

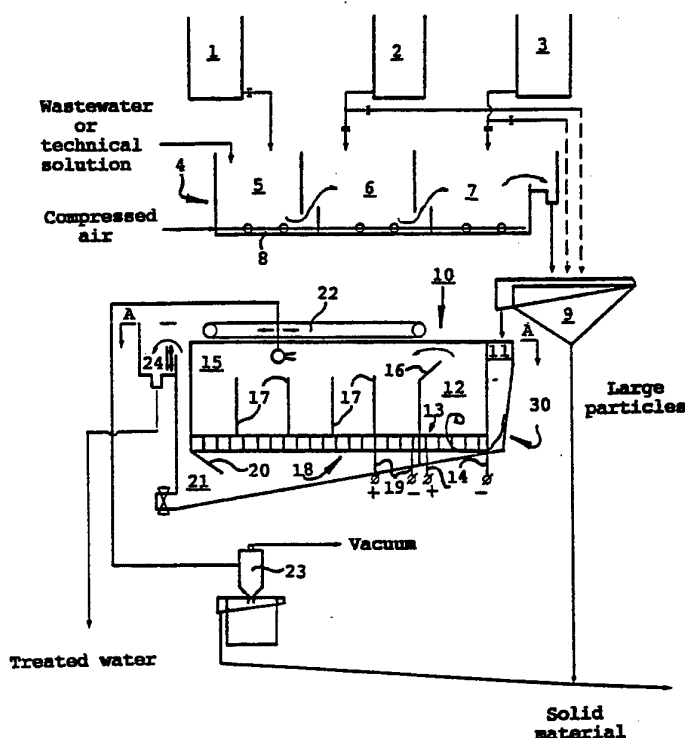


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(21) International Application Number: PCT/FI93/00485 (22) International Filing Date: 17 November 1993 (17.11.93) (30) Priority data: 925204 17 November 1992 (17.11.92) FI (71) Applicant (for all designated States except US): TURUN EKOVESI OY [FI/FI]; Virusmäentie 65, FIN-20300 Turku (FI). (72) Inventor; and (75) Inventor/Applicant (for US only) : NENNO, Vladimir Erosovits [MD/MD]; 1. per. Panfilova, 17-13, Kishinev, 277008 (MD). (74) Agent: TURUN PATENTTITOIMISTO OY; P.O. Box 99, FIN-20521 Turku (FI).		(81) Designated States: AT, AU, BB, BG, BR, BY, CA, CH, CZ, DE, DK, ES, FI, GB, HU, JP, KP, KR, KZ, LK, LU, LV, MG, MN, MW, NL, NO, NZ, PL, PT, RO, RU, SD, SE, SK, UA, US, VN, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG). Published <i>With international search report.</i> <i>In English translation (filed in Finnish).</i>

(54) Title: METHOD AND DEVICE FOR PURIFICATION OF AQUEOUS SOLUTIONS BY ELECTROFLOTATION**(57) Abstract**

The object of the invention is a method and device for purifying aqueous solutions or the like, where the component to be removed, for example in a dissolved or solid state, is converted to the solid phase, the said method including the addition of coagulating and flocculating agents; the separation of larger solid particles from the suspension thus obtained by settling; and the removal of the remaining components from the suspension by electroflotation. The invention is intended especially for purifying water and technical solutions of different types of dissolved and solid substances. In the method, electroflotation is used to remove the remaining components by saturating the suspension with the gas released in the electrolysis in connection with turbulence and by removing the foam in laminar conditions. In the electroflotation device (10) the chamber (30) consists of a gas saturation chamber (12) in which is mounted an electrode unit (13) for saturating the suspension with the gas released in the electrolysis, and of a flotation compartment (15), in which is mounted an electrode unit (18) for foaming.



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METHOD AND DEVICE FOR PURIFICATION OF AQUEOUS
SOLUTIONS BY ELECTROFLOTATION

- The object of the invention is a method for purifying aqueous solutions or the like, where the component to be removed, for example in a dissolved or solid state, is converted to the solid phase, the said method including the
- 5 addition of a precipitant, coagulating agents, and/or flocculating agents; the separation of larger solid particles from the suspension thus obtained by settling; and the removal of the remaining components from the suspension by electroflotation.
- 10 The object of the invention also comprises an electroflotation device for purifying aqueous solutions or the like, consisting of a chamber, filling and discharge compartments, a foam removal device and electrode units in which the electrodes are parallel sheet elements, for
- 15 carrying out the method relating to the invention.

- Known water purification methods include a purification process carried out by using coagulating and flocculating agents, where the reagents are introduced into the liquid to be purified in solution form, as disclosed in Finnish
- 20 patent application no. 890493. Finnish patent no. 77 008 discloses a method for carrying out a purification process using coagulating agents, in which method coagulating agents are obtained through the anodic dissolution of aluminium sheets.
- 25 The disadvantages common to the above processes include the low capacity of the devices which separate the flocculants formed from the liquid flow, the considerable losses of liquid in connection with the removal of the coagulating agent that has risen to the surface or settled on the
- 30 bottom - which usually requires additional measures for removing excess water - and also the need to collect the

sediment at different points, for example, from both the bottom and surface of a sedimentation basin. When it is in addition considered that the consumption of coagulating agent amounts to 20 - 200 g per cubic metre of liquid to be
5 purified, it is obvious that the slowness of the separation process and the high loss of anode metal - due to the fact that in connection with the electrochemical dissolution of the anodes (FI patent no. 77 008), aluminium transfers from the metal sheets to the sediment of the hydroxide formed in
10 the reaction - make the process uncompetitive where the required purification capacity exceeds 10 m³/h.

US patent no. 4 156 648 discloses a solution for an apparatus for the utilisation of the above methods. The publication describes a process diagram and apparatus for
15 treating water to remove grit by using centrifugal force to remove solid suspension particles, and coagulation and flocculation to sediment large flocculants and suspensions into a thin layer in a sedimentation basin. Saturation with a gas is followed by foaming with a gas in connection with
20 pressurization and simultaneous oxidization of organic matter with ozone introduced into the flotation tank.

The disadvantages of the method disclosed in the US patent publication no. 4 156 648 include the fact that it comprises several stages, the use of separation processes
25 involving high-speed equipment (centrifugation) and the use of pressure processes (compression flotation) which require special equipment. The use of special flotation equipment to maintain and carry the suspension in the foam and the high water content of the foam - requiring further
30 saturation with solid particles and removal of water - must also be considered disadvantages of the said method.

The aim and object of the present invention is to achieve a new method and device for eliminating the disadvantages of the above-mentioned known purification processes, making
35 the process more efficient and increasing the suspension

separation capacity.

It is characteristic of the method relating to the invention that the removal of the remaining components is carried out according to the following stages:

- 5 - by electroflotation at the first stage of the method, the suspension is saturated in a turbulence process with the bubbles of electrolytic gases, the density of the electric current exceeding 20 mA/cm², and
- at the second stage, the foam is separated by the laminar
10 flow of a liquid, the density of the electric current being below 20 mA/cm².

It is also characteristic of the invention that a precipitant is added to the aqueous solution, the said precipitant being a substance that dissolves in water or a
15 fine sorbent or a combination of these. Suitable dissolving precipitants include saline solutions such as iron oxide sulphates which, together with the component to be removed, form components insoluble in water. Activated carbon, for example, is a suitable sorbent.

- 20 It is characteristic of the device relating to the invention that the chamber comprises

- a gas saturation chamber, in which is mounted an electrode unit for saturating the suspension with the gas released during the electrolysis,
- 25 - a flotation compartment in which is mounted an electrode unit for flotation,
- a suspension flow suppressor of a multicellular structure mounted above the electrode unit in the flotation compartment, and

- an adjustable separating wall installed between the gas saturation chamber and the flotation compartment.

By means of the invention a method and device are achieved which can be used for purifying the wastewaters of
5 foodstuff, chemical, varnish and paint production plants, or for treatment of the technical solutions from mining chemistry and metallurgical plants, these solutions containing small amounts of useful recoverable components and, for example, heavy metal salts.

10 The invention is described in the following with examples, with reference to the appended drawings in which

Figure 1 shows the purification method relating to the invention as a block diagram;

15 Figure 2 shows diagrammatically the purification process of figure 1 and the purification device relating to the invention;

Figure 3 shows a cross-sectional view of the electroflotation device relating to figure 2, along line A-A and as seen from above.

20 Figure 1 shows the purification method relating to the invention as a block diagram. The method for purifying water and technical solutions of different types of dissolved and solid components is carried out as follows:
25 to the flow of water or a technical solution is added a saline solution acting as a precipitant, which together with the component to be removed forms insoluble components which settle as a sediment. Instead of, or together with, the precipitant solution, fine-dispersed sorbent can be added to the mixture, the said sorbent sorbing the compound
30 to be separated from the aqueous solution or the ion of a useful component. The suspension formed is treated with coagulating and flocculating agents. Solid suspensions are removed by precipitation and settling, and the fine-dispersed solid phase is removed by electrofoaming, that

is, electroflotation.

According to a preferred embodiment of the invention, the electroflotation process is carried out in two stages combined in the same device. At the first stage, the suspension is saturated with the bubbles of electrolytic gases which are separated in the turbulence process of the suspension being purified, due to the high density of the electric current which is well in excess of 20 mA/cm². At the second stage, the foam is separated in the foaming compartment of the chamber, the laminar flow of the liquid and the density of the electric current being minimal, significantly below 20 mA/cm².

Figure 2 shows diagrammatically the purification process of figure 1 and the structure of the purification device relating to the invention, by means of which the method shown in figure 1 is carried out. The purification device used in the purification process consists basically of reagent dosage units 1, 2 and 3, a mixer 4, a clarifier 5, tank spaces 6 and 7, a clarifier and sedimenter 9, an electroflotation device 10, a foam removal device 22, and a foam separator 23.

In the chamber 30 of the electroflotation device 10 is built a filling compartment 11, a gas saturation chamber 12 and a flotation compartment 15. The gas saturation space 12 is separated from the foaming or flotation compartment 15 by means of a separating wall 16. In the flotation compartment 15 is mounted a suspension flow suppressor 17 built as a multicellular structure. The lower part of the discharge device 21 for purified liquid is separated from the lower part of the flotation compartment 15 by means of a separating wall 20. The vertically movable shutter flap 24 in the top part of the discharge device 21 regulates the area of the liquid to be purified in the electroflotation device 10. The electrode units 13 and 18 of the gas generators are built in the form of groups of flat parallel

sheets, which are mounted in the vertical plane with respect to the direction of flow of the liquid, above the bottom of the electroflotation device 10.

Power supply from the power source to the electrode units 5 13 and 18 is by current conductors fastened to rails 14 and 19. The current supplied from the power source is pulsating, as a result of which the polarisation of the electric current can be inverted. The aim is to prevent the premature passivation of the electrodes and to achieve an 10 even, efficient generation of gas. The material used for the sheet-like electrodes is, for example, stainless steel, which is a virtually insoluble material. Commonly used electrodes made from other corrosion-resistant materials, such as platinum, acid-resistant steel or platinated 15 titanium, can also be used.

The purification of aqueous or technical solutions takes place as follows: wastewater is fed into the mixer 4; at the same time fine-dispersed sorbent and/or saline solution, and/or coagulating and flocculating agents 20 precipitating the component to be removed are fed alternately into the clarifier 5 and tank spaces 6 and 7 from the reagent dosage units 1, 2 and 3. The suspension formed is mixed in the mixer 4 by means of compressed air supplied along feeder line 8, through the perforated hose 25 fixed on the bottom of the mixer 4. The pretreated suspension is fed into the sedimenter 9 in which solid suspension parts and large flocculants are separated from it and removed in stages through the bottom of the sedimenter 9. If necessary, more coagulating and 30 flocculating agents can be fed from the dosage units 2 and 3 into the sedimenter 9.

From the sedimenter 9 the suspension, from which the largest particles have already been separated, is fed with its components to be removed, dissolved and/or fine- 35 dispersed components, into the receiving compartment 11 of

the chamber 30 of the electroflotation device 10. From there the suspension solution passes from the spaces between the sheets of the electrode unit 13 into the gas saturator 12. In the gas saturator 12, the electrodes of the electrode unit 13 are partly or completely immersed in the liquid. The first stage of the process, that is, the intensive release of gas takes place in the electrodes 13 of the gas saturator 12, the gas bubbles rising above the electrode unit 13 creating strong turbulence in the liquid. The adjustable separating wall 16 separating the gas saturator 12 and the flotation compartment 15 from each other, essentially adjustable by $1/3 - 5/6$, partly prevents the discharge of foam from the gas saturator 12. In space 12 the turbulence of the suspension flow intensifies when the discharge duct is completely or partly closed by the inclined, adjustable separating wall 16.

The suspension saturated with the bubbles of the electrolysis gases is led to the flotation compartment 15 of the electroflotation device 10. The so-called basic mass of the suspension rises to the surface of the liquid after the treatment of the suspension in the gas saturator 12 and the solid particles remaining in the suspension move into the foam due to the gas bubbles created in the electrode unit 18 of the flotation compartment 15. The flow of the suspension in the flotation compartment 15 is laminar flow and the densities of the electric current are lower than in the gas saturator 12. The laminarity of the suspension flow is guaranteed by suppressor gratings 17 mounted above the electrode unit 18.

In the flotation compartment 15, the foam deposited on the surface of the liquid is removed from the process by the vacuum foam remover 22 which removes periodically downstream and upstream the top surface of the foam, which has a low water content. The periodic nature of the foam removal is regulated by the operating frequency of the foam removal device 22. The solid components are separated from

the foam by means of a vacuum, with a foam separator 23. From the flotation compartment the water - from which solid components have been removed as described above - discharges completely or partly through the spaces in the electrode unit 18 in the liquid and through the discharge device 21. The discharge device 21 is equipped with a suspension flow separator 20 which prevents the entry of solid components into the purified liquid flow discharging from the electroflotation device 10. In the electroflotation device 10 the height of the suspension surface level is regulated by means of a shutter flap 24.

Figure 3 shows a cross-sectional view of the electroflotation device 10 relating to figure 2, along line A-A and as seen from above. From the sedimenter the suspension, from which the largest particles have been removed, is fed with the components to be removed into the filling compartment 11 of the electroflotation device 10. From there the suspension liquid passes through the spaces between the sheets of the electrode unit into the gas saturator 12. In the gas saturator 12 the gas bubbles released from the electrode unit create strong turbulence in the solution. The turbulence of the suspension flow intensifies when the discharge duct is partly closed with the inclined separating wall 16. The suspension saturated with the bubbles of the electrolysis gases is led to the flotation compartment 15. The so-called basic mass of the suspension rises to the surface of the liquid after the treatment of the suspension in the gas saturator 12 and the solid particles remaining in the suspension pass into the foam due to the gas bubbles created in the electrode unit of the flotation compartment 15. The flow of the suspension is laminar flow and the densities of the electric current are lower than in the gas saturator 12. The laminarity of the suspension flow is guaranteed by suppressor gratings 17 mounted above the electrode unit. The purified water is discharged through the sheets in the electrode unit and through the discharge device 21. The discharge device 21 is

equipped with a suspension flow separator which prevents the entry of solid components into the purified liquid flow discharging from the electroflotation device 10. In device 10 the height of the suspension surface level is regulated
5 by means of a shutter flap 24.

According to a preferred embodiment, the purification method and device relating to the invention were used to purify the wastewaters of a meat-processing plant. The plant's wastewater was fed into the mixer 4, the water
10 containing, for example, 750 mg/l of suspension, 44 mg/l of common phosphorus, the biological oxygen demand (BOD) of the wastewater being 1000 mg O_2 /l. From dosage unit 1 was fed iron oxide sulphate solution in the amount of 120 g/m³, including salt, which acted as the precipitant for
15 phosphorus. From dosage unit 2 was fed aluminium sulphate coagulating agent solution in the amount of 30 g/m³, and from dosage unit 3 flocculating agent solution, polyacryl amide, in the amount of 4 g/m³.

After mixing, the suspension formed in the mixer 4 was fed
20 into the sedimenter 9, where the solid suspension settled on the bottom and was removed. From the sedimenter 9 the suspension containing the components to be removed was conducted to the electroflotation device 10, in which the saturation of the suspension with the gas bubbles released
25 in the electrolysis took place in the saturation space 12, the density of the electric current being 40 mA/cm². The solid phase part rose onto the surface of the suspension immediately on being discharged from the gas saturator 12.

The suspension was further led to the flotation compartment
30 15, where the final purification of the wastewater took place, the density of the electric current being 10 mA/cm². The purified water was removed from the device through discharge device 21. The waste material collected in the foam in the flotation compartment and was removed by the
35 foam removal device 22 as necessary. The foam yield did not

exceed 8.0% and its solid phase content was 2.2%.

Measurements gave the following specific values for the purified water:

- suspended components 44 - 60 mg/l
- 5 - common phosphorus content 1.1 - 2.1 mg/l
- BOD 350 - 390 mg/l

The degree of purification was 93% when calculated on the basis of the suspended components, and 96% when calculated on the basis of phosphorus.

- 10 According to a second preferred embodiment, the purification method and device relating to the invention were used for purifying oil-containing wastewaters. The oil-containing starting wastewater, containing 360 mg of oil/l, was fed into a mixer 4, into which was also dosed,
- 15 in consecutive order, finely ground activated carbon in the amount of 500 g/m³, aluminium sulphate coagulating agent in the amount of 50 g/m³, and polyacryl amide flocculating agent in the amount of 2 g/m³. After mixing, the suspension was fed into the electroflotation device 10 in which gas
- 20 saturation was carried out with the density of the electric current being 40 mA/cm², and foaming with the density of the electric current being 8 mA/cm². After the process the oil content of the purified water did not exceed 1.0 mg/l. Foam yield was between 0.7 - 1.2%.
- 25 According to a third preferred embodiment, the purification method and device were used for purifying the wastewaters of a leather manufacturing plant of chromium and other suspended substances. The wastewaters of the leather manufacturing plant contained 480 mg/l of suspended
- 30 substances, 230 mg/l of sulphides, 160 mg/l of common chromium, and the BOD was 1280 mg O₂/l. The wastewater was fed into the mixer 4, into which was dosed 280 g/m³ iron monoxide sulphate to sediment the sulphides and to regenerate the chromium ions. Into the mixer was also fed

aluminium sulphate coagulating agent and polyacryl amide flocculating agent in the respective amounts of 100 g/m^3 and 4 g/m^3 . The suspension thus pre-prepared was fed into the sedimentation tank 9, and after the sedimentation tank 9
5 the discharged material was led to the electroflotation device.

The solid phase of the suspension was separated in the gas saturator 12 with the density of the electric current being 30 mA/cm^2 , and in the flotation compartment 15 with the
10 density being 8 mA/cm^2 . The purified water contained 35 mg/l of suspended substances, less than 2.0 mg/l of sulphides, less than 0.1 mg/l of common chromium, and the BOD was $240 \text{ mg O}_2/\text{l}$.

According to a fourth preferred embodiment, the
15 purification method and device relating to the invention were used for removing heavy metal ions from the wastewaters of a refinery and/or chemical plant. According to the analysis, the starting solution contained 0.7 mg/l of silver, 4.8 mg/l of lead and 21 mg/l of copper. Sodium
20 sulphide in the amount of 35 g/m^3 was used as precipitant. The fine-dispersed heavy metal sulphide sediment settled on the bottom of the suspension tank was flocculated with polyacryl amide having a content of 1.0 g/m^3 . The removal of the solid phase from the suspension was carried out in the
25 gas saturator 12 with the density of the electric current being 20 mA/cm^2 , and in the flotation compartment 15 with the density of the electric current being 5 mA/cm^2 . There were practically no ions of silver or lead in the purified technical solution, and the content of copper ions did not
30 exceed 0.1 mg/l . The foam yield was 0.05% .

It is obvious to one skilled in the art that the different embodiments of the invention may vary within the scope of the claims presented below.

CLAIMS

1. A method for purifying aqueous solutions or the like, where the component to be removed, for example in a dissolved or solid state, is converted to the solid phase, the said method including the addition of a precipitant, coagulating agents and/or flocculating agents; the separation of larger solid particles from the suspension thus obtained by settling; and the removal of the remaining components from the suspension by electroflotation, characterized in that the removal of the remaining components is carried out according to the following stages:
- by electroflotation at the first stage of the method, the suspension is saturated in a turbulence process with the bubbles of electrolytic gases, the density of the electric current exceeding 20 mA/cm², and
 - at the second stage, the foam is separated by the laminar flow of a liquid, the density of the electric current being below 20 mA/cm².
2. A method as claimed in claim 1, characterized in that in electroflotation, the electrodes in the electrode units are fastened to rails, to which a pulsating current with invertible polarisation is conducted from a power source, in order to prevent the passivation of the electrodes.
3. A method as claimed in claim 1, characterized in that a precipitant is added to the aqueous solution, the said precipitant being a substance that dissolves in water or a fine sorbent or a combination of these.
4. An electroflotation device (10) for purifying aqueous solutions or the like, consisting of a chamber (30), filling and discharge compartments (11, 21), a foam removal device (22, 23) and electrode units (13, 18) in which the

electrodes are parallel sheet elements, for carrying out the method as claimed in method claims 1, 2 or 3, characterized in that the chamber (30) comprises

- a gas saturation chamber (12), in which is mounted an
5 electrode unit (13) for saturating the suspension with the gas released during the electrolysis,

- a flotation compartment (15) in which is mounted an electrode unit (18) for flotation,

- a suspension flow suppressor (17) of a multicellular
10 structure mounted above the electrode unit (18) in the flotation compartment (15), and

- an adjustable separating wall (16) installed between the gas saturation chamber (12) and the flotation compartment (15).

15 5. An electroflotation device (10) as claimed in claim 4, characterized in that the electrodes in the electrode units (13, 18) are fastened to rails (14, 19), to which a pulsating current with invertible polarisation can be conducted from a power source, in order to prevent the
20 passivation of the electrodes.

6. An electroflotation device (10) as claimed in claim 4 or 5, characterized in that the electrode units (13, 18) are made mainly of insoluble material, such as stainless steel or a similar material.

25 7. An electroflotation device (10) as claimed in claim 4, 5 or 6, characterized in that the electrodes in the electrode units (13, 18) are mounted in the vertical plane with respect to the direction of flow of the suspension to be purified, above the bottom of the chamber (30) of the
30 electroflotation device (10).

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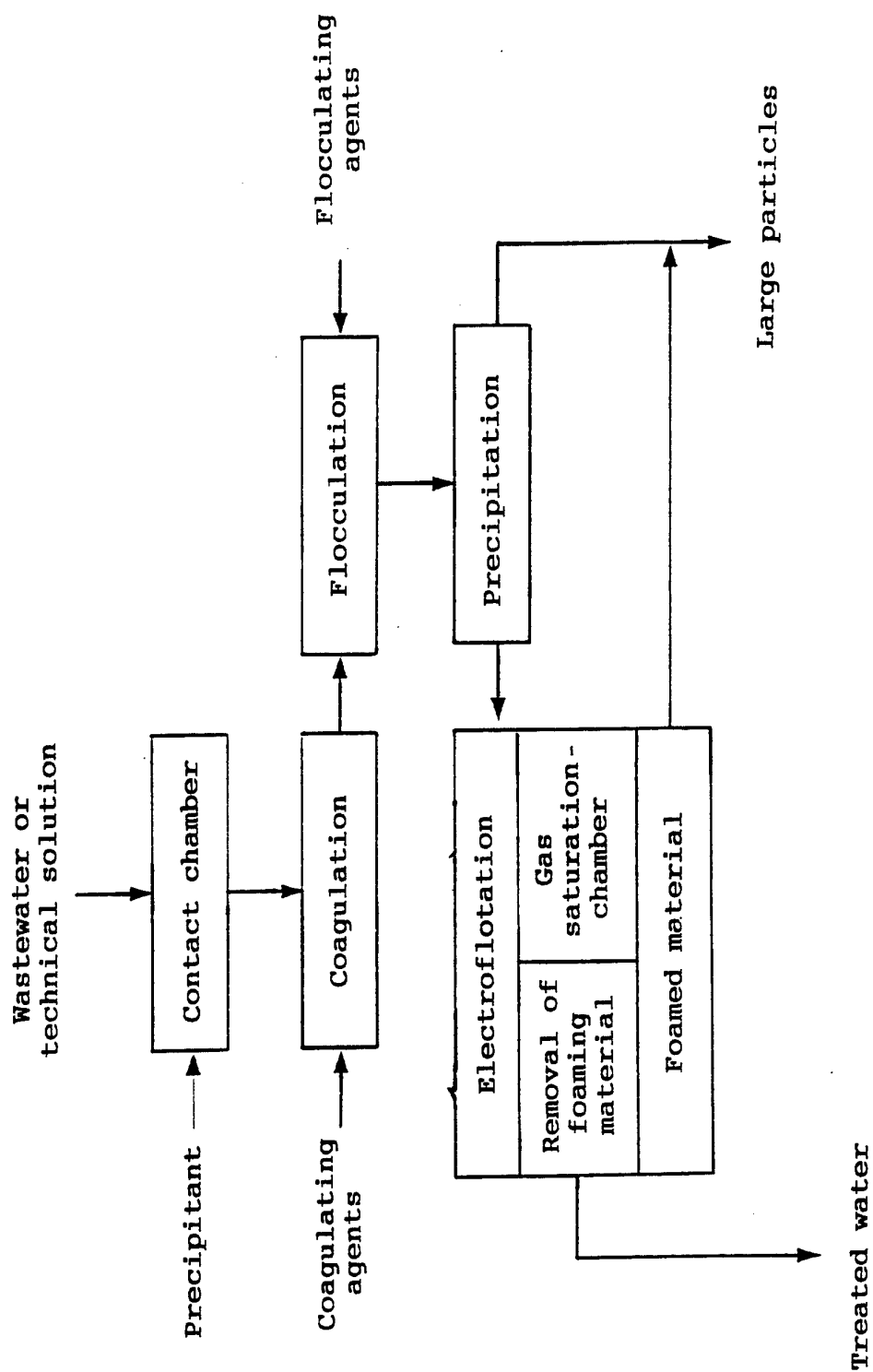


FIG. 1

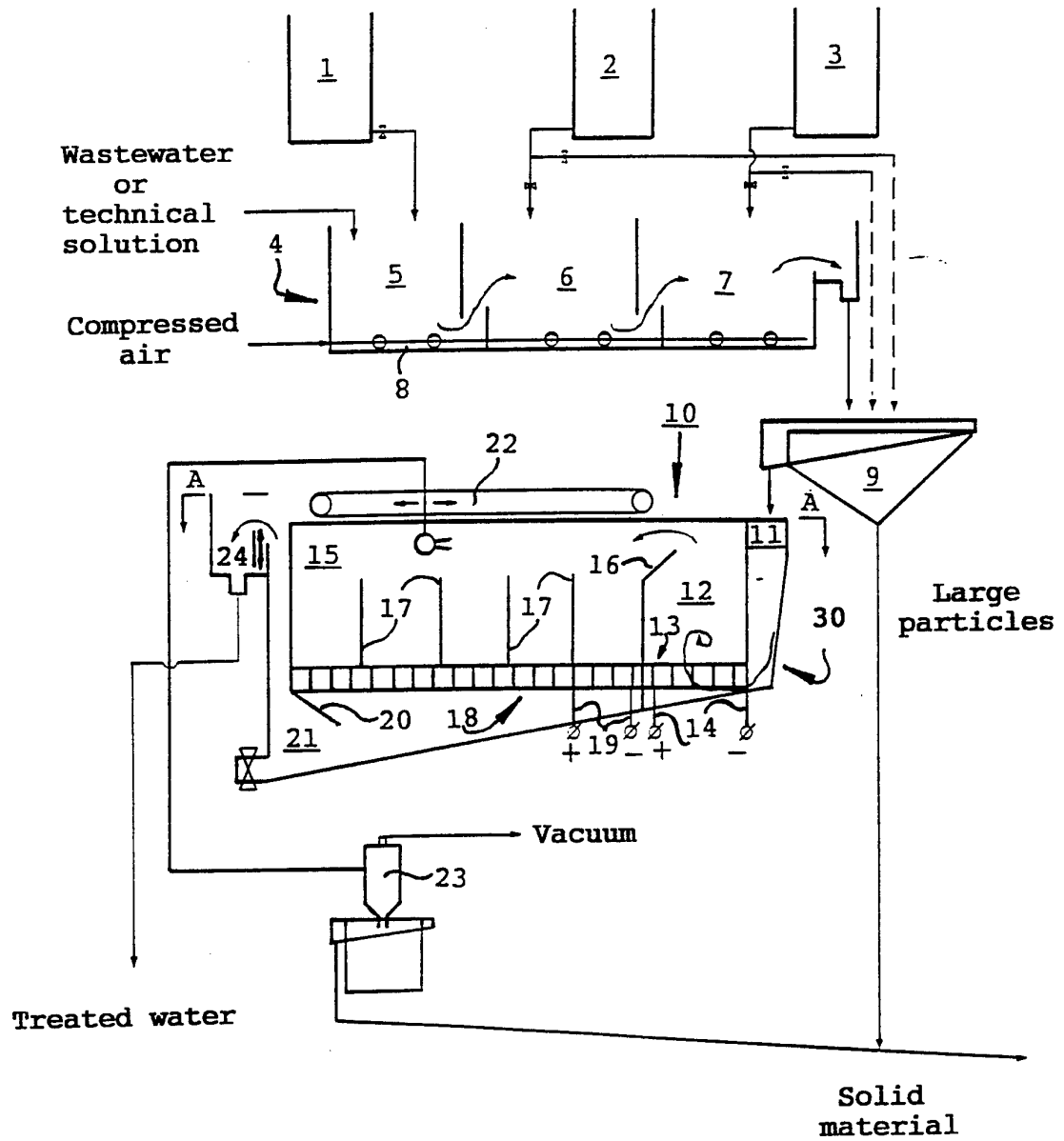


FIG. 2

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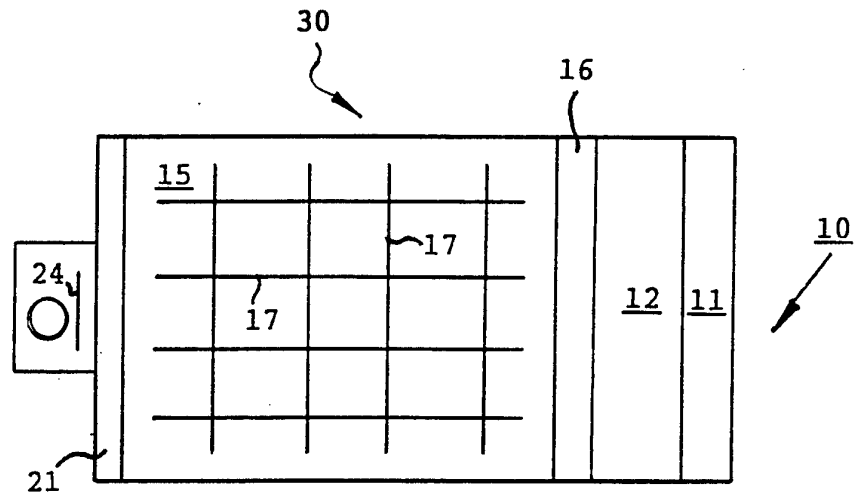


FIG. 3

INTERNATIONAL SEARCH REPORT

International application No.

PCT/FI 93/00485

A. CLASSIFICATION OF SUBJECT MATTER

IPC5: C02F 1/465, B03D 1/00

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC5: C02F, B03D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

WPAT, USPM

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	DE, B2, 2500455 (SWIFT & CO.), 9 February 1978 (09.02.78), column 5, line 14 - line 40, claims 1-2 --	1-7
X	US, A, 3822204 (FUMIO F. SAKO ET AL), 2 July 1974 (02.07.74), column 3, line 37 - line 53 --	4-7
A	--	1-3
A	EP, A1, 0469582 (IWATECH KONRADT GMBH), 5 February 1992 (05.02.92) --	1-7

☒ Further documents are listed in the continuation of Box C.☒ See patent family annex.

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Date of the actual completion of the international search

2 March 1994

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INTERNATIONAL SEARCH REPORT

International application No.

PCT/FI 93/00485

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US, A, 4156648 (THEODORE A. KUEPPER), 29 May 1979 (29.05.79) -----	1-7

INTERNATIONAL SEARCH REPORT

Information on patent family members

28/01/94

International application No.

PCT/FI 93/00485

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US-A- 3822204	02/07/74	CH-A- 478065 FR-A- 1545879 GB-A- 1151950	15/09/69 00/00/00 14/05/69
EP-A1- 0469582	05/02/92	DE-A- 4024424 DE-U- 9108712	06/02/92 05/12/91
US-A- 4156648	29/05/79	NONE	