

The invention relates to a process for the periodic heating and cooling of the separator of an installation for producing phthalic anhydride (PA) or maleic anhydride (MA), in which vaporous product is fed to the separator in the cooling phase, said separator is switched into a cooling circuit and the product is deposited on cooling surfaces of the separator, in the heating phase the separator is switched into a heating circuit containing liquid heat-transfer medium, the cooling surfaces are indirectly heated, the product is melted and is passed out of the separator in liquid form, and for the heat consumption steam which is produced as a surplus in the installation is used at a rate which is largely constant over time.

In the conventional performance of the separation of the crude PA or MA product produced in the gas phase, the gas coming from the production plant is cooled and predominantly deposition takes place by desublimation on the outside of an indirectly-cooled heat-exchanger. The discharging of the deposited crude product takes place by melting, in which case for the most part the same liquid heat-transfer medium as in the cooling phase, usually a thermal oil, is passed through the heat-exchanger at a temperature which is above the melting point of the product. The liquid heat-transfer medium is heated outside the product separator in the heater for the heat-transfer medium, which is heated with steam. A process of this type is described in EP-A-35 173. In order to compensate the widely fluctuating heat requirement, a storage circuit which is superimposed on the heating circuit is provided here for the liquid heat-transfer medium, from which storage circuit liquid heat-transfer medium is periodically taken. General explanations on steam accumulators can

be found in "Lexikon der Energietechnik und Kraftmaschinen", L-Z, Volume 7, page 539.

5 It is an object of the invention to perform the compensation of the fluctuating heat requirement as simply and economically as possible.

In the process referred to first hereinbefore, the object is achieved in that

- 10 a) the liquid heat-transfer medium is heated indirectly in a heat-exchanger (heater for the heat-transfer medium) with steam from the installation,
- 15 b) at the beginning of the melting operation during the period of the maximum steam requirement, a mixture of two streams of steam is fed to the heater for the heat-transfer medium, one stream coming directly from the installation and the other, larger, stream being brought in from a steam accumulator, and
- c) the steam accumulator is charged with steam from the installation during the period in which the heater for the heat-transfer medium is out of operation or is operated at a low load.

25 The rate of steam produced in the entire installation is usually greater than the total steam consumption within the installation, so that a certain quantity of excess steam is constantly available and can be used for other purposes. What is important is that the rate of the steam given off to the outside should fluctuate as little as possible, since the consumers can only

30 adapt to such fluctuations with very great difficulty,

and only the rate of steam which is constantly available can be used economically.

5 The use of the steam accumulator in accordance with the invention makes it possible to keep the rate of the steam withdrawn from the plant for producing PA or MA constant with fluctuations of at most $\pm 5\%$ and usually at most $\pm 2\%$, so that a largely constant rate of steam is also available for other consumers.

7 The steam accumulator is a known unit in which hot water is kept under pressure, it being possible to remove a desired rate of steam by controlled pressure-relief. In the process according to the invention, the pressure in the steam accumulator for the most part fluctuates within about the range of 3 to 40 bar.

15 Since the melting temperature of the PA or MA product is far lower than the boiling temperature of the fully-laden steam accumulator, the majority of the quantity of heat required for heating and melting can be fed to the heater for the heat-transfer medium at a relatively low temperature level. This yields a low specific storage volume and small dimensions of the container of the steam accumulator.

25 One further advantage of the process consists in that the liquid heat-transfer medium is only heated to relatively low temperatures, so that inexpensive oils on the basis of mineral oil can also be used.

One possible configuration of the process will be explained with the aid of the drawings.

Fig. 1 shows a diagram of the process and

30 Fig. 2 shows a comparison of the steam consumption.

The reactor 1 for producing PA is fed through line 2 with the mixture of the starting materials, air from line 3 and orthoxylene or naphthalene from line 4. If MA is to be produced, the starting materials are air and benzene or low-boiling hydrocarbons (C_4 cut). Since these synthesis processes are known, it is not necessary to go into them in greater detail here. A molten salt, which enters the reactor in line 5 and leaves it in line 6, serves for indirect cooling of the strongly exothermic reaction in the reactor 1. The molten salt is cooled in the heat-exchanger 7 by means of feed water from line 8; the steam produced thereby is removed in line 9.

The reaction gas produced, a mixture of vaporous product (PA or MA) and air, leaves the reactor 1 in line 10, and is subjected to a first indirect cooling step in a cooler 11 likewise with feed water, and the steam produced thereby can likewise be fed into line 9. Pre-cooled reaction gas is fed to the separator 13 in line 12 for further cooling and solidification of the product.

A reactor 1 usually consists of at least 3 separators 13, to which product-containing reaction gas is fed alternately from line 12. However, only one separator is shown in the drawing for purposes of simplification. Cooling liquid, which is brought in in line 14 and leaves the separator 13 in line 15, serves to cool the separator 13 and to deposit the product on the cooling surfaces. The cooling liquid circuit is under the action of the circulating pump 16 and passes via a cooler 17. A tap line 18 connects the cooling liquid circuit to an expansion vessel 19. The temperature of the cooling liquid in line 14 is usually in the range of 45 to 60°C and in line 15 in the range of 50 to 70°C.

Usually an oil serves as the cooling liquid, and is also used as the liquid heat-transfer medium for heating up the separator 13 and for melting the deposited product. In the heating phase, the supply of reaction gas from line 12 to the separator 13 is interrupted and diverted to other separators (not shown). At the same time, the supply of cooling liquid is switched off.

Then liquid heat-transfer medium at a temperature in the range of 140 to 210°C enters the separator 13 in line 21, causes the product deposited therein to melt, with the product flowing through line 22 into a tank 23. The liquid circuit which heats up the separator 13 is formed of the return line 24 with the circulating pump 25 and the tap line 26 with the expansion vessel 27. The temperature of the liquid in line 24 is usually in the range of 100 to 200°C. The liquid heat-transfer medium, usually an oil, is heated indirectly in the heater 28 for the heat-transfer medium with steam from line 30. Cooled steam and condensate leave the heater 28 through line 31.

At the beginning of the heating phase in the separator 13, if there is a large heat requirement in the heater 28, the major part of the steam required comes from the steam accumulator 32 and flows through line 33, in which there is a check valve 34, and through line 30 to the heater 28.

A rate of steam which is constant over time is brought in in line 35, the constancy of the throughflow being monitored in a throughflow meter 36 and being set with the aid of a controller 37 and a control valve 38. The pressure in line 35 is usually in the range of 15-40 bar. During the first heating phase, when the steam consumption in the heater 28 is great and the

pressure therein is correspondingly low, the steam flows out of line 35 into line 40, and mixes with the steam from the steam accumulator 32 coming from line 33. Towards the end of the heating phase, when the steam consumption has diminished and the pressure in the heater 28 has increased, the control valve 42 in the line 43 leading to the steam accumulator 32 is opened by a pressure controller 41, and the accumulator 32 is loaded.

As soon as the condensed quantity of steam in the heater 28 falls below the quantity of steam brought in from line 40, the pressure on the steam side of the heater increases, and no further steam is taken from the steam accumulator 32. The pressure-controlled control valve 42 and the check valve 34 initially prevent the further incoming steam from passing into the accumulator 32. This causes the steam pressure in the heater 28 to rise to its maximum value in a short time, as desired. Not until this maximum value has been achieved and the incoming quantity of steam exceeds the requirement at this point is the control valve 42 opened to such an extent that the steam pressure in the heater 28 is maintained. The excess quantity of steam then passes through line 43 into the accumulator 32, and heats the latter with a constant pressure increase.

When the melting operation in the separator 13 has been terminated, the separator is re-cooled again by switching to the cooling circuit (lines 14 and 15, pump 16 and cooler 17). The steam consumption in the heater 28 for the heat-transfer medium approaches zero, and the incoming quantity of steam is almost completely absorbed by the accumulator 32, until the same pressure is achieved everywhere downstream of the line 35.

A steam accumulator 32 and a heater 28, to which a plurality of separators 13 are alternately connected serve for separating the product.

5 The graph of Figure 2 shows a comparison of the rate over time of the consumption of steam X in the heater 28 in the form of the sawtooth line A and the steam brought in in line 35 with its constant rate over time (dashed line B). By interposing the steam accumulator 32, it is possible to operate with a constant amount of
10 steam being supplied, despite strong consumption peaks (line A). Since only part of the steam conveyed in line 9 is passed on in line 35, a constant quantity of steam can be supplied to other consumers in line 44.

15 If, as usual, one operates with at least 3 separators, each of the three sawtooth peaks of Figure 2 is associated with a different separator. At the time I, one of the separators is switched to melting, and immediately thereafter, at the time II, the maximum quantity of steam is consumed in the heater 28. At the
20 time III, the steam accumulator 32 does not need to give off any more steam, and the pressure in the accumulator and in the heater 28 has reached a minimum at this point. At the time IV, the heating of the separator which is switched to melting in each case is
25 terminated.

Example:

In a process corresponding to Figure 1, one operates as follows:

30 81,000 kg per hour of a mixture of vaporous crude PA and air at a temperature of 380°C leave the reactor 1 through line 10. After preliminary cooling in the cooler 11, the mixture flows into one of four

separators 13 at a temperature of 170°C. The heat dissipation in the cooler 11 and heat-exchanger 7 means that 24,000 kg steam, which is saturated steam at 20 bar, can be removed per hour in line 9. Of this quantity of saturated steam, 10% (2,400 kg/h) is passed through line 35 into the heater 28 and/or the steam accumulator 32, and the remainder can be fed to other consumers in line 44.

A conventional thermal oil on the basis of mineral oil serves for cooling or heating the separators. The quantity of this oil flowing per hour through line 14 or 21 is 110,000 kg. The heat requirement for melting the product in a separator, which changes over time, leads to fluctuating values which are given in the table below. I signifies the time immediately before the beginning of the heating of the separator; adjacent points I, cf. Figure 2 of the drawings, are about 2 hours apart. The points II, III and IV have the meanings already explained together with Figure 2. P is the pressure, Q is the throughflow rate and T is the temperature in the respective line. The pressure in lines 33 and 30 is equal to the pressure in line 40.

Table

Time

Line	I	II	III	IV
40	P = 19 bar Q = 100 kg/h	P = 18 bar Q = 2400 kg/h	P = 6 bar Q = 2400 kg/h	P = 19 bar Q = 100 kg/h
33	Q = -	Q = 5600 kg/h	Q = -	Q = -
43	P = 18 bar Q = 2300 kg/h	P = 18 bar Q = -	P = 6 bar Q = -	P = 8 bar Q = 2300 kg/h
30	Q = 100 kg/h	Q = 8000 kg/h	Q = 2400 kg/h	Q = 100 kg/h
14	T = 55°C	-	-	-
15	T = 70°C	-	-	-
21	-	T = 140°C	T = 150°C	T = 190°C
24	-	T = 100°C	T = 135°C	T = 185°C



Claims

1. A process for the periodic heating and cooling of the separator of an installation for producing phthalic anhydride (PA) or maleic anhydride (MA), in which
5 vaporous product is fed to the separator in the cooling phase, said separator is switched into a cooling circuit and the product is deposited on cooling surfaces of the separator, in the heating phase the separator is switched into a heating circuit containing
10 liquid heat-transfer medium, the cooling surfaces are indirectly heated, the product is melted and is passed out of the separator in liquid form, and for the heat consumption steam which is produced as a surplus in the installation is used at a rate which is largely
15 constant over time, characterised in that

- a) the liquid heat-transfer medium is heated indirectly in a heat-exchanger (heater for the heat-transfer medium) with steam from the installation,
- 20 b) at the beginning of the melting operation during the period of the maximum steam requirement, a mixture of two streams of steam is fed to the heater for the heat-transfer medium, one stream coming directly from the installation and the
25 other, larger, stream being brought in from a steam accumulator, and
- c) the steam accumulator is charged with steam from the installation during the period in which the heater for the heat-transfer medium is out of
30 operation or is operated at a low load.



2. A process according to Claim 1, characterised in that the quantity of the steam brought in from the installation is kept constant with fluctuations of at most $\pm 5\%$.

- 5 3. A process according to Claim 1 or 2, characterised in that the steam accumulator is not charged until the maximum pressure has been reached in the heater for the heat-transfer medium.