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(71) Applicant: **Itelyum Regeneration S.p.A.**
26854 Pieve Fissiraga (LO) (IT)

(72) Inventors:
- **MAGNONE, Giuseppe**
  26854 Pieve Fissiraga (LO) (IT)
- **NEGRI, Francesco**
  26854 Pieve Fissiraga (LO) (IT)
- **PELIS, Oscar**
  26854 Pieve Fissiraga (LO) (IT)
- **QUAGLIA, Alessandro**
  26854 Pieve Fissiraga (LO) (IT)
- **ROLDI, Aldo**
  26854 Pieve Fissiraga (LO) (IT)
- **FUGAZZA, Gianluca**
  26854 Pieve Fissiraga (LO) (IT)
- **CODOGNOLA, Marco**
  26854 Pieve Fissiraga (LO) (IT)
- **GALLO, Francesco**
  26854 Pieve Fissiraga (LO) (IT)

(74) Representative: **Faggioni, Carlo Maria et al**
Fumero S.r.l.
Pettenkoferstrasse 20/22
80336 München (DE)

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Amended claims in accordance with Rule 137(2) EPC.

(54) PROCESS FOR THE PRODUCTION OF BIOLUBRICANTS FROM WASTE MATERIALS

(57) A process for the production of biolubricants from waste materials is decribed, wherein said waste materials are UCO, RUCO and a biological fraction of waste lubricants. Two lines (22, 46) are combined for a subsequent reaction (80) after two separate preliminary treatments (76, 78; 79), wherein the first line (46) brings UCO and RUCO which have been processed (79) and the second line (22) brings a biological fraction of lubricants separated (76, 78) from waste lubricants.

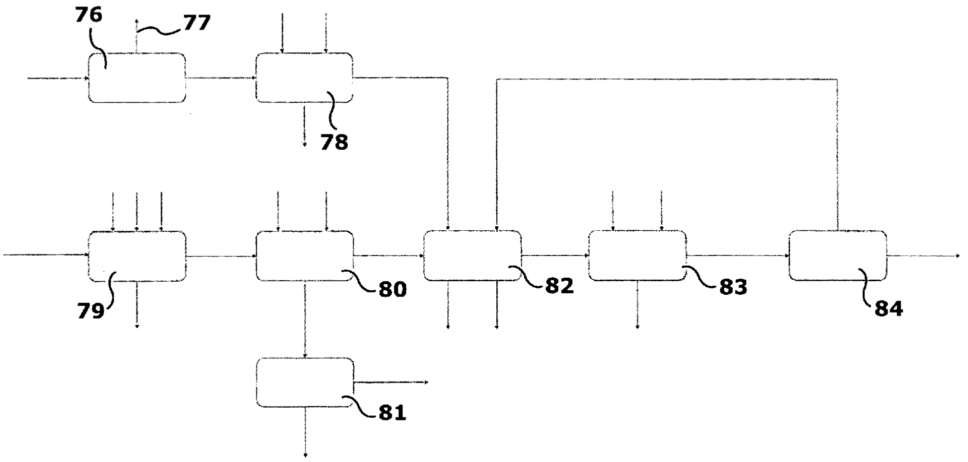

Fig. 2

Description

FIELD OF THE INVENTION

[0001] This invention relates to a process for the production of biolubricants, using waste materials as raw materials. This process is within the so-called circular economy.

BACKGROUND OF THE INVENTION

[0002] The massive industrial production has led to the creation of a large amount of wastes of any kind (solid, liquid, gaseous). This happens at any level. There are industrial wastes and there are wastes produced by common people, when replacing some object or tool. Such wastes should be disposed of, and this is a big issue.

[0003] The disposal of wastes was hardly a problem before the mass production since objects used to be used and repaired until they failed or broke down. In this way, wasted products did not pollute air, water, or soil, since there was not a critical mass for that. This is no longer the situation, now.

[0004] A particular kind of wastes are wastes coming from chemicals. Many of the chemicals used every day, in the normal life, are in fact very harmful for health and, in case they are simply left in the environment, they would pollute rivers, lakes and seas, or air, or the soil and plants growing up there. Oils, solvents, lubricants, bleaching agents, chelating agents and others are among the examples of chemicals which are commonly wasted and which lead to health issues.

[0005] Lubricants are in use since a very long time and they are used when mechanical members are moving one with respect to the other and they need to have a relatively low friction, so as to avoid gripping, overheating and the loss of some materials by abrasion. Since machines and cars are presently a lot, it can be imagined how much waste lubricants are discarded every day around the world. Most lubricants are harmful when ingested and their spontaneous decomposition can be even very time consuming.

[0006] When it was realised that the amount of waste lubricants was so high that their careless disposal of would have led to an ecological disaster, some ways to dispose of it were thought of. A first idea was to burn it. This can work, but it increases the amount of produced CO_2 (the product of a complete combustion), and the recovered energy is however not enough to compensate the energy employed to produce the fresh lubricant. Efforts have been employed to regenerate exhausted lubricants, so as to prepare bases for new lubricants, which can be obtained by the bases by formulating them according to needs.

[0007] This kind of reclamation has generally proven effective, leading to a good product, to bitumen as an enhanced by-product (to be sold), to a dramatic reduction in pollution and to good savings in oil to be used to prepare lubricants. The main issue in this kind of processes is to improve a proper collection of waste, because a waste collected properly is easier to be treated, leads to a better quality of products, involves lower costs and leads to a reduced disposal of remaining wastes.

[0008] The Applicant has filed a number of patents referred to this kind of processes. The basic scheme, with a number of possible modifications, involves a flash initial step, removing water which entered the waste lubricant, followed by a fractioned distillation, removing bitumen and similar substances therefrom and a final hydrorefining step. Other steps can be inserted in the process, often improving it. For instance, the liquid leaving the flash column can be centrifuged, decanted and the bases to be hydrofinished can undergo ultrafiltration, for getting very pure bases. Solids can be further ground and can undergo further purification steps, so as to increase the yield, waste lubricants can be admixed with strong bases, like KOH, before the flash distillation, and so on.

[0009] Water removed from the waste lubricants during the flash step is not pure enough to be discarded and should undergo purification steps, which involve costs for energy and chemicals, and, in any case, the disposal of such water can increase the pollution of rivers and lakes. It would be good to find a way to dispose of this water with no problem or even to use it for some industrial purpose.

[0010] Another kind of waste are food grade oils, like used olive oil, sunflower oil, maize oil and so on.

[0011] Such oils are often used to fry food or to prepare some preserved food, especially mushrooms and artichokes. Normally, people waste such oils by pouring them in the sewage, thinking that such oils are not dangerous, being healthy as food. However, although they are not toxic, they are in any case a source of pollution, remaining as such for a long time and not decomposing spontaneously in a reasonably short time. This leadS to the creation of some fatberg, a sort of mass of oil, butter and other fats, with other impurities contained in the sewage. Fatbergs can lead to the failure of sewage, with dirty water spreading on the roads. It would be advisable to avoid situations like this. It would therefore be suitable to find a way to reutilise UCO (Used Cooking Oils), and possibly to find a good application for RUCO (Regenerated Utilised Cooking Oils), into which often UCO is transformed.

[0012] Generally, two different plants and processes are needed for regenerating waste lubricants on one hand, and UCO, RUCO and waste biolubricants, on the other hand.

PROBLEM AND SOLUTION

[0013] The problem underlying this invention is to propose a process for the production of biolubricants from waste materials, which allows to overcome the drawbacks set forth above and which allows to reutilise

UCO and to find a purpose for RUCO and simultaneously to regenerate lubricants within the same plant and process, without soil consumption related to the building of new premises. This object is achieved through a process for the production of biolubricants from waste materials, wherein said waste materials are waste biolubricants, UCO, RUCO and a biological fraction of waste lubricants, characterised in that two lines are combined for a subsequent reaction after two separate preliminary treatments, wherein the first line brings waste biolubricants, UCO and RUCO which have been processed and the second line brings a biological fraction of lubricants separated from waste lubricants. Subclaims disclose preferential features of the invention.

[0014] According to this invention, a process is provided which allows to reuse the biological fraction of waste lubricants on one hand and waste vegetable oils, like UCO and RUCO, and biolubricants on the other hand, comprising a series of treatments leading the two groups of waste oils to be transformed into the same product, which is then mixed, hydrolysed and treated, in order to get biolubricants.

[0015] According to an embodiment, the treatments which the biological fraction of waste lubricants undergo include a pre-flash step. This pre-flash step results in the removal of water from the lubricants. The lubricants exiting the pre-flash step, present in the form of soaps, undergo a decanting step, which allows to separate mineral oils from the biological fraction, containing mainly derivatives of fatty acids. The mineral oils are preferably sent to a plant for the regeneration of waste oils, which is not part of this invention. The biological fraction is then added with sulfuric acid or another strong mineral acid, in order to get fatty acids from the soaps.

[0016] According to an embodiment, the treatments that waste vegetable oils and biolubricants undergo include centrifugation. Preferably, such a centrifugation takes place in more than one step, most preferably in three steps. In case three steps are provided, the first one is carried out on the waste vegetable oils as such, preferably after heating and stirring, so removing wastewater and solids; the second step is carried out on the liquids leaving the first centrifugation step; preferably, before such second centrifugation step, the liquid is washed with distilled water. The second centrifugation step allows to remove wastewater and rubbers which are soluble in water. The third step takes place on the liquid coming from the second centrifugation step. Preferably, before undergoing the third centrifugation step, the liquid is reacted with H_3PO_4. According to a preferred embodiment, wastewater coming from the centrifugation steps are neutralised with a strong base. The third centrifugation step allows to remove wastewater and rubbers which are not soluble in water.

[0017] According to this invention, the liquids coming from the previous two treatments are mixed together, before undergoing further process steps.

[0018] According to the invention, the mixture undergoes a hydrolysis reaction, catalysed with ZnO, which leads to an aqueous and an oily phases. Preferably, more than one hydrolysis step is carried out, more preferably two hydrolysis steps are carried out. The aqueous phase, containing mainly glycerine, is purified, in order to get pure glycerine. Preferably, the purification of glycerine includes a pre-flash step, in order to remove water therefrom. Preferably, the purification of glycerine includes a distillation in a plate distillation column, separating wastewater, pure glylcerine and bitumen.

[0019] According to an embodiment, the oily phase is dehydrated.

[0020] According to an embodiment, the dehydrated oily phase undergoes a purification step. Preferably, such a purification step includes distillation. According to a preferred embodiment, such a distillation takes place in a plate distillation column. According to an embodiment, this distillation allows to separate the oily phase from bitumen, which is stored for sale, and ZnO, which can be recovered and employed. According to an alternative embodiment, the distillation residue, containing bitumen and ZnO undergoes an enhancement process. Preferably, such enhancement includes dissolving this residue in an apolar solvent, filtering off and/or centrifuging the product of such a dissolution, obtaining a solid and a liquid phase, wherein the solid phase is directly used as hydrolysis catalyst and the liquid phase undergoes a removal of the solvent by distillation, which is recycled for the dissolution of further residue, obtaining a paste which is used as raw material for dyes.

[0021] The oily phase undergoes esterification with alcohols. Preferably, such an esterification is carried out in the presence of acids as catalyst. Preferably, the products of the esterification step are purified, preferably by distillation. The residue can be partially recycled to the esterification step.

[0022] Preferably, one or more steps of the inventive process are carried out batchwise.

BRIEF DESCRIPTION OF DRAWINGS

[0023] Further features and advantages of the invention will be anyway clearer from the following detailed description of a preferred embodiment, given by way of example only and not limiting and shown in the annexed drawing, wherein:

fig. 1 is a scheme of a plant, able to carry out the process according to a preferred embodiment of this invention; and

fig. 2 is a flowchart, showing a process according to a preferred embodiment of this invention.

BEST MODE TO CARRY OUT THE INVENTION

[0024] A plant able to carry out the invention according to a preferred embodiment thereof is depicted in fig. 1.

[0025] There are two accesses of matters to be regenerated, namely inlet 1, from which waste lubricants are fed, and inlet 2, from which waste biolubricants, UCO and RUCO are fed.

[0026] Inlet 1 feeds a pre-flash column 3, which removes water possibly present in the waste lubricants. An outlet 4 feeds a decanter 5, from which two outlets -6 and 7- exit. Outlet 6 is the top part of the liquid in the decanter 5 and brings mineral oils to a plant, per se known, for their regeneration 8. The plant 8 is per se known, like the ones present in the premises of the Applicant.

[0027] Outlet 7 feeds a tank 9. An outlet 10 leaves the tank 9, passes through a heat exchanger 11 into a reactor 12. The reactor 12 also receives another feed 13. The reactor 12 is stirred by a stirrer 14 and outputs an outlet 15, which, in turn, feeds another stirred reactor 16, which is fed also by an inlet 17. Part of the product (bottom fraction) is downloaded by a pipe 18 and another part through a pipe 19 which, in turn, feeds a tank 20.

[0028] The content of the tank 20 can reach a reactor 21 through a pipe 22.

[0029] Turning now to the inlet 2, it feeds a tank 23. The contents of the tank 23 can be fed, through a pipe 24, to a heat exchanger 25 and from it to a centrifugal separator 26. Solids, together with some wastewater, separated in the centrifugal separator 26 are brought by means of a pipe 27 to a reactor 28. Liquids are brought by the pipe 29 to another reactor 30, which is preferably heated and stirred, in order to ensure the best conditions for the reaction. The reactor 30 has another inlet 31 and its outlet 32 feeds a centrifugal separator 33. Wastewater leaving the centrifugal separator 33 is fed through a pipe 34 to the reactor 28. The other fraction is fed, through a pipe 35 to another reactor 36, preferably stirred and heated for ensuring appropriate reaction conditions; the reactor 36 is fed also through a pipe 37. After the reaction within the reactor 36, the product leave the reactor 36 through a pipe 38, which, in turn, feeds a centrifugal separator 39, from which an outlet 40 is another feed of the reactor 28. The reactor 28 is fed also by an inlet 41 and has two outlets 42 and 43. Therefore, the reactor 28 is fed by pipes 27, 34, 40 and 41.

[0030] The other fraction coming from the centrifugal separator 39 is brought, through a pipe 44 to a tank 45. The contents of the tank 45 can be brought, through a pipe 46, to the reactor 21. The reactor 21 is fed also by two further inlets, 47 and 48. Therefore, the reactor 21 is fed by pipes 22, 46, 47 and 48.

[0031] An outlet 49 leaves the reactor 21; the outlet 49 is split into a pipe 50 and a pipe 51. The pipe 51 feeds a tank 52. The contents of the tank 52 can be brought by a pipe 53 through a heat exchanger 54 to a packed distillation column 55. This column 55 has two outlets, 56 and 57. The outlet 57 feeds a tank 58. The contents of the tank 58 can be sent by a pipe 59 and through a heat exchanger 60 to a plate distillation column 61, which gives three different outlets, 62, 63 and 64; outlet 63 feeds a tank 65 and outlet 64 feeds a tank 66.

[0032] Another outlet 67 leaves the reactor 21 and feeds a tank 68. The contents of the tank 68 can be fed through a pipe 69 to a reactor 70, preferably stirred and heated, in order to ensure the best reaction conditions. The reactor 70 is fed also by inlets 71 and 72.

[0033] Two outlets, 73 and 74 leave the reactor 70. The outlet 74 feeds a tank 75.

[0034] The process according to the invention will be explained with reference to fig. 2, with further reference to the plant of fig. 1, already described above.

[0035] The inlet 1 leads to a pre-flash column 3, wherein a pre-flash step 76 takes place. Normally, the pre-flash step 76 is carried out at a temperature of 100 to 200 °C, preferably at 130-150 °C, in order to balance a low energy consumption and a good removal of water from the oils. Water is removed through the outlet 77, while the oils to be regenerated go to a subsequent step 78, wherein fatty acids, coming from the oils to be regenerated are recovered. This step 78 takes place in the plant units bearing the numerals 4-7 and 9-20. As an integration of what was reported above, the oils leaving the pre-flash column 3 through the outlet 4 feed a decanter 5, where the light oils - generally mineral oils- of the upper layer are brought, through the outlet 6, to the plant 8 for their regeneration and leave this process. On the contrary, the heavy fraction -generally made up by biolubricants present as soaps- leaves the decanter 5 through the outlet 7 and feeds a tank 9. Therefore, the biological fraction of waste lubricants is got through a pre-flash step (76) of waste lubricants and a subsequent step in a decanter (5), from which such biological fraction is taken from the bottom. Since the process is generally carried out batchwise (because of the non-continuous arrival of waste to be treated), the oils are contained in the tank 9, until the next process is started. At this point, the oils are heated in the heat exchanger 11 up to a temperature ranging from 50 to 110 °C, preferably from 70 to 90 °C, in order to ensure the best reaction conditions. The biolubricants are then fed to the reactor 12, where they are added of sulfuric acid from the feed 13. Sulfuric acid is added from the feed 13 at a concentration of 30 to 98 wt.% and the addition is such that the solution added to the oils in the reactor 12 ranges from 1 to 20 wt.%, preferably from 5 to 10 wt.%; at such a concentration, the hydrolysis and the conversion of soaps into acids is complete. The product exits through the outlet 15 and is washed with water coming from the inlet 17 in the reactor 16. The wastewater is downloaded through the pipe 18, while the product is fed to the tank 20 through the pipe 19.

[0036] Turning now to the part of the process -dealing with UCO, RUCO and biolubricants- starting from the inlet 2, when the process, which is normally carried out batchwise, since the matters to be treated do not arrive continuously, is started, the UCO and RUCO are sent from the tank 23, through the pipe 24 and the heat exchanger 25 to a centrifugation step 79, which takes place firstly in the centrifugal separator 26, where the flow of wastes is centrifuged as such. The centrifugation in the centrifugal separator 26 takes place at a temperature ranging from 50 to 110 °C, preferably from 70 to 90 °C and with a stirring of 2,000 to 10,000 rpm. The preliminary treatment 79 of UCO and RUCO consists of a centrifugation step, separating two fractions: a liquid one and one containing wastewater and solids. In fact, preferably more stages of centrifugation 79 are provided, in order to have a better separation.

[0037] The liquids undergo a washing in the reactor 30. Distilled water, coming from the inlet 31, is added at a concentration of 1 to 20 wt.%, preferably from 5 to 10 wt.%. The centrifugation step 79 continues in the centrifugal separator 33, at a temperature ranging from 50 to 110 °C, preferably from 70 to 90 °C and with a stirring of 2,000 to 10,000 rpm. Wastewater and solids, containing rubbers which are not water soluble, are removed through the pipe 34, while the matters to be regenerated are fed to the reactor 36, where 1 to 20 wt.%, preferably 5 to 10 wt.% of an aqueous solution of H_3PO_4, generally at a concentration of 30 to 80 wt.%, is added by the pipe 37. The reaction product is fed to the last centrifugal separator 39 at a temperature ranging from 50 to 110 °C, preferably from 70 to 90 °C and with a stirring of 2,000 to 10,000 rpm. Wastewater and solids are removed through the outlet 40 and the matter to continue the treatment is fed, through a pipe 44 to the tank 45, completing the step 79.

[0038] Wastewater and solids fed to the reactor 28 are acid, so that they are neutralised with a strong base, like CaOH, NH_3 or NaOH; wastewater coming from the outlet 42 is sent to the water purification and phosphate-based fertilisers, which are a valuable co-product, are then recovered from the outlet 43.

[0039] Matters coming from the preliminary steps are contained in tanks 20 and 45. They are sent from the said tanks 20 and 45 to the reactor 21, where they are combined and where a hydrolysis step 80 takes place. They are added with ZnO which acts as a catalyst (from the inlet 47) and water (from the inlet 48). Preferably, ZnO is added at 1 to 5 wt.%, preferably at 2 wt.% and water is added in order to be 20 to 40 wt.%, preferably 30 wt.%. The hydrolysis takes preferably place at 150-250 °C and 10-20 bar for 6 to 18 hours. The products of the hydrolysis step 80 are an aqueous phase, containing raw glycerine, and an oily phase, mainly containing fatty acids. Such product of hydrolysis is splitted into two flows, one bringing glycerine to be purified, which is another valuable co-product, and the other one is purified and esterified.

[0040] Part of the products -the aqueous phase- go, through the outlet 49, to a step 81, where glycerine is purified. At this stage, a second hydrolysis step can take place in the reactor 21, under the same conditions of the first one. The pipe 50 downloads bitumen as a byproduct, while the rest of this fraction is sent to the tank 52, from where the purification step 81 starts. Flash distillation takes place in the column 55, normally at a temperature of 130-150 °C and a pressure of 0.5-1.0 bar. The head fraction is sent to the water treatment facility and the tail in the outlet 57 is then sent to the column 61, where temperature is 250-350 °C at the bottom and 30 to 60 °C at the head and pressure is 0.0130-0.0150 at the bottom and 0.002 to 0.005 at the head, from where wastewater exits from the outlet 62, glycerine from the outlet 63 and is stored in the tank 65 and bitumen through the outlet 64 and is stored in the tank 66, both for delivery and subsequent sale.

[0041] The other part of the product is temporarily stored in the tank 68, after dehydration -which can take place under stirring at 130-200 °C for 1-2 hours- from where, after a conventional purification step 82 -preferably distillation-, it passes to an esterification step 83. Such a distillation is normally carried out at a pressure of 6.6×10^{-3} to 0.0020 bar and is normally stopped when the boiling point, referred to atmospheric pressure, is 405°C, normally with a residence time of 3-6 hours. The distillation residues contain mainly bitumen and ZnO. ZnO is recovered and can easily be reused. The bitumen can be added to the other bitumen fractions obtained. The esterification takes place batchwise in the reactor 70, where the acids arrive through the pipe 69 from the tank 68, where (vide infra) also a recycle is added. Also some weak acids, like pelargonic or adipic acids, are added as co-reactants from the inlet 72 and alcohols, like n-decanol or trimethylolpropane (TMP) are added in a stoichiometric amount through the inlet 71. Wastewater leaves the plant through the outlet 73 and goes to the water treatment plant, while raw biolubricants leave through the outlet 74 to a purification step 84 and subsequently to the tank 75, where they are stored for the delivery and sale. A recycle of fatty acids taken from wastewater in 73 takes place through a pipe 85 to the tank 68. The fatty acids are distilled at 6.6×10^{-3} to 0.0020 bar, stopping the distillation as soon as the flow rate dramatically drops, normally after 3 to 6 hours.

[0042] According to a preferred, alternative embodiment, the bitumen coming from the distillation residue of the step 82 can undergo a process for the enhancement of the organic molecules therein contained. In this case, instead of mixing this residue to the other bitumen obtained in other steps of this process or even in other processes, taking place in the same industrial site, this residue is dissolved in an apolar solvent, like a saturated, linear hydrocarbons, such as n-pentane, n-hexane, n-heptane, n-octane, n-nonane, n-decane. The solution obtained is stirred for 1 to 4 hours. The solution is then filtered off and/or centrifuged and two phases are obtained, one being solid and the other being liquid. The solid part contains ZnO and can directly be used as catalyst in the reactor 21 without any further purification, in the hydrolysis step 80, so reducing the consumption of fresh catalyst, with a cost saving. As for the liquid part, the solvent can be removed by distillation and recycled to the first step of this part of the process and the paste remaining can be used as raw material for the production of dyes.

[0043] The present invention allows to combine two processes for the regeneration of different kind of oils

-namely lubricants on one hand and UCO and RUCO on the other hand-, providing a useful product and avoiding wastes which are not wastewater, with energy costs limited and it is therefore surely part of the so called circular economy.

[0044] It is however understood that the invention should not be seen as limited by the above arrangement, which is only an exemplary embodiment thereof, but that a number of variants is possible, all at reach of the skilled person, without departing from the scope of the invention itself, as defined by the appended claims.

LIST OF REFERENCE NUMERALS

[0045]

1	Inlet
2	Inlet
3	Pre-flash column
4	Outlet
5	Decanter
6	Outlet
7	Outlet
8	Regeneration plant
9	Tank
10	Outlet
11	Heat exchanger
12	Reactor
13	Feed
14	Stirrer
15	Outlet
16	Reactor
17	Inlet
18	Pipe
19	Pipe
20	Tank
21	Reactor
22	Pipe
23	Tank
24	Pipe
25	Heat exchanger
26	Centrifugal separator
27	Pipe
28	Reactor
29	Pipe
30	Reactor
31	Inlet
32	Outlet
33	Centrifugal separator
34	Pipe
35	Pipe
36	Reactor
37	Pipe
38	Pipe
39	Centrifugal separator
40	Outlet
41	Inlet
42	Outlet
43	Outlet
44	Pipe
45	Tank
46	Pipe
47	Inlet
48	Inlet
49	Outlet
50	Pipe
51	Pipe
52	Tank
53	Pipe
54	Heat exchanger
55	Pre-flash column
56	Outlet
57	Outlet
58	Tank
59	Pipe
60	Heat exchanger
61	Plate distillation column
62	Outlet
63	Outlet
64	Outlet
65	Tank
66	Tank
67	Outlet
68	Tank
69	Pipe
70	Reactor
71	Inlet
72	Inlet
73	Outlet
74	Outlet
75	Tank
76	Pre-flash step
77	Outlet
78	Step of regeneration and recovery of fatty acids
79	Centrifugation step
80	Hydrolysis step
81	Step of glycerine purification
82	Purification step
83	Esterification step
84	Purification step
85	Pipe

Claims

1. Process for the production of biolubricants from waste materials, wherein said waste materials are waste biolubricants, UCO, RUCO and a biological fraction of waste lubricants, **characterised in that** two lines (22, 46) are combined for a subsequent reaction (80) after two separate preliminary treatments (76, 78; 79), wherein the first line (46) brings waste biolubricants, UCO and RUCO which have been processed (79) and the second line (22) brings a biological fraction of lubricants separated (76, 78) from waste lubricants.

2. Process as claimed in claim 1), **characterised in that** the preliminary treatment (79) of UCO and RUCO consists of a centrifugation step, separating two fractions: a liquid one and one containing wastewater and solids.

3. Process as claimed in claim 2), **characterised in that** more stages of centrifugation (79) are provided.

4. Process as claimed in claim 3), **characterised in that** between two centrifugation stages distilled water and/or an aqueous solution of H_3PO_4 are added to the liquid fraction under treatment.

5. Process as claimed in any claim 2) to 4), **characterised in that** the fraction containing wastewater and solids is neutralised with a strong base, like CaOH, NH_3 or NaOH.

6. Process as claimed in any previous claim, **characterised in that** the biological fraction of waste lubricants is got through a pre-flash step (76) of waste lubricants and a subsequent step in a decanter (5), from which such biological fraction is taken from the bottom.

7. Process as claimed in claim 6), **characterised in that** the biological fraction taken from the bottom of the decanter (5) is reacted with sulfuric acid.

8. Process as claimed in any previous claim, **characterised in that** the liquids having undergone the preliminary treatments are combined in a reactor (21) where a hydrolysis reaction (80) takes place.

9. Process as claimed in claim 8), **characterised in that** the matters entering the reactor (21) are added with ZnO (from the inlet 47) and water (from the inlet 48) at 150-250 °C and 10-20 bars.

10. Process as claimed in claim 9), **characterised in that** ZnO is added at 1 to 5 wt.% and water is added in order to be 20 to 40 wt.%.

11. Process as claimed in any claim 6 to 10), **characterised in that** the product of hydrolysis is splitted into two flows, one containing glycerine to be purified and the other one is purified and esterified.

12. Process as claimed in any previous claim, **characterised in that** one or more steps are carried out batchwise.

13. Process as in any claim 11) or 12), **characterised in that** the bitumen coming from the distillation residue of the said purification step (82) of the product coming from a plate distillation column (61) undergoes a process for the enhancement of the organic molecules, wherein such residue is dissolved in an apolar solvent, the obtained solution is stirred, then is filtered off and/or centrifuged to separate catalyst and a raw material for dyes.

Amended claims in accordance with Rule 137(2) EPC.

1. Process for the production of biolubricants from waste materials, wherein said waste materials are waste biolubricants, UCO, RUCO and a biological fraction of waste lubricants, wherein two lines (22, 46) are combined for a subsequent reaction (80) after two separate preliminary treatments, wherein the first line (46) brings waste biolubricants, UCO and RUCO which have been processed and the second line (22) brings a biological fraction of lubricants separated from waste lubricants, wherein the biological fraction of waste lubricants is got through a pre-flash step (76) of waste lubricants and a subsequent step in a decanter (5), from which such biological fraction is taken from the bottom and wherein the product of hydrolysis is split into two flows, an aqueous phase, containing raw glycerine, and an oily phase, mainly containing fatty acids **characterised in that** bitumen coming from distillation residue of a purification step (82) of product coming from a plate distillation column (61) undergoes a process for the enhancement of the organic molecules, wherein such residue is dissolved in an apolar solvent, the obtained solution is stirred, then is filtered off and/or centrifuged to separate catalyst and a raw material for dyes.

2. Process as claimed in claim 1), **characterised in that** the preliminary treatment (79) of UCO and RUCO consists of a centrifugation step, separating two fractions: a liquid one and one containing wastewater and solids.

3. Process as claimed in claim 2), **characterised in that** more stages of centrifugation (79) are provided.

4. Process as claimed in claim 3), **characterised in that** between two centrifugation stages distilled water and/or an aqueous solution of H_3PO_4 are added to the liquid fraction under treatment.

5. Process as claimed in any claim 2) to 4), **characterised in that** the fraction containing wastewater and solids is neutralised with a strong base, like CaOH, NH_3 or NaOH.

6. Process as claimed in any previous claim, **characterised in that** the biological fraction taken from the bottom of the decanter (5) is reacted with sulfuric acid.

7. Process as claimed in any previous claim, **characterised in that** liquids having undergone the preliminary treatments are combined in a reactor (21) where a hydrolysis reaction (80) takes place.

8. Process as claimed in claim 7), **characterised in that** the matters entering the reactor (21) are added with ZnO (from the inlet 47) and water (from the inlet 48) at 150-250 °C and 10-20 bars.

9. Process as claimed in claim 8), **characterised in that** ZnO is added at 1 to 5 wt.% and water is added in order to be 20 to 40 wt.%.

10. Process as claimed in any previous claim, **characterised in that** one or more steps are carried out batchwise.

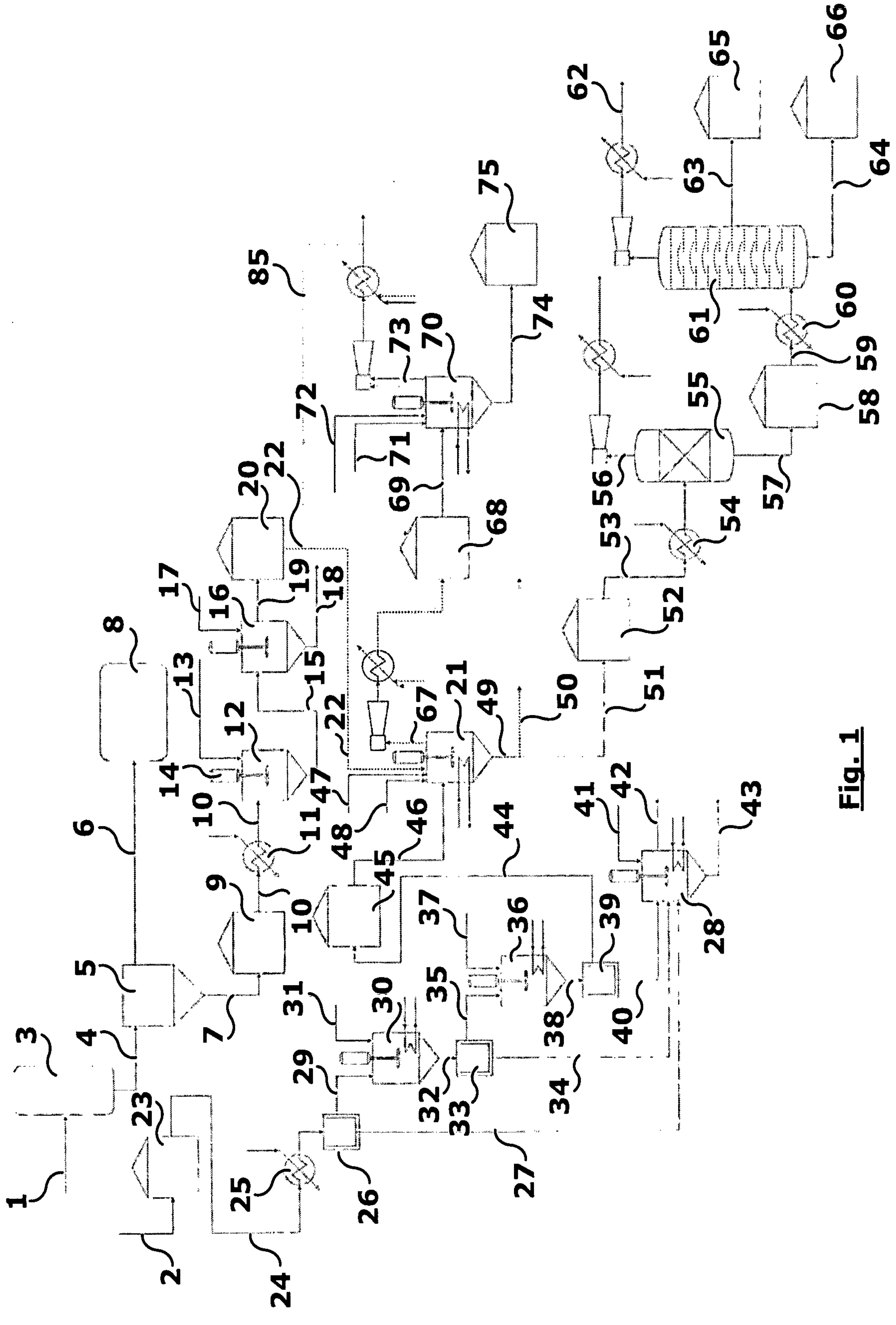
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Fig. 1

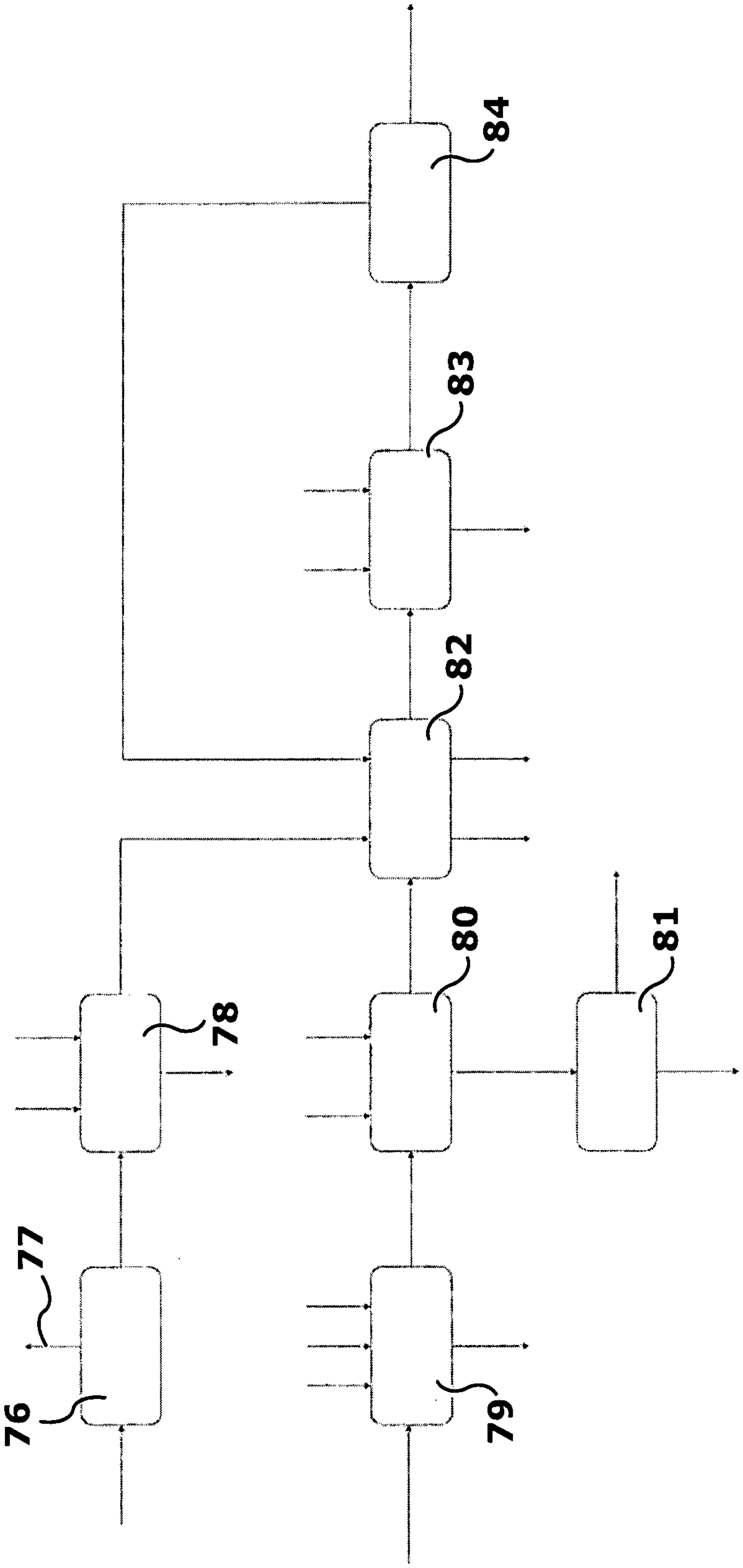

Fig. 2

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EUROPEAN SEARCH REPORT

Application Number
EP 23 42 5054

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
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Y A	CÁRDENAS JULIANA ET AL: "Pre-treatment of used cooking oils for the production of green chemicals: A review", JOURNAL OF CLEANER PRODUCTION, ELSEVIER, AMSTERDAM, NL, vol. 289, 17 November 2020 (2020-11-17), XP086495277, ISSN: 0959-6526, DOI: 10.1016/J.JCLEPRO.2020.125129 [retrieved on 2020-11-17] * abstract * * paragraph [3.2.1] *	1-7 13	
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The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
Munich	6 March 2024	Pöllmann, Klaus

CATEGORY OF CITED DOCUMENTS

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