

Process for Reclaiming Electricity-Conducting Adsorbents**Charged with Organic Substances**

This invention concerns a process for reclaiming electricity-conducting adsorbents charged with organic substances, which are heated up by transition of electrical power. For many years, processes for separation of organic gaseous matters from exhaust gas streams by adsorption to adsorbents, especially to activated carbon, have been used industrially, for example, in solvent reclaiming plants. The activated carbon is mostly in a solid bed packed with granules. Activated carbon is sometimes used in the shape of mats, such as fleeces, woven or knitted material wrapped into ring-adsorbents or set in a frame. The total process of exhaust gas purification by adsorption is divided into three main phases: the loading phase, the regeneration phase and the cooling phase. During the loading phase, the adsorbent is charged with exhaust gas. In this process it binds the organic matter in its inner pore structure. During the loading phase the front charge moves through the adsorber in the direction of the flow. When the adsorbent, at the end of the adsorber, (viewed in flow direction), is loaded to the extent, that the pure gas concentration reaches a pre-set

limiting level, then the loading of this particular adsorber is finished, and another freshly regenerated adsorber is used. For economic reasons one normally aims to regenerate the adsorbent. In numerous cases, however, regeneration is neither possible nor economically viable. This is especially the case if the concentration of organic matter is very low, such as in the air intake for buildings or in the air circulating in building air conditioners. In these cases the charged adsorbent is removed and replaced with a new one.

Adsorption is a reversible process, therefore the bound organic matter can be removed from the adsorbent in a regeneration phase, by changing the balance conditions. This may be effected through heating the adsorbent and expelling the adsorbed organic matter by means a stream of flushing gas. This process is used in activated carbon adsorption plants regenerating either by water vapor or by an inert gas.

In both cases the flushing medium also serves as a heating medium for the adsorbent. Heating the adsorbent and desorption of the organic matter are thus inseparably linked. Due to this indirect heating of the charged adsorbent by the flushing gas, very large volume flows of flushing gas are required. In the case of regeneration by water vapor, this water vapor has to be condensed after discharge from the adsorber, together with the organic matter desorbed by the adsorbent. The condensate formed has to be treated by further involving advanced processes, for example, rectification, in order to separate water and organic matter. The regeneration by inert gas which normally employs nitrogen as inert gas, is even more involved than regeneration by water vapor due to the fact that the nitrogen has to be introduced into a cycle over the adsorber and this adsorber must be

cooled to a very low temperature in the condensation phase, in order to condensate the desorbed organic matter and to keep the residual charge of the adsorbent at the end of the regeneration phase low enough to comply with the desired pure gas concentration in the following charging phase.

Due to the fact that the adsorbent is heated up very slowly by the inert gas, owing to its low heat capacity, and high flow speeds between 0.1 and 0.5 m/s that are required, this process can only achieve concentration factors (= maximum regeneration gas concentration/raw gas concentration) of about 40. A significant improvement may be effected if the adsorbent is not heated indirectly by the flushing gas, but directly by passing electrical energy. In this case the required amount of flushing gas may be significantly reduced due to the separation of the heating and flushing processes, and significantly higher concentration factors can be achieved.

Likewise, a device for reclaiming organic solvents is known from DE 195 13 376 A1, consisting of a ring-shaped solid bed filled with activated carbon granules and heated by electricity. This device is charged first with exhaust gas stream in order to adsorb the solvent and consecutively with the regeneration gas, in order to effect desorption or regenerating. For this device, the outer and inner and outer shell of the ring-shaped solid bed consists of a grid metal, forming the electrodes. This process allows concentration factors of 120, and thereby the required flushing gas stream may be reduced to 5 to 10% of the raw gas volume streams. The specific gas volume involved is very small, when related to the contact area. Therefore the carbon can only be charged evenly with the flushing gas, which is a pre-requisite for a high-quality regeneration, if charging is effected centrally within the adsorber by means of a distributor tube featuring numerous small bore holes.

Detrimental to the charging standing period and the quality of pure gas concentrations in this process is the fact that the regeneration gas stream itself is heated up by the activated carbon and during regeneration from inner to outer layer, therefore the carbon in the innermost carbon layer reaches a significantly lower regenerating temperature than in the outer layer. In consequence, the innermost carbon layer is insufficiently regenerated and therefore it is not possible to achieve very low pure gas concentrations during the charging phase, for example, some $\mu\text{g}/\text{m}^3$ as required for air inflow into highly clean rooms.

An adsorber is known from patent script DE 41 04 513 C2, which likewise is heated up by electricity, it is characterized by using fibrous activated carbon for example in the shape of mats. One sample of such an adsorber is shown in drawing figure 1.

For the treatment of larger volumes of exhaust air, several of these frames may be combined in a number of useful arrangements, with respect to air and electricity. The fabrics heat up very fast by using electricity, in maybe less than a minute, and thus very high concentration factors of up to 1500 may be achieved, which are necessary for reclaiming solvents, for example.

The disadvantage is that it is difficult to distribute the regenerating gas evenly across the filter area. The regeneration gas speed, in relation to the filter area in the example mentioned above, of a 1m x 3m filter is only 0.01m/s. At this flow speed a pressure loss of 10 Pa results through five layers of activated carbon, for example, so

that the regenerating gas must reach the filter in an evenly distributed manner. This may be effected for example by a system of distribution tubes featuring numerous bore holes spread over its height and its cross-section, allowing the regenerating gas to be emitted in a free jet. (Figure 2)

A large volume of air to be treated i.e. a number of filter frame modules, requires the installation of a large number (3 times the number of filter frames) of distribution tubes or to choose a very large distance between a distribution tube and a filter (0.87m), which makes the building volume of the devices too large to be economically viable.

A further detrimental factor is that during this process of electrical regeneration, the desorption gas is heated by heat transmission from the electrically heated fabric to the air. This results in negative consequences, as described above, as to the effectiveness of the exhaust air cleaning process by forming a temperature gradient. If activated carbon fiber fabric is used, this effect is even more pronounced, as shown in figure 3, due to the small amount of carbon utilized, rather than in granulated carbon, where the same volume of exhaust air is treated using more than 100 times the amount of activated carbon. A consequence of this steep temperature gradient is the fact that during regeneration the first layers of fabric, viewed in the direction of regeneration, which are especially important for the final cleaning of exhaust air are not optimally regenerated. This results in a shortening of charging standing periods as well as a worsening of the achievable pure gas concentrations during the following charges. The higher the regeneration gas speeds, the more pronounced this negative effect.

Apart from this, a further detrimental effect is shown in the following table. Higher desorption speeds

require a drastically increasing electric heating power. As a consequence, for economical reasons the regeneration gas speeds are kept as low as possible for electrically regenerated adsorbers consisting of activated carbon fiber fabric, thus in turn necessitating installation of a costly regeneration gas distributor system.

Electric Power requirement during regeneration of activated carbon fiber fabric Dependent on regeneration speed						
Desorption gas speed (cm/s)	0	1	2	5	10	20
Overall electric heating power (kW/m ²)	3.1	5.2	7.5	14.4	26.2	49.4

Finally, what is detrimental to this process is that it is very difficult to regulate the temperature of the activated carbon fabric, a matter of essential importance to the practical operation of this process for safety reasons. The reason for this problem may be found in the low heat conductivity and heat capacity of activated carbon fabric.

Conventional methods of temperature measurement, for example, using thermal elements insulated from electricity, react with great time delay and the "measurement value" is very prone to inaccuracy, because it depends on a close mechanical contact of thermal element and fabric. The time it takes for the fabric to heat up from room temperature to 200°C is only about a minute. Moreover, the electric resistance of activated carbon has a negative temperature coefficient, hence this process is very prone to the danger of overheating the activated carbon fabric.

The two known processes to electrically regenerate adsorbents incorporate a cooling phase to follow, whereby the adsorbent is cooled down, by means of passing air or inert gas, to the temperature required for the subsequent charging phase.

It is therefore the invention's task to find a process to electrically regenerate electricity-conducting adsorbents, especially activated carbon fiber fabrics, that incorporate, on the one hand, the advantages of electrical regeneration as compared with conventional regenerating processes e.g. inert gas regeneration and water vapor regeneration, such as:

- Very short desorption periods
- Very short heating periods
- Very large concentration factors at regeneration
- If required, regeneration temperatures up to 300° C
- For fibrous sorbents, extremely rapid kinetics of mass transfer from gaseous to sorbent phase
- Economically viable, the processes using electrical regeneration only heat up the activated carbon and the regeneration gas, but not tubes, containers and other plant parts.

On the other hand, it must not feature any of the disadvantages of conventional methods to electrically regenerate adsorbents, as have been described above, especially:

- Should permit high desorption gas speeds of significantly more than 2 cm/s, without requiring a significantly higher-rated electric power input than at desorption gas speeds below 1 cm/s. This is desirable especially if air is used as the regeneration gas and this regeneration gas does not require further purification, such as, for example, in the task of filtering gaseous substances existing in the outside air from the air intake into a building.

- Should require no complicated device for the even distribution of the regeneration gas over the contact area of the filter, so that on the one hand the machinery expense for this distribution device is not incurred, and on the other hand the space required for these filters compared to conventional building methods is significantly reduced.
- The regeneration process is intrinsically safe from overheating, burning of the activated carbon fiber, so that temperature control of the electrical power is not necessary.

According to this invention, this task is solved by a regeneration process according to patent claims 1 to 7.

This will be explained according to the following example, which is based on values measured at an experimental plant in which all the characteristics of the invention process have been implemented.

In a laboratory experimental plant, which has been dimensioned for air volumes ranging from 0 up to 30 m³/h, trials of adsorption and desorption have been carried out with interval timing during desorption.

The figures show:

Figure 4 a segment view of the adsorber according to this invention

Figure 5 the resulting breakthrough curve Toluol

Figure 6

and

Figure 7 schematic representations of desorption via the distributor tube and without the distributor tube

Figure 8

To

Figure 11 The time sequences of the timed desorption

As illustrated in figure 4, the charging of laboratory adsorber 1 was effected in the direction indicated, coming from the raw gas pipe 5 to filter 2 to the pure gas pipe 4. During this process, five layers of ACF were clamped in the filter frame. The charging and discharging pipes 3 and 6 are shut during the charging phase. The charging concentration was selected to be approximately 40mg/m^3 for the solvent Toluol, whereby the relative humidity of the raw gas was maintained at 50% at a temperature of 23°C .

The speed at filter, with an exposed filter area of 0.07 m width and 0.21 m height, was measured as 0.3 m/s. The trials were continued up to a breakthrough concentration of 10% of the raw gas concentration (Fig.5). The selected breakthrough concentration was 4 mg/m^3 , which was reached after approximately 40 minutes.

During the subsequent desorption, the airflow was directed in the opposite direction as in adsorption, and the desorption gas was introduced either via desorption pipe 3 or the normal pipe conduit 4, depending on the gas speed (see Fig. 6 and 7). The regeneration air was removed via the regeneration air pipe 6. During the whole regeneration process, which is composed of two parts: heating and desorption/cooling, the gas pipe 5 is always shut.

Temperature measurements during desorption were carried out by means of IR measuring, without contact with the filter surface and using specially treated thermal elements between the filter layers.

The timed desorption was carried out as follows (see Fig. 8 to 11):

- Heating up the filter material by direct electrical heating at constant power input over a period of 2.5 minutes. During this time the valves at the adsorber were closed.
- After 2.5 minutes the electric power input was stopped, the valves at the adsorber were opened for 0.5 minutes. During this period the desorbed solvent and the desorbed water is discharged and the temperature at the filter decreases. After 0.5 minutes the valves at the adsorber are closed.
- Heating up of the filter material by direct electrical heating with constant power input over a period of 2.5 minutes.

By this interval timing and process design and control, the heating and flushing were effected seven times in all trials, after which the process was stopped so that another adsorption process could start.

The lower temperatures achieved at the first two intervals during heating at the filter are due to the predominant desorption of the adsorbed water on the ACF. From the third interval onwards the stationary temperatures were achieved for the respective power input and time duration.

Surprisingly, it was now found, that by using this interval timed regeneration the activated carbon fiber fabric can be largely desorbed with only seven intervals, even though during the actual desorption time- which is identical to the cooling time- no

electric heating input was supplied, and that this time was very short - for example, only 30 seconds at a regeneration air speed of 20 cm/s - and also, that during this time the temperature of the activated carbon fiber fabric decreased from the origin 220°C to approximately 40°C. During the following charging phase the same standing time was achieved. This shows that a stationary condition is present i.e. that during interval desorption the same amount of Toluol was desorbed as had been adsorbed previously in the charging phase.

Surprisingly, and to great advantage for the suggested process, it was further discovered that by using the regeneration process according to this invention the regeneration gas speed in a broad range has practically no influence on the desorption quality, on the use of electrical energy or the electrical connected load respectively and on the pure gas quality in the subsequent charging phase, if the number of desorption intervals remain unchanged.

This is clarified by the following table:

Regeneration gas speed (cm/s)	5	10	20
Regeneration gas distributor	yes	No	no
Number of desorption intervals	7	7	7
Electrical heating input (kW)	0.16	0.16	0.16
Energy input per desorption interval (kJ)	24	24	24
Maximum desorption gas concentration (g/m ³)	10.2	11.5	10.5
Raw gas concentration (mg/m ³ Toluol)	40	40	40
Relative humidity (%)	50	50	50
Pure gas concentration at the start of the subsequent charging phase (mg/m ³ Toluol)	0.1	0.18	0.13
Standing time until breakthrough concentration of 10% of the raw gas concentration (min)	44	39	40



What is claimed is:

1. A regeneration process for electric-current-conducting adsorbents loaded with organic substances and which can be heated by applying electric current, **wherein** electric current is passed through the adsorbents which are heated alternately and successively, in which case purge gas is not fed through the adsorbents but only once the electric current has been switched off, is purge gas routed through the adsorbents thus desorbing the adsorbed organic substances and cooling the adsorbents.
2. The process of claim 1 **wherein** both said stages of heating and desorption/cooling are repeated several times in succession.
3. The process of claims 1 and 2 **wherein** said heating stage is conducted for a fixed time span.
4. The process of claim 3 **wherein** said fixed specific heating period is calculated in such a way that the time span is equal to the minimum time required to heat the totally unloaded adsorbents to a pre-determined maximum temperature at a defined electric heating capacity.
5. The process of claims 1 to 4 **wherein** the superficial velocity through the adsorbents amounts to 0.1 to 0.5 m/s.
6. The process of claims 1 to 5 **wherein** adsorbents are in the form of fleece, cloths or knitted fibres consisting of fibrous activated carbon.
7. The process of claims 1 to 3 **wherein** said time spans are specified so as to allow the adsorbed mixtures to be desorbed and trapped in succession according to their volatility.