,

oil of relatively high purity is removed via an overflow, while the settled aqueous emulsion is recycled to the beginning of the process, i.e. to the collecting/storage tank. The filtrate passing through the membrane of the ultra-filter unit can be used again as a degreasing chemical after suitable enrichment.

According to a preferred embodiment, it is of advantage if the moving part of a valve built into the recirculation pipe line connecting the storage tank with the ultra-filter unit is controlled by electrical impulses received from a turbidity meter fitted into the sedimentation space of the hydrocyclone arranged in the lower part of the separation column and by impulses from a turbidity meter and a level sensing element fitted into the recirculation-type sedimentator arranged in the upper part of the separation column. By virtue of this design the quantity of the water or liquid to be cleaned in any given time can be monitored and controlled in a simple way.

The after-sedimentation tank is preferably provided with a lower emulsion outlet which is connected to a further valve controlled by additional turbidity meters arranged in the upper and lower parts of the said after-sedimentation tank, said further valve being connected to the collecting/storage tank by way of a pipe. This particular technological feature of the apparatus makes the automatic cleaning of the after-sedimentator possible and ensures that the enriched liquid (liquor) is returned into the storage space.

Finally, in another preferred embodiment of the invention, the filtrate side of the ultrafilter unit or its regeneration pipe may be connected to a syphon-tank fitted with an inlet for regenerating material and connected to a compressed air supply through a pipe line.

The invention is described, purely by way of example, by means of a preferred embodiment illustrated in the accompanying drawing which shows a schematic lay-out of the apparatus.

The lower part of the mechanical separation column or tower 1 consists of a hydrocyclone 2 with three off-take taps, while the upper part of the separation column 1 is constructed as a recirculating type sedimentator 3. The hydrocyclone 2 is separated from the recirculating-type sedimentator 3 by an upwardly tapered and upwardly open frusto-conical funnel 4. The rim of the base of funnel 4 adjoining the inner surface of the separation column 1 is provided with concentric suction orifices 5. At the base of funnel 4, in the line of axis of hydrocyclone 2, there is an inlet pipe 6 for introducing compressed air. The funnel 4 provides a flow connection between the hydrocyclone

2 and the upper part of the column 1.

The lower cone-shaped part of the separation column 1 constructed as a hydrocyclone 2 is connected to a trap 7 for collecting 5 mechanical impurities and provided with a discharge pipe fitted with a discharge valve 8. The heavier, separated liquid particles are removed through this pipe, in order of their specific gravities, the heaviest being discharged first. In addition, the conical lower 10 part of hydrocyclone 2 is fitted with a draw-off pipe 9 capped by a deflector cap. The draw-off pipe 9 capped by a deflector cap. The draw-off pipe 9 is connected to a 15 three-way valve 10 forming part of a recirculation circuit consisting of a circulating pump 11, a shunting by-pass pipe line 12 a filter and pressure regulator 13, a so-called macrofilter unit 15 (which is an ultrafilter 20 unit the elements of which comprise filaments of macroscopic size) equipped with a valve 14 and a recirculation pipe line 16 connected to the inlet orifice of the hydrocyclone 2.

25 An emulsion-collecting storage tank 19 can be connected to the recirculation circuit by a pipe line 17 via the three way valve 10 and a valve 18. By appropriately controlling and actuating the valve 10 the pump 11 can 30 draw liquid either from the tank 19 via the pipe 17 or from the separation column 1 via the emulsion draw-off pipe 9.

The recirculation sedimentator 3 which forms the upper part of the separation column 1 is connected via an overflow device 35 20 arranged on its upper part to an after-sedimentation tank 21. On the upper part of the after-sedimentation tank 21 an oil overflow pipe 22 is disposed for the removal in 40 operation of the separated oil while at the bottom of the after-sedimentation tank 21 a discharge pipe fitted with a valve 23 is disposed for the removal of sedimented emulsion of high specific gravity. The latter discharge pipe is connected via a pump 24 to 45 the emulsion collecting storage tank 19.

At the bottom of the hydrocyclone 2 of the separation column 1, a nephelometer (turbidity meter) 25 is disposed. In the 50 upper part of the separation column 1 in the recirculating sedimentation space 3 another turbidity meter 26 as well as a liquid level sensor 27 are arranged. The turbidity meters 25 and 26 and the level sensor 27 are 55 electrically connected via a control unit (actuator unit) 28 to the moving part of the three-way valve 10, the valve 14 of the macrofilter unit 15 and the valve 18 of the storage tank 19. Similarly, further turbidity 60 meters 29 and 30 arranged on the upper and lower parts respectively, of the after-sedimentation tank 21 are electrically connected to the control element of the valve 23 and of the pump 24, via the unit 28.

65 The filtrate side of the macrofilter unit 15

is connected via a valve 31, a pipe line 32 for the removal in operation of the filtrate and a three-way valve 33 to a syphon tank 34. A filtrate drain pipe is connected to the three-way valve 33. The syphon-tank 34 is 70 connected to a pipe line 35 for receiving a supply of regenerating material for the regeneration of the membrane of the macrofilter unit 15 and to an air supply pipe line 37 fitted with a blow-off valve 36. The 75 syphon tank 34 is connected via a pipe line 38 and a three-way valve 39 to the regenerating material inlet of the macrofilter 15.

Level-sensing elements *a* and *b* on the one 80 hand and *c*, on the other hand, of a liquid level detector 40 are arranged respectively in the upper and lower parts of the syphon tank 34. The level sensing elements *a*, *b*, and *c* are electrically connected via a non- 85 illustrated amplifier with the moving parts of valves 33, 36 and 39.

In the course of operation of the apparatus according to the invention, the emulsion to be separated is collected and 90 stored in the collecting storage tank 19. By actuating the circulation pump 11 the separation column 1 is filled up to the level of the overflow via the high-pressure by-pass line 12. By opening the valve 14 of the 95 recirculation duct 16 and slowly throttling the valve of the by-pass line 12, the desired inlet pressure of the macrofilter unit 15 is set. By bringing the apparatus into normal operation in this way, the liquid is kept in 100 circulation via the drainage pipe 9 arranged at the bottom of the hydrocyclone 2 and provided with a reflector cap, the pump 11, the by-pass line 12, the recirculation duct 16 and the macrofilter unit 15. 105

As has already been mentioned, the bottom part of the separation tower 1 functions as a hydrocyclone 2 with three off-takes. The upper inlet apertures of the hydrocyclone 2 are arranged tangentially so that a 110 swirl is imparted to the liquid in the hydrocyclone as it arrives via the recirculation duct 16 or by-pass duct 12. The contaminants of high specific gravity settle or collect in the collecting space 7 disposed at the 115 downwardly conically formed end of the hydrocyclone 2. These contaminants may be periodically removed from the collecting space 7 by opening the valve 8 for a short time. 120

The emulsion of higher specific gravity precipitates beneath the conical funnel 4 arranged at the top of the hydrocyclone 2. The oil separating from the emulsion of high 125 oil content and low specific gravity is forced upwardly through the narrowing or tapering part of the funnel 4 by the stream of compressed air issuing from the pipe 6; in other words, the oil-enriched liquid advances upwardly through the funnel 4 and passes to 130

the recirculating sedimenter 3 of the separating tower 1, where the separated-out oil particles float to the surface of the liquid. The bound emulsion of higher specific gravity settles to the bottom of the recirculating sediment space 3 from where, via the suction orifices 5 disposed between the lower rim of the funnel 4 and the inner surface of the separation tower 1, it flows back into the hydrocyclone 2 under the suction effect of the hydrocyclone.

In the flow circuit associated with the hydrocyclone 2 of the separation tower 1 the materials of low molecular weight (water and surfactants) are filtered through the membrane of the macrofilter unit 15 while the oil particles of high molecular weight rebound from the membrane and are recirculated in the circuit to the hydrocyclone 2. By detaching a part of the water and of the emulsifying chemicals from the emulsion in this way, the mechanical separation as already described above becomes possible.

In normal operation, the pulse signals of the level sensor 27 arranged in the recirculatory sedimenting space 3 of the separation tower 1 controls the three-way valve 10 as well as the actuating element of the valve 18 of the collecting/storage tank 19, by way of the control device or unit 28. Thus the level sensor 27 controls or meters the feed from the tank 5 as a function of filtrate flow. In normal operation the level sensing device 27 is interlocked with the turbidity meter 26.

The efficiency of the macrofilter unit 15 suddenly deteriorates when the oil reaches a concentration of about 50-60%. To avoid this, before attaining an oil concentration of 50% the turbidity meter 25 gives out a pulse signal and switches the turbidity meter 26 in. Because of the above-mentioned interlock, after its actuation the turbidity meter 26 disconnects the level sensor 27 from the control loop and takes the control over itself.

In the case where the turbidity meter 26 measures a concentration of oil which can be permitted to pass into the after-sedimentation tank 21, the infeed rate is regulated or set by adjustment via the control unit 28 of the valve 18 of the collecting/storage tank 19 and the three-way valve 10, and closing the valve 14 disconnects the macrofilter unit 15, whereby to circulate the emulsion through the by-pass pipe line 12. Consequently, the level of the liquid in the separating column 1 rises continuously and the liquid of high oil concentration flows via the overflow device 20 into the after-sedimentation tank 21. While the oil-enriched liquid is rising in the separation column 1, the oil content in the hydrocyclone 2 is decreasing. As soon as the turbidity

meter 26 registers an oil concentration value below the permissible predetermined limit concentration and the turbidity meter 25 arranged in the space of the hydrocyclone 2 in the lower part of the separation column 1 registers an oil concentration below 50%, the control is taken over again by the level sensor 27.

The solution of high oil concentration flows from the separation column 1 through the overflow device 20 into the after-sedimentation tank 21 where the oil particles still bound to the emulsifying chemicals settle and sink down to the bottom of the after-sedimentation tank 21. The relatively pure oil is continuously removed via the oil outlet 22. If the turbidity meter 29 of the after-sedimentation tank 21 registers a lower oil concentration of the aqueous solution than a predetermined value, an impulse from the turbidity meter 29 monitored by actuator/control unit 28 opens the valve 23 fitted to the oil outflow pipe 22 and starts the pump 24, thus recirculating the aqueous emulsion liquid from the after-sedimentation tank 21 into the collecting/storage tank 19. If the level of the aqueous solution contained in the after-sedimentation tank 21 sinks below the level where the turbidity meter 30 can detect recyclable liquid, it emits an impulse signal via the control unit 28 to stop the pump 27 and closes the valve 23 of the outlet for the remove of the aqueous emulsion.

The trouble-free operation of the macrofilter unit 15 is ensured by the filter and the pressure limiter 13 fitted in the recirculation duct 16. The impurities must from time to time be removed from the membrane of the macrofilter unit 15 and the membrane must be regenerated by a disinfecting material.

In the basic position before regeneration, the three-way valve 39 is closed towards the macrofilter unit 15 and the recirculation pipe 16. The quantity of the regenerating material sufficient for one regeneration is fed through the pipe line 35 of the syphon tank 34 and then the valve of the pipe line 35 is closed. In normal operation of the apparatus the syphon tank 34 is filled up with filtrate via the valve 31 arranged at the filtrate side of macrofilter unit 15, pipe line 32 and three-way valve 33 until the level of the uppermost sensing element *a* of level detector 40 is reached. At this stage, the electric impulse signal emitted by the uppermost sensing element *a* of liquid level sensor 40 sets the moving part of three-way valve 33 into operation in such way that the valve is closed towards the syphon-tank 34. At the beginning of the regenerating of macrofilter unit 15, the three-way blow-off valve 36 of the compressed air supply pipe line 37 is opened between the pipe line 37

and the syphon tank 34 and after opening the valve 39 into the recirculating pipe line 16, the regenerating material is pressed into the macrofilter unit 15 by the compressed 5 air. When the level of the liquid contained in the syphon tank 34 sinks below the second sensing element *b* positioned in the middle part of the syphon tank 34, the sensing element *b* of the level-detector 40 emits an 10 impulse signal which is monitored by the control unit 28 and this impulse signal opens valve 39 towards the filtrate side and presses the filtrate through the membrane of the macrofilter unit 15 and back into the emul- 15 sion space. On receiving an impulse signal of sensing element *c* of the liquid level detector 40 positioned on the lower part of syphon tank 34 the apparatus is set again into its normal working position.

20 The great advantages of the apparatus according to the invention are: continuous operation, simple but full automation and low investment costs. Another advantage is that, due to the continuous regeneration, it 25 can be directly connected to any technological process in industrial plants.

WHAT WE CLAIM IS:—

1. Apparatus for continuously mechanically separating oil-water emulsions and for 30 recovering their constituent parts, comprising a collecting/storage tank for containing the emulsion and connected to an ultrafilter unit including a membrane which allows passage of water and surfactants from the 35 emulsion whilst not allowing passage of oil, an after-sedimentation tank with an overflow outlet for separated oil, a mechanical separation column the lower part of which is formed as a hydrocyclone provided with 40 tangentially arranged inlet apertures and a discharge pipe for heavier liquids separated in the hydrocyclone while its upper part has a flow-connection with the hydrocyclone for lighter liquids separated in the hydrocyc- 45 lone, the said upper part being also connected to said after-sedimentation tank, the ultrafilter unit having an outlet on the filtrate side thereof and being by-passably connected in a recirculation ducting that is 50 connected between the said discharge pipe and the said inlet apertures of the hydrocyclone, and a circulation pump connected, by appropriately setting a valve, to the collect-

ing/storage tank, to the discharge pipe of said separation column or to both said col- 55 lecting/storage tank and said discharge pipe of said separation column, the pump circulating the liquid from the discharge pipe and/or the collecting/storage tank through the recirculation ducting. 60

2. Apparatus according to claim 1 wherein the upper part of the separation column is constructed as a recirculating sedimenting device, the hydrocyclone and the recirculating sedimenting device being sepa- 65 rated by an upstanding frustoconical funnel in the rim of which suction apertures are provided, while under the base plane of the funnel and on the axis of the separation column an inlet pipe for the introduction of, 70 compressed air is arranged.

3. Apparatus according to claim 1 or 2 wherein the said valve has a moving part that is controlled by electric impulses received from a turbidity meter arranged in 75 the sedimentation space of the said hydrocyclone and by signals received from a further turbidity meter and a liquid level sensor arranged in the upper part of the said column. 80

4. Apparatus according to any of claims 1 to 3 wherein the said after-sedimentation tank is provided with a lower emulsion outlet which is connected to a further valve controlled by additional turbidity meters 85 arranged in the upper and lower parts of the said after-sedimentation tank, said further valve being connected to the collecting/storage tank by way of a pipe.

5. Apparatus according to any preceding 90 claim wherein the filtrate side of the ultrafilter unit is connected to a syphon-tank which is fitted with an inlet pipe for the introduction of filter regenerating material and which tank is connectable to a source of 95 compressed air.

6. Apparatus according to claim 1 substantially as herein described with reference to and as shown in the accompanying drawing.

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