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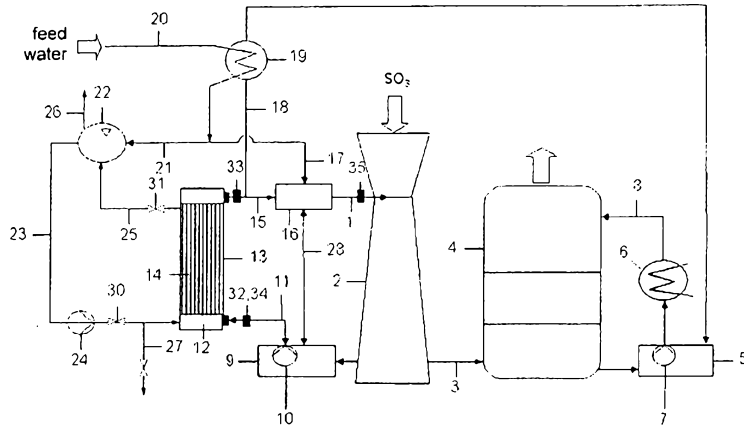


Fig. 1

(57) Abstract: When cooling acid which is withdrawn from an absorption apparatus of a sulfuric acid plant, the acid is pumped from an acid pump tank into a heat exchanger and subsequently again supplied to the absorption apparatus, wherein water as heat transport medium is heated in the heat exchanger with the heat of the acid and is at least partly converted into steam, and wherein the water is separated from the steam. It is provided that the acid is supplied to a shell space of the heat exchanger and the water is supplied to heat transfer elements arranged in the shell space and at least partly converted into steam, that the steam generated in the heat exchanger is separated from water in a steam drum, and that the water thus obtained is recirculated to the heat exchanger by means of a pump.

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Process and Plant for Cooling Sulfuric Acid

Field of the Invention

5 The present invention relates to the cooling of acid which is withdrawn from an absorption apparatus of a sulfuric acid plant, wherein the acid is pumped from an acid pump tank into a heat exchanger and subsequently is again supplied to the absorption apparatus, wherein water as heat transport medium is heated in the heat exchanger with the heat of the acid and is at least partly converted into steam, and wherein the water is separated from the steam.

10 Any discussion of the prior art throughout the specification should in no way be considered as an admission that such prior art is widely known or forms part of common general knowledge in the field.

15 Sulfuric acid usually is produced by the so-called double absorption process as it is described in Ullmann's Encyclopedia of Industrial Chemistry, 5th edition, vol. A25, pages 635 to 700. Sulfur dioxide (SO_2) obtained as waste gas of metallurgical plants or by combustion of sulfur is converted to sulfur trioxide (SO_3) in a multistage converter by means of a solid catalyst, e.g. with vanadium pentoxide as active component. The SO_3 obtained is withdrawn after the contact stages of the converter and supplied to an intermediate absorber or after the last contact stage of the converter to a final absorber in which the gas containing SO_3 is guided in counterflow to concentrated sulfuric acid and absorbed in the same.

20

25 The absorption of the SO_3 in sulfuric acid is a strongly exothermal process, so that the acid is heated up and must be cooled again. At the same time, the heat of the acid can be utilized for steam generation and energy recovery. Due to the temperatures of distinctly $> 140^\circ\text{C}$, which exist during the sulfuric acid absorption, acid cooling so far has exclusively been effected in kettle-type boilers, wherein the hot acid flows through U-shaped tubes to be passed through a kettle filled with water as heat transport medium. The circulation here is based on the thermosiphon principle. The heated water is converted into low-pressure steam and rises due to the lower density. The steam can be utilized in the plant (cf. Ullmann's Encyclopedia of Industrial Chemistry, loc. cit. p. 662).

30

While such kettle-type boilers have a simple construction and therefore can be manufactured at low cost, large amounts of water are required for filling the kettle type boiler. In addition, problems can arise when leakages occur in the acid circuit. Since acid leaks from the tubes into the water tank, a large amount of a highly corrosive weak acid is obtained, whose temperature in addition greatly rises due to the hydration heat produced. The corrosion resistance of the steels used in the system greatly drops below a sulfuric acid concentration of 99.1 wt-% (steel 310SS) or 97.9 wt-% (steel 3033). There is a risk of damage of the tube bundle or even of the entire kettle-type boiler. In addition, the acid/water mixture can only be separated with a disproportionate effort, so that in practice the user mostly will have to completely empty one or both of the systems.

Summary of the Invention

It is an object of the present invention to overcome or ameliorate at least one of the disadvantages of the prior art, or to provide a useful alternative.

It is also the object of the invention in at least one preferred form to provide for a reliable cooling of sulfuric acid and to increase the safety of the plant.

A first aspect of the invention provides a process for cooling acid which is withdrawn from an absorption apparatus of a sulfuric acid plant, wherein the acid is pumped from an acid pump tank into a heat exchanger and subsequently is again supplied to the absorption apparatus, wherein water as heat transport medium is heated in the heat exchanger with the heat of the acid and is at least partly converted into steam, and wherein the water is separated from the steam, wherein the acid is supplied to a shell space of the heat exchanger and the water is supplied to heat transfer elements arranged in the shell space and at least partly converted into steam, wherein the steam generated in the heat exchanger is separated from water in a steam drum, and wherein the water thus obtained is recirculated to the heat exchanger by means of a pump.

As in the heat exchanger less water is brought in contact with the acid than in the conventional kettle-type boiler filled with water, the amount of water mixed with acid is distinctly reduced in the case of a leakage. Since the functions of the conventional kettle-type boiler are split up into the separate elements heat exchanger, steam drum

and recirculation pump, handling additionally is facilitated in the case of a leakage. Water and acid circuit can easily be separated.

To further reduce the risks in the acid cooling circuit, a fast error detection is desired. As soon as an error is detected, the supply of water to the heat exchanger can be
5 switched off and the error can be eliminated.

In accordance with one preferred form of the invention, the interruption of the inflow of water is initiated e.g. upon measurement of a changed acid concentration. For this purpose, various measurement points are provided, which preferably are based on different measurement principles. In the absorber, methods can be used in particular
10 which determine the concentration of the acid via the conductivity, whereas in the cooling stage around the associated heat exchanger the sound velocity in the medium or the refractive index of the acid preferably is determined. A redundancy of the instruments and measurement principles used ensures that the acid concentration can safely be determined at any time.

However, the acid concentration only can effectively be used for leakage detection when it is linked with the process water stream. At the beginning, leaks usually are small, so that only small amounts of water enter into the acid circuit. As a result, the acid concentration is decreased, but this is compensated by the process control in that less process water is added. The larger the leak becomes, the more process water is
15 replaced by the water leaking in the heat exchanger. In this way, a uniform acid concentration is maintained. The leakage in the heat exchanger, however, only is detected when the process water valve is completely closed and the acid concentration still continues to drop. The equipment then would probably already be severely damaged. In accordance with a preferred form of the invention, the process water
20 supply, the plant loading and the acid concentration therefore are monitored at the same time.

In accordance with a particularly preferred form of the invention the temperature of the acid is measured at the inlet and outlet of the heat exchanger and linked with the flow rate of the heat transport medium supplied to the heat exchanger. Via the temperature
30 difference ΔT , the heat balance can be created via the heat exchanger. A leakage in the heat exchanger will lead to a disturbance of the heat balance, since water enters

into the acid circuit and generates additional heat. The ratio between the steam produced and the heat produced in the heat exchanger is changed, which can serve as a control variable for an interruption of the water flow.

5 To check the composition of the boiler feed water, its conductivity is measured in accordance with the invention directly behind the heat exchanger. Since the pressure on the water/steam side of the heat exchanger is distinctly higher than on the acid side, there is usually no risk of major amounts of acid entering into the water circuit. For leakage monitoring, this measurement can therefore only be used to a restricted extent.

10 A second aspect of the invention provides a plant for cooling acid which is withdrawn from an absorption apparatus of a sulfuric acid plant, in particular for performing a process according to the first aspect of the invention, with a heat exchanger to which hot acid is supplied from an acid pump tank by means of a pump and in which heat is transferred from the acid to water as heat transport medium, with a steam generator in which steam is generated from the water, and with a return conduit for at least partly
15 recirculating the cooled acid to the absorption apparatus, wherein the heat exchanger is a tube bundle heat exchanger with a plurality of tubes or a plate heat exchanger with a plurality of plates as heat transfer elements, wherein the heat exchanger is connected with the acid pump tank and the return conduit and via a conduit with a steam drum to which the heated heat transport medium from the heat exchanger is supplied and in
20 which the steam generated is separated from water, and wherein the steam drum is connected with the heat exchanger via a recirculation conduit for the circulation of the water.

25 In a first embodiment of the invention a shell space of the heat exchanger surrounding the heat transfer elements is connected with the acid pump tank and the return conduit, and the heat transfer elements of the heat exchanger are connected with the recirculation conduit and with the steam drum.

30 Alternatively, it can be provided in accordance with the invention to connect the shell space of the heat exchanger surrounding the heat transfer elements with the recirculation conduit and the steam drum and to connect the heat transfer elements of the heat exchanger with the acid pump tank and the return conduit.

In the tube bundle or plate heat exchangers provided in accordance with one preferred form of the invention, the amount of water to be supplied to the tube, plate or respective shell volume is substantially lower than in the conventionally used kettle-type boilers due to the high packing densities used today. The amount of water/acid mixture produced in a case of leakage thus is also reduced.

In accordance with a development of the invention it is provided that the heat exchanger is arranged at a higher level than the acid pump tank. When the acid pump is switched off, the heat exchanger thus is emptied gravimetrically without further action. Further safety installations, which would have to withstand the contact with hot acid, are not necessary.

In the recirculation conduit a circulation pump is provided in accordance with the invention, in order to circulate the cooling water by force.

For adjusting the optimum acid concentration for the operation of the absorber, a mixing chamber preferably is provided in the return conduit to the absorption apparatus, in which the recirculated acid is mixed with process feed water.

Between the heat exchanger and the steam drum and/or in the recirculation conduit a shut-off valve expediently is provided, in order to uncouple the heat exchanger from the water circuit upon occurrence of an error and for maintenance work.

Shutting off the water supply to the heat exchanger is effected e.g. upon detection of a leakage. In accordance with one preferred form of the invention, temperature measurement stations for detecting the acid temperature therefore are provided before and behind the heat exchanger and/or concentration measurement stations are provided before the heat exchanger and/or the absorber for detecting the acid concentration.

When a plurality of heat exchangers are provided parallel to each other in accordance with a development of the invention, one of the heat exchangers can be uncoupled and repaired or maintained in a case of damage or maintenance, while the other continues to operate. The flexibility of the plant is increased thereby and a continuous operation is promoted.

Further developments, advantages and possible applications of the invention can also be taken from the following description of an embodiment and the drawing. All features described and/or illustrated form the subject-matter of the invention per se or in any combination, independent of their inclusion in the claims or their back-reference.

5

Brief Description of the Drawings

A preferred embodiment of the invention will now be described, by way of example only, with reference to the accompanying drawings, set out below.

Fig. 1 schematically shows a plant for performing the process in accordance with one embodiment of the invention.

10 Fig. 2 shows the course of the process water and boiler feed water streams in dependence on the plant loading.

Fig. 3 shows the course of the process water and boiler feed water streams in dependence on the leak size.

Detailed Description of a Preferred Embodiment

15 As can be taken from the flow diagram of the process of one embodiment of the invention as shown in Fig. 1, gaseous sulfur trioxide from a non-illustrated converter for converting SO_2 into SO_3 is introduced into a Venturi absorber 2 in cocurrent flow with concentrated sulfuric acid supplied via a conduit 1 and is partly absorbed in the hot acid, whose concentration is increased thereby. Via a conduit 3 the non-absorbed
20 sulfur trioxide is introduced into an intermediate absorber 4, which it traverses in counterflow to concentrated sulfuric acid for further absorption. Non-absorbed sulfur trioxide is withdrawn from the top of the intermediate absorber 4 and supplied to a non-illustrated catalytic conversion stage, while the enriched sulfuric acid is withdrawn at the bottom, partly removed as product or otherwise utilized in the plant, and upon
25 dilution with water in an acid pump tank 5 and cooling in a heat exchanger 6 the rest is recirculated by means of a pump 7 via the conduit 8 to the intermediate absorber 4.

Sulfuric acid withdrawn at the bottom of the Venturi absorber 2 is supplied to an acid pump tank 9 and is introduced from the same by means of a pump 10 via the conduit

11 into the shell space 12 of a heat exchanger 13 located at a higher level, in which the acid is cooled by means of water as heat transport medium.

The heat exchanger 13 preferably constitutes a tube bundle heat exchanger with a plurality of water-conveying tubes 14 serving as heat transfer elements, since here a particularly good heat transfer can be combined with a relatively small volume of heat transport medium. Alternatively, a plate heat exchanger can be used.

Via a conduit 15, the cooled acid flows into a mixing chamber 16 in which its concentration is adjusted to the desired value by means of process feed water supplied via a conduit 17, before the sulfuric acid is supplied to the Venturi absorber 2. Part of the acid can be branched off via a conduit 18 and be supplied to the acid pump tank 5 of the intermediate absorber 4. The elevated acid temperature can be utilized to heat up the process feed water in a heat exchanger 19.

The feed water is supplied via a conduit 20 and after heating in the heat exchanger 19 divided into the process feed water stream of conduit 17, which is supplied to the mixing chamber 16, and a stream supplied to a steam drum 22 via a conduit 21. Boiler feed water is withdrawn from said steam drum via a recirculation conduit 23 and supplied to the heat exchanger 13 by means of a circulation pump 24. In the heat exchanger 13, the boiler feed water is guided in cocurrent flow with the hot sulfuric acid supplied from the acid pump tank 9 and is heated up, so that a water/steam mixture is formed, which is supplied via the conduit 25 to the steam drum 22 and separated there. The steam is withdrawn via the conduit 26, while the water is recirculated to the heat exchanger 13. The sulfuric acid correspondingly is cooled in the heat exchanger 13. Although a cocurrent cooling is shown in the drawing, it is of course within the scope of the invention to guide the acid and the cooling water in countercurrent flow. The remaining plant construction is not influenced thereby.

As in the heat exchanger less water is brought in contact with the acid than in the conventional kettle-type boiler, the amount of water mixed with the acid is distinctly reduced in the case of a leakage. In a system in a tube bundle heat exchanger of the invention, which is comparable in terms of cooling capacity, about 2 m³ of water (about 36 m³ of acid) are contained, whereas a conventional kettle-type boiler contains about 10 m³ of water (about 30 m³ of acid). In the case of water and acid being intermixed

completely (worst case) in the configuration of the invention, this leads to a dilution of the sulfuric acid supplied from the absorber with a concentration of about 99 wt-% and a temperature of about 200 °C to 95.6 wt-% and an increase in temperature to 230 °C, whereas in the case of a leakage in the conventional kettle-type boiler the acid is diluted to 81 wt-% and the temperature rises to 274 °C. Such a sulfuric acid has a reducing effect and is highly corrosive. Since the materials of the heat exchanger are designed for an oxidizing stress acting with a sulfuric acid concentration > 90 wt-%, considerable damages will occur very quickly.

In the conduits 23, 25 shut-off valves 30, 31 are provided, by means of which the heat exchanger 13 can be separated from the water circuit e.g. for maintenance and repair purposes. The water from the heat exchanger 13 can be removed via a drainage conduit 27.

Since the heat exchanger 13 is arranged above the acid pump tank 9, the acid automatically runs back into the acid pump tank 9 merely by gravity when the pump 10 is switched off, which acid pump tank 9 has an acid-proof lining like the absorbers 2, 4 and the acid pump tank 5. Additional mechanical drainage means can be omitted. In addition, the user is not exposed to the hot acid.

At the inlet and outlet of the heat exchanger 13 the acid temperature is measured via sensors 32, 33. In addition, a plurality of concentration measurement stations 34, 35 are provided in the acid circuit, which are used for determining the acid concentration at the inlet of the heat exchanger 13 and at the inlet of the Venturi absorber 2, respectively. At the inlet of the Venturi absorber 2, the conductivity of the acid preferably is measured, whereas at the inlet of the heat exchanger 13 the sound velocity in the acid or its refractive index is measured. Due to the redundancy of the measurement stations and principles, a reliable control of the acid concentration is ensured, which can be adapted if necessary by supplying process feed water via the conduit 17 or acid from the acid pump tank 9 via a conduit 28.

From the temperature measurement 32, 33 at the inlet and outlet of the heat exchanger 13, the amount of heat transferred to the water can be calculated. The temperature difference ΔT over the heat exchanger can be expressed as a function of the heat exchanger feed water. This function represents the heat balance over the heat

exchanger and describes an almost constant curve which is independent of the plant loading and other process conditions. A leakage in the heat exchanger will lead to a disturbance of the heat balance, since water enters into the acid circuit and generates additional heat. The ratio between the steam produced and the heat produced in the heat exchanger is changed, which can serve as a control variable for an interruption of the water flow. The volume flows of the water discharged and introduced likewise can be used for leakage detection, since the same are in equilibrium in stationary operation.

In the case of normal operation, the boiler feed water stream and the process water stream represent linear curves dependent on the plant loading, as is shown in Fig. 2. In the case of a leakage, the temperature is increased due to the entry of water into the acid, whereby more water is required in the cooling circuit. At the same time, the concentration of the acid is decreased, so that less process water is admixed to the acid. This is illustrated in Fig. 3. In dependence on the size of the leak, the demand of boiler feed water rises, whereas the supply rate of the process water goes down.

In accordance with one embodiment of the invention, the boiler feed water stream is linked with the plant loading. When a fixed alarm level is exceeded, the acid pump 10 is switched off and the heat exchanger 13 is separated from the water circuit, in order to be able to perform the necessary repair.

When several heat exchangers 13 are provided in parallel, one heat exchanger can run on, whereas the other is maintained or repaired.

In the described embodiment, the acid is supplied to the shell space 12 and the cooling water is supplied to the tubes 14 or plates of the heat exchanger 13. However, the invention also covers the reverse case, in which the cooling water is supplied to the shell space 12 and the acid is supplied to the tubes 14 or plates of the heat exchanger 13.

Even if the acid temperature at the outlet of the intermediate absorber 4 generally is too low because of the lower amount of SO_3 absorbed, in order to be economically used for energy recovery, the arrangement described above for the cooling circuit of the Venturi absorber 2 can also be provided in principle for the intermediate absorber 4. Likewise, instead of the Venturi absorber 2 in the first absorption stage there can also be used a

countercurrently operated absorption tower similar to the intermediate absorber 4. The second absorption stage possibly can also be omitted.

Although the invention has been described with reference to specific examples, it will be appreciated by those skilled in the art that the invention may be embodied in many other forms.

5

List of Reference Numerals:

- | | | |
|----|--------|------------------------------------|
| | 1 | conduit |
| 5 | 2 | Venturi absorber |
| | 3 | conduit |
| | 4 | intermediate absorber |
| | 5 | acid pump tank |
| | 6 | heat exchanger |
| 10 | 7 | pump |
| | 8 | conduit |
| | 9 | acid pump tank |
| | 10 | pump |
| | 11 | conduit |
| 15 | 12 | shell space |
| | 13 | heat exchanger |
| | 14 | tube |
| | 15 | conduit |
| | 16 | mixing chamber |
| 20 | 17, 18 | conduit |
| | 19 | heat exchanger |
| | 20, 21 | conduit |
| | 22 | steam drum |
| | 23 | recirculation conduit |
| 25 | 24 | circulation pump |
| | 25, 26 | conduit |
| | 27 | drainage conduit |
| | 28 | conduit |
| | 30, 31 | shut-off valves |
| 30 | 32, 33 | temperature sensors |
| | 34, 35 | concentration measurement stations |

Claims:

1. A process for cooling acid which is withdrawn from an absorption apparatus of a sulfuric acid plant, wherein the acid is pumped from an acid pump tank into a heat exchanger and subsequently is again supplied to the absorption apparatus, wherein
5 water as heat transport medium is heated in the heat exchanger with the heat of the acid and is at least partly converted into steam, and wherein the water is separated from the steam, wherein the acid is supplied to a shell space of the heat exchanger and the water is supplied to heat transfer elements arranged in the shell space and at least partly converted into steam, wherein the steam generated in the heat exchanger is
10 separated from water in a steam drum, and wherein the water thus obtained is recirculated to the heat exchanger by means of a pump.
2. The process according to claim 1, wherein that the temperature of the acid at the inlet and outlet of the heat exchanger and/or the acid concentration is measured and linked with the flow rate of the heat transport medium supplied to the heat
15 exchanger.
3. A plant for cooling acid which is withdrawn from an absorption apparatus of a sulfuric acid plant, in particular for performing a process according to any of the preceding claims, with a heat exchanger to which hot acid is supplied from an acid pump tank by means of a pump and in which heat is transferred from the acid to water
20 as heat transport medium, with a steam generator in which steam is generated from the water, and with a return conduit for at least partly recirculating the cooled acid to the absorption apparatus, wherein the heat exchanger is a tube bundle heat exchanger with a plurality of tubes or a plate heat exchanger with a plurality of plates as heat transfer elements, wherein the heat exchanger is connected with the acid pump tank
25 and the return conduit and via a conduit with a steam drum to which the heated heat transport medium from the heat exchanger is supplied and in which the steam generated is separated from water, and wherein the steam drum is connected with the heat exchanger via a recirculation conduit for the circulation of the water.
4. The plant according to claim 3, wherein a shell space of the heat exchanger
30 surrounding the heat transfer elements is connected with the acid pump tank and the return conduit, and wherein the heat transfer elements of the heat exchanger are connected with the recirculation conduit and with the steam drum.

5. The plant according to claim 3, wherein a shell space of the heat exchanger surrounding the heat transfer elements is connected with the recirculation conduit and with the steam drum, and the heat transfer elements of the heat exchanger are connected with the acid pump tank and the return conduit.
- 5 6. The plant according to any of claims 3 to 5, wherein the heat exchanger is arranged at a higher level than the acid pump tank.
7. The plant according to any of claims 3 to 6, wherein a circulation pump is provided in the recirculation conduit.
- 10 8. The plant according to any of claims 3 to 7, wherein in the return conduit to the absorption apparatus a mixing chamber is provided, in which the recirculated acid is mixed with process feed water.
9. The plant according to any of claims 3 to 8, wherein between the heat exchanger and the steam drum and/or in the recirculation conduit a shut-off valve is provided.
- 15 10. The plant according to any of claims 3 to 8, wherein before and behind the heat exchanger temperature measurement stations are provided for detecting the acid temperature and/or concentration measurement stations are provided before the heat exchanger and/or the absorption apparatus for detecting the acid concentration.
- 20 11. The plant according to any of claims 3 to 10, wherein a plurality of heat exchangers are provided parallel to each other.
12. A cooled acid produced according to the process of claim 1 or claim 2.
13. A process for cooling acid which is withdrawn from an absorption apparatus of a sulfuric acid plant, substantially as herein described with reference to any one of the embodiments of the invention illustrated in the accompanying drawings and/or
25 examples.
14. A plant for cooling acid which is withdrawn from an absorption apparatus of a sulfuric acid plant, substantially as herein described with reference to any one of the embodiments of the invention illustrated in the accompanying drawings and/or examples.

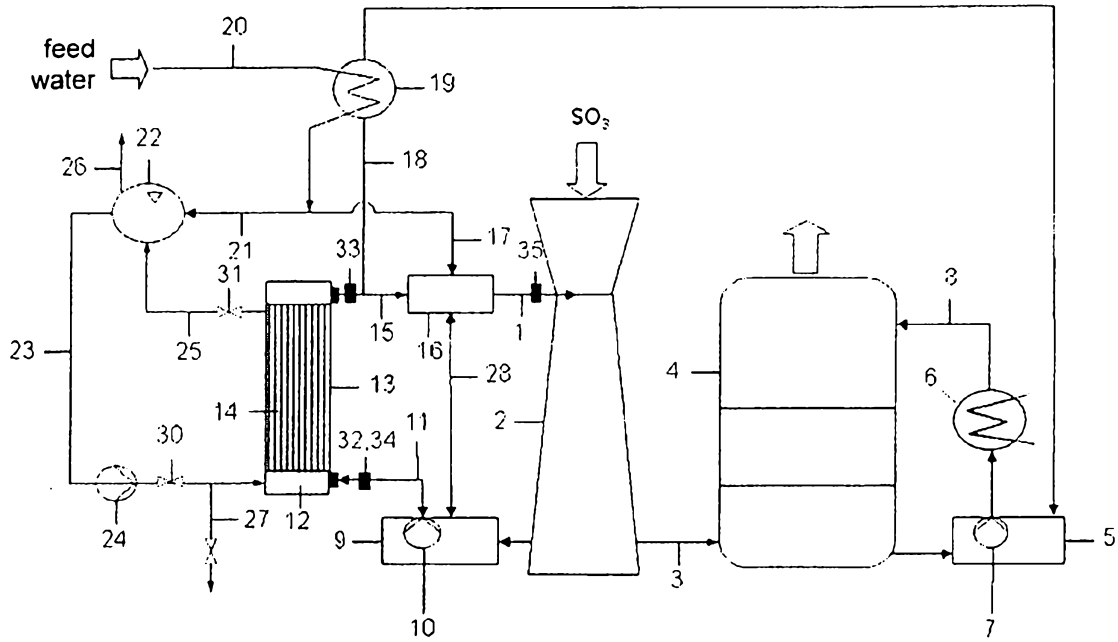


Fig. 1

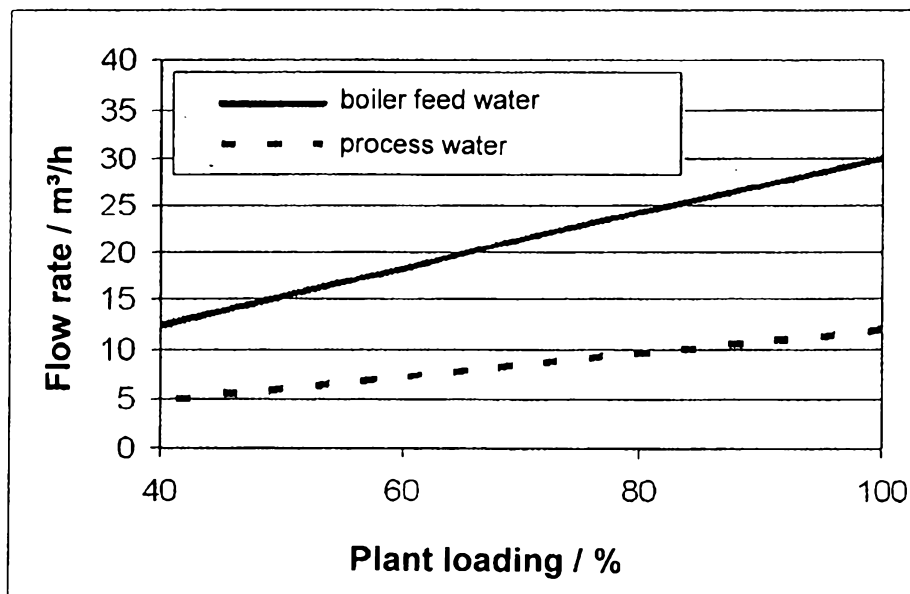


Fig. 2

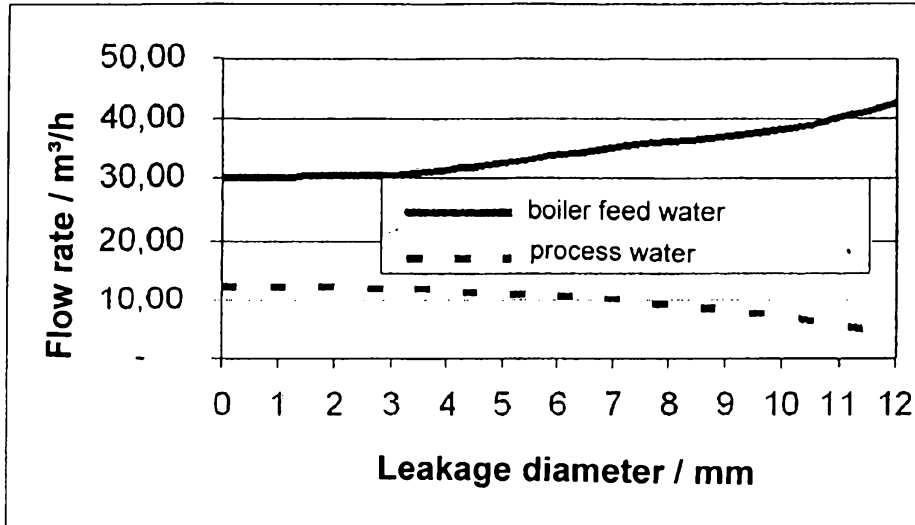


Fig. 3