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The invention relates to a method for increasing the absorbency of a material containing alkaline-earth metal carbonate and alkaline-earth metal hydroxide in relation to sulphur oxides and/or other pollutants in flue gas. The invention furthermore relates to the use of this material for dry flue gas scrubbing.

In the field of off-gas scrubbing, numerous methods are employed. Besides wet off-gas scrubbing, dry off-gas scrubbing is also employed. Materials containing alkaline-earth metal carbonate and/or alkaline-earth metal hydroxide, in particular lime products, are used in various dry flue gas scrubbing processes as sorbents for the deposition of acid-forming off-gas components in various temperature ranges.

The aim is to neutralise the acidic pollutants present in the off-gas flow, such as sulphur dioxide, hydrogen chloride and hydrogen fluoride, and to deposit on suitable deposition devices the neutral salts formed. In this case, for example, bed filters, entrained-flow processes, in conjunction with electro-filters or fabric filters, are used.

Dry off-gas scrubbing is used in different variants. The most essential fields of use are scrubbing the off-gases of coal and lignite power stations, waste incineration plants, hazardous waste incineration plants, heat engines and furnaces with various fuels.

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One widespread technique in the temperature range of up to mostly about 200°C is the bed filter technique. In this case, sorbents based on limestone (CaCO_3), in particular granulated or pelleted products based on limestone (CaCO_3) and/or lime hydrate ($\text{Ca}(\text{OH})_2$), and/or the corresponding dolomitic products are used. In these filters, the off-gas to be scrubbed flows through a granular bed of material containing alkaline-earth metal carbonate and/or alkaline-earth metal hydroxide. Here, deposition of the acidic off-gas components on the material (sorbent) containing alkaline-earth metal carbonate and/or alkaline-earth metal hydroxide takes place.

With the aid of off-gas scrubbing, the off-gases containing pollutants can be very substantially scrubbed. A disadvantage, however, is that the consumption of sorbent containing alkaline-earth metal carbonate and/or alkaline-earth metal hydroxide is very high. The moderate efficiency of dry off-gas scrubbing is attributable to the fact that the sorbents do not react fully through. A layer of reaction products, which makes further penetration of the acidic pollutants to be deposited difficult, is formed on the sorbent.

A deficiency of the bed filter technique is the relatively high consumption for the deposition of sulphur oxides (SO_2 and SO_3) and the sealing of the reactive surface of the sorbents by the reaction products being formed, for example calcium sulphite (CaSO_3) and calcium sulphate (CaSO_4).

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Attempts have repeatedly been made to reduce the high sorbent consumption. One method consists in mechanically reprocessing the deposited product, which consists of unreacted sorbent and the reaction products formed, after the off-gas
5 scrubbing. The intent and purpose of the mechanical treatment is to separate the unreactive outer layers. Another method provides intermediate storage of the reaction product with reuse after 1-2 days of storage.

10 All these methods, however, are characterised by insufficient effectiveness in terms of increasing the absorbency of the sorbent.

Increasing the absorbency of the sorbent is intended to mean
15 reducing the amount of sorbent to achieve a particular degree of deposition of the acidic pollutants. A higher absorbency in this case leads to a reduction in the stoichiometric factor.

20 There is significant interest in producing activated materials containing alkaline-earth metal carbonate and/or alkaline-earth metal hydroxide, which have an increased absorbency in relation to sulphur oxides and/or other pollutants in the flue gas.

25

WO 2006/113301 A1 describes a method for increasing the adsorption capability of a lime hydrate containing material which is activated through heating to 475 °C for a duration of four hours. The activated lime hydrate is subsequently

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used for the deposition of sulphur dioxide from an off-gas by an entrained-flow process.

WO 2011/039034 A1 describes a granulated desulphurisation material containing calcium carbonate and lime hydrate. It is used in a temperature range from 100 °C to 900 °C, preferably from 130 °C to 450 °C in the off-gas desulphurisation of combustion engine, in particular also of large ship diesel engines.

10

This object is achieved according to the invention by a method according to claim 1.

In the context of this invention, a material containing alkaline-earth metal carbonate and alkaline-earth metal hydroxide is intended to mean all materials which contain at least one alkaline-earth metal carbonate and alkaline-earth metal hydroxide, or consist of one of these substances. In particular, a material containing alkaline-earth metal carbonate and alkaline-earth metal hydroxide is intended to mean both lime and dolomite derived material. According to a preferred embodiment of the invention, the material containing alkaline-earth metal carbonate and alkaline-earth metal hydroxide contains calcium carbonate, calcium hydroxide, magnesium carbonate and/or magnesium hydroxide.

According to the invention, alkaline-earth metal carbonates are intended to mean all salts and esters of carbonic acid, i.e. in particular secondary carbonates, hydrogen carbonates, orthocarbonates and carbonate esters, which contain an

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alkaline-earth metal. The alkaline-earth metals include inter alia magnesium, calcium, beryllium, strontium and barium.

According to a preferred embodiment of the invention, the alkaline-earth metal carbonate is magnesium carbonate or calcium carbonate, or a mixture thereof. Alkaline-earth metal carbonates particularly suitable are present in products derived from lime and/or dolomite. According to a preferred embodiment of the invention, a material based on limestone and/or dolomite is used as the material containing alkaline-earth metal carbonate.

According to the invention, alkaline-earth metal hydroxides are intended to mean all compounds which contain an alkaline-earth metal and the monovalent group of atoms -OH as a functional group or ion. The alkaline-earth metals include inter alia magnesium, calcium, beryllium, strontium and barium. According to a preferred embodiment of the invention, the alkaline-earth metal hydroxide is magnesium hydroxide or calcium hydroxide, or a mixture thereof. Alkaline-earth metal hydroxides particularly suitable are present in products derived from lime and/or dolomite. According to a preferred embodiment of the invention, a material based on lime hydrate (slaked lime) and/or dolomite hydrate is used as the material containing alkaline-earth metal hydroxide.

Surprisingly, it has been found that the deposition capacity of materials containing alkaline-earth metal carbonate and alkaline-earth metal hydroxide in relation to acidic gas components, in particular sulphur dioxide in flue gas, can be improved when the material is heated to temperatures of between about 200°C and about 850°C. Without wishing to be

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bound by scientific theory, it appears that the heating leads to activation of the sorbents containing alkaline-earth metal carbonate and alkaline-earth metal hydroxide. Thus, a significant increase in the absorbency of materials

5 (sorbents) containing alkaline-earth metal carbonate and/or alkaline-earth metal hydroxide is already achieved by single heating to temperatures of between about 200°C and about 850°C.

10 The method according to the invention thus allows more effective deposition of pollutants and, hence, minimisation of the demand for material (sorbent) containing alkaline-earth metal carbonate and alkaline-earth metal hydroxide in dry flue gas scrubbing.

15

Practical tests have shown that a particularly strong increase in the absorbency of the sorbent can be achieved when the material containing alkaline-earth metal carbonate and alkaline-earth metal hydroxide is heated to temperatures
20 of from about 250°C to about 750°C, preferably from about 250°C to about 700°C, in particular from about 300°C to about 500°C. It has been observed that the activation effect no longer occurs above about 850°C. This is probably because less readily absorbing burnt products are formed at these
25 temperatures. When using lime-derived material, for example, it has been observed that the less readily absorbing calcium oxide is formed at activation temperatures above about 850°C. When heating to temperatures below 200°C, likewise no significant activation of the alkaline-earth metal carbonate
30 and/or alkaline-earth metal hydroxide was observed.

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The heating of the material containing alkaline-earth metal carbonate and alkaline-earth metal hydroxide may be carried out in various ways known to the person skilled in the art.

5 For example, the heating may be carried out in a kiln or by passing over hot off-gas in a fluidised bed or fluid bed, or in bed filters.

The duration for which the material containing alkaline-earth
10 metal carbonate and alkaline-earth metal hydroxide is heated, and therefore activated, may vary in wide ranges. In particular, it has been found that the optimal activation time depends on the material used and the activation temperature selected. The person skilled in the art can
15 determine the optimal activation parameters, in particular activation time and activation temperature, for a particular material by test runs.

For reasons of energy, it is advantageous to limit the
20 duration of the heating. It has been found particularly expedient to heat the material containing alkaline-earth metal carbonate and alkaline-earth metal hydroxide for a duration of from 1 minute to 12 hours, preferably from 10 minutes to 12 hours, particularly preferably from 1 hour to 6
25 hours, in particular from 2 to 5 hours. In the case of very fine-grained materials and/or suitable selection of the activation temperature and an optimised heating method, shorter heating times are also possible.

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According to the invention, the material containing alkaline-earth metal carbonate and alkaline-earth metal hydroxide is activated in a separate step before it is used as a sorbent.

5 Tests have shown that the thermal activation according to the invention also persists when the material containing alkaline-earth metal carbonate and alkaline-earth metal hydroxide is cooled again after the activation. According to one embodiment of the invention, accordingly, the activated
10 material containing alkaline-earth metal carbonate and alkaline-earth metal hydroxide is cooled to room temperature in a further step.

According to the invention, the material containing alkaline-earth metal carbonate and alkaline-earth metal hydroxide is
15 heated in the scope of its use in dry flue gas scrubbing once or continuously to temperatures of from about 300°C to about 500°C. The material containing alkaline-earth metal carbonate and alkaline-earth metal hydroxide may, according to another
20 embodiment, already be contained in a filter ready for use for the flue gas scrubbing when it is heated, particularly a bed filter or a filter cartridge.

According to the invention, all materials based on limestone
25 and/or dolomite, which are suitable for the deposition of acidic components in flue gas, and in particular sulphur dioxide, are suitable in particular as materials containing alkaline-earth metal carbonate and alkaline-earth metal hydroxide. Particularly good results are achieved when using
30 products with a particularly large surface area, derived from

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lime or dolomite, which are specially developed for flue gas scrubbing. According to the invention, calcium hydroxide and/or calcium carbonate, as well as products which partially contain calcium hydroxide and/or calcium carbonate, are used
5 as the material containing alkaline-earth metal carbonate and/or alkaline-earth metal hydroxide.

Practical tests have shown that the thermal activation according to the invention works particularly well for
10 materials which at least partially contain alkaline-earth metal hydroxides. Particularly good activations are reached when the material has an alkaline-earth metal hydroxide content of from about 5 to about 25 wt.%, or from about 10 to about 15 wt.%.

15 The particle size of the material containing alkaline-earth metal carbonate and alkaline-earth metal hydroxide may vary in wide ranges. Particularly good deposition capacities are achieved with granules as well as granulated or pelleted products. The particle sizes of the granules, or granulated
20 or pelleted materials, preferably vary in the range of from about 0.1 to about 50 mm, particularly preferably between about 1 mm and about 10 mm, and in particular between about 2 mm and about 6 mm.

25 The activated product containing alkaline-earth metal carbonate and alkaline-earth metal hydroxide, produced by the method according to the invention, is outstandingly suitable as a sorbent for the absorption of sulphur oxides and/or other pollutants, in dry flue gas scrubbing. Furthermore, the
30 present invention also relates to the use of the product

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containing alkaline-earth metal carbonate and alkaline-earth metal hydroxide produced by the method according to the invention in dry flue gas scrubbing.

5 Practical tests have shown that particularly good deposition capacities are achieved when the material containing alkaline-earth metal carbonate and alkaline-earth metal hydroxide is used as a filler material in a bed filter. In this embodiment of the invention, the gas to be scrubbed
10 flows through a loose granular layer of material containing alkaline-earth metal carbonate and alkaline-earth metal hydroxide, which is used as a filter medium. The particle size range of the material containing alkaline-earth metal carbonate and alkaline-earth metal hydroxide is preferably
15 between about 0.1 mm and about 10 mm, more preferably between about 2 mm and about 6 mm, in particular between about 3 mm and about 5 mm. In this case either the activation may be carried out during operation of the bed filter, or the material containing alkaline-earth metal carbonate and
20 alkaline-earth metal hydroxide is activated beforehand according to the invention, i.e. before it is used as a sorbent in the bed filter.

The flow speeds in the bed filter may vary in wide ranges.
25 For example, speeds of between 0.1 m/s and 5 m/s may be set. Depending on the required degree of deposition and pressure loss, the layer heights may be up to a few metres. Preferred layer heights lie in the range of from about 100 mm to about 500 mm, in particular from about 200 mm to about 400 mm.

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The deposition of particles in bed filters may, according to the invention, take place in a fixed bed (stationary bed), a fluid bed, a migrating bed (moved bed) and a fluidised bed (layer carried by the gas flow). The use of bed filters with
5 a stationary bed is particularly expedient.

According to a preferred embodiment, the operating temperature in the bed filter is increased to temperatures of more than 200°C, and an increase in the absorbency of the
10 material containing alkaline-earth metal carbonate and alkaline-earth metal hydroxide is thus achieved. For many materials containing alkaline-earth metal carbonate and alkaline-earth metal hydroxide, a maximum of the effectiveness may be achieved at an activation temperature of
15 about 400°C.

As shown in Figure 1, in the case of an increase in the activation temperature with subsequent use of the activated material as a filter material in the bed filter, a
20 substantial improvement in the deposition capacity takes place. A maximum of the effectiveness is in this case achieved at about 400°C.

According to another embodiment according to the invention,
25 however, it is likewise possible to carry out the heating of the material containing alkaline-earth metal carbonate and alkaline-earth metal hydroxide directly in the bed filter.

For energy reasons the material containing alkaline-earth
30 metal carbonate and alkaline-earth metal hydroxide is heated

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before its use, for example once, to a temperature of between 300°C and 500°C.

The essential advantages of this procedure according to the
5 invention are as follows:

1. The method is energy-efficient, since the filter does not have to be operated constantly at high temperatures.
- 10 2. The filter can be operated as before at the conventional low temperatures below 200°C, and therefore more cost-efficiently.
- 15 3. The activation by heating may be carried out at the manufacturer of the material (sorbent) containing alkaline-earth metal carbonate and/or alkaline-earth metal hydroxide.
- 20 4. The demand for material containing alkaline-earth metal carbonate and alkaline-earth metal hydroxide can be minimised by more effective deposition.

The method according to the invention will be explained in more detail below with the aid of exemplary embodiments.

25 Example 1

In a laboratory test, the effect of thermal activation on the absorbency of a sorbent for dry flue gas scrubbing was studied. A sorbent was used consisting of granules that
30 contain about 90 wt.% calcium carbonate and about 10% wt.%

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lime hydrate. First, the sorbent was divided into 7 batches of 200 g. The first batch was used as a reference sample, and was not treated further. Batches 2 to 7 were stored for 6 hours in correspondingly thermally regulated kilns at 200°C, 300°C, 400°C, 500°C, 600°C or 900°C. Batches 2, 6 and 7 are comparative examples. The differently activated sorbents were subsequently cooled to room temperature and the cartridge, respectively provided therefor, of a 160 ml laboratory bed filter was filled therewith. For each material, the absorbency was then determined in comparison with the reference material by recording SO₂ permeation curves in the laboratory bed filter. To this end, the bed filters filled with the sample material activated at different temperatures, or reference material, were flowed through at 160 to 170°C by a likewise thermally regulated N₂/SO₂ test gas mixture with an SO₂ concentration of 2000 ppm. The gas in this case flowed through the filter with a speed of 0.1 m/s at a pressure of about 30 to 60 mmWC (residence time about 2 s). Arranged downstream of the filter there was a computer-assisted continuous gas analysis unit (company MSI, Type MSI 2000), which recorded the SO₂ concentration in the flow through the filter. The difference between the SO₂ concentration before the filter (2000 ppm) and after the filter was calculated as the degree of deposition. At time zero, the degree of deposition in all cases was 100%, i.e. the filter material was capable of fully retaining the SO₂ in the test gas flowing through. Beyond a certain time, however, a reduction in the degree of deposition was found, i.e. permeation of SO₂ in the flow through the filter, which is probably due to gradual saturation of the sorbent with SO₂. The greater the

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absorbency of the sorbent is, the longer SO₂ in the test gas is retained in the filter, and the slower the reduction in the values of the degree of deposition, or of the SO₂ permeation takes place. Characteristic values are the times
5 after the start of the test at which the degree of deposition has fallen below 90%, 70% or 50%. These values are plotted in Figure 1 for the untreated reference material and the sample materials activated at different temperatures. Surprisingly, it was found that single thermal activation of the sorbent
10 already led to a strong increase in the absorbency. Thus, a material which has been activated at 400°C shows an increase in the SO₂ absorbency by about 200% compared with the reference material. Even with an activation temperature of only 200°C, slight improvements in the absorbency of the
15 material activated in this way were found. With an activation temperature of 900°C, on the other hand, the absorbency was degraded. Best results were achieved with activation temperatures of from 300 to 500°C.

20 Example 2

In a second test run, the effect of the activation time on the absorbency of the material was studied. To this end, a procedure corresponding to the conduct of the experiments
25 according to Example 1 was adopted. Merely the activation time (residence time in the kiln) was varied. In accordance with Example 1, the SO₂ absorbency of the materials activated for different lengths of time and at different temperatures was studied. The results and the activation conditions are
30 shown in Figure 2. In this case, it is found that, the closer

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the activation temperature is to 400°C, the shorter are the activation times required in order to achieve a relatively good absorbency. For instance, 30-minute activation at 400°C shows approximately the same improvement in the absorbency as
5 12-hour activation at 300°C. The tests furthermore show that, with an optimal activation temperature, short activation times (see Fig. 2, 5-minute activation at 400°C) already lead to significant improvements in the absorbency compared with the reference material. For many activation temperatures, a
10 further improvement in the absorbency is shown with an increasing activation time (cf. Fig. 2, 300°C and 400°C). For an activation temperature of 500°C, on the other hand, 1-hour activation leads to better results than 6-hour activation. As in Example 1, the material activated at 900°C showed inferior
15 absorbency than the reference material.

Example 3

A bed filter filled with a sorbent containing alkaline-earth
20 metal carbonate and alkaline-earth metal hydroxide in a thermal power station was activated once by gas at a temperature of 400°C flowing through for 2 hours. The bed filter was subsequently operated at a regular operating temperature below 200°C. The thermal activation leads to an
25 improvement in the pollutant absorbency of the bed filter.

Example 4 (Comparative Example)

Gas at a temperature of 270°C flows through granules of CaCO_3
30 and Ca(OH)_2 in an industrial fluid-bed process, the granules

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thereby being dried and hardened. By lengthening the residence time, the material was heated beyond the drying point to the hot gas temperature, and thereby activated.

P a t e n t k r a v

- 5 1. Fremgangsmåde til øgning af et materiales absorptionsevne, hvilket materiale indeholder jordalkalicarbonat og jordalkalihydroxid, i forhold til svovloxider og/eller andre skadelige stoffer i røggas, **kendetegnet ved, at** materialet, der indeholder jordalkalicarbonat og jordalkalihydroxid, før dets anvendelse som sorptionsmiddel aktiveres i et separat trin ved opvarmning til 300 °C til 500 °C i et tidsrum på 1 minut til 12 timer, hvor materialet, der skal aktiveres i det separate trin og indeholder jordalkalicarbonat und jordalkalihydroxid, har en 10 jordalkalihydroxid-andel på 5 til 25 vægt-%, og hvor der som materiale, der indeholder jordalkalicarbonat og jordalkalihydroxid, anvendes calciumcarbonat og calciumhydroxid.
- 15 2. Fremgangsmåde ifølge et af de foregående krav, **kendetegnet ved, at** opvarmningen af materialet, der indeholder jordalkalicarbonat og jordalkalihydroxid, gennemføres i et tidsrum på 10 minutter til 12 timer.
- 20 3. Fremgangsmåde ifølge et af de foregående krav, **kendetegnet ved, at** materialet, som indeholder jordalkalicarbonat og jordalkalihydroxid, foreligger som granulat, granalier eller pellets.
- 25 4. Fremgangsmåde ifølge et af de foregående krav, **kendetegnet ved, at** materialet, som indeholder jordalkalicarbonat og jordalkalihydroxid, har en middelpartikelstørrelse på 0,1 til 50 mm, især på 1 til 10 mm.
5. Fremgangsmåde ifølge et af de foregående krav, **kendetegnet ved, at** det aktiverede materiale, som indeholder jordalkalicarbonat og jordalkalihydroxid, køles til en rumtemperatur i et yderligere trin.
- 30 6. Fremgangsmåde ifølge et af de foregående krav, **kendetegnet ved, at** materialet, der indeholder jordalkalicarbonat og jordalkalihydroxid, befinder sig i et filter, især et packed bed-filter eller en filterpatron.
- 35 7. Anvendelse af et aktiveret materiale, som fremstilles i henhold til en fremgangsmåde ifølge et eller flere af kravene 1 til 6 og indeholder jordalkalicarbonat og jordalkalihydroxid, til absorption af svovloxider og/eller andre skadelige

stoffer til tør røggasrensning.

5 **8.** Anvendelse ifølge krav 7, **kendetegnet ved, at** det aktiverede materiale, som indeholder jordalkalicarbonat og jordalkalihydroxid, anvendes som fyldmateriale i et packed bed-filter.

10 **9.** Anvendelse ifølge krav 8, **kendetegnet ved, at** det aktiverede materiale, der indeholder jordalkalicarbonat og jordalkalihydroxid, fremstilles ved opvarmning af materialet i packed bed-filteret.

10. Anvendelse ifølge krav 7, **kendetegnet ved, at** det aktiverede materiale, som indeholder jordalkalicarbonat og jordalkalihydroxid, anvendes i en moving eller fluidized bed.

15 **11.** Anvendelse ifølge krav 7, **kendetegnet ved, at** det aktiverede materiale, som indeholder jordalkalicarbonat og jordalkalihydroxid, anvendes i en flyvestrømsproces.

Fig. 1

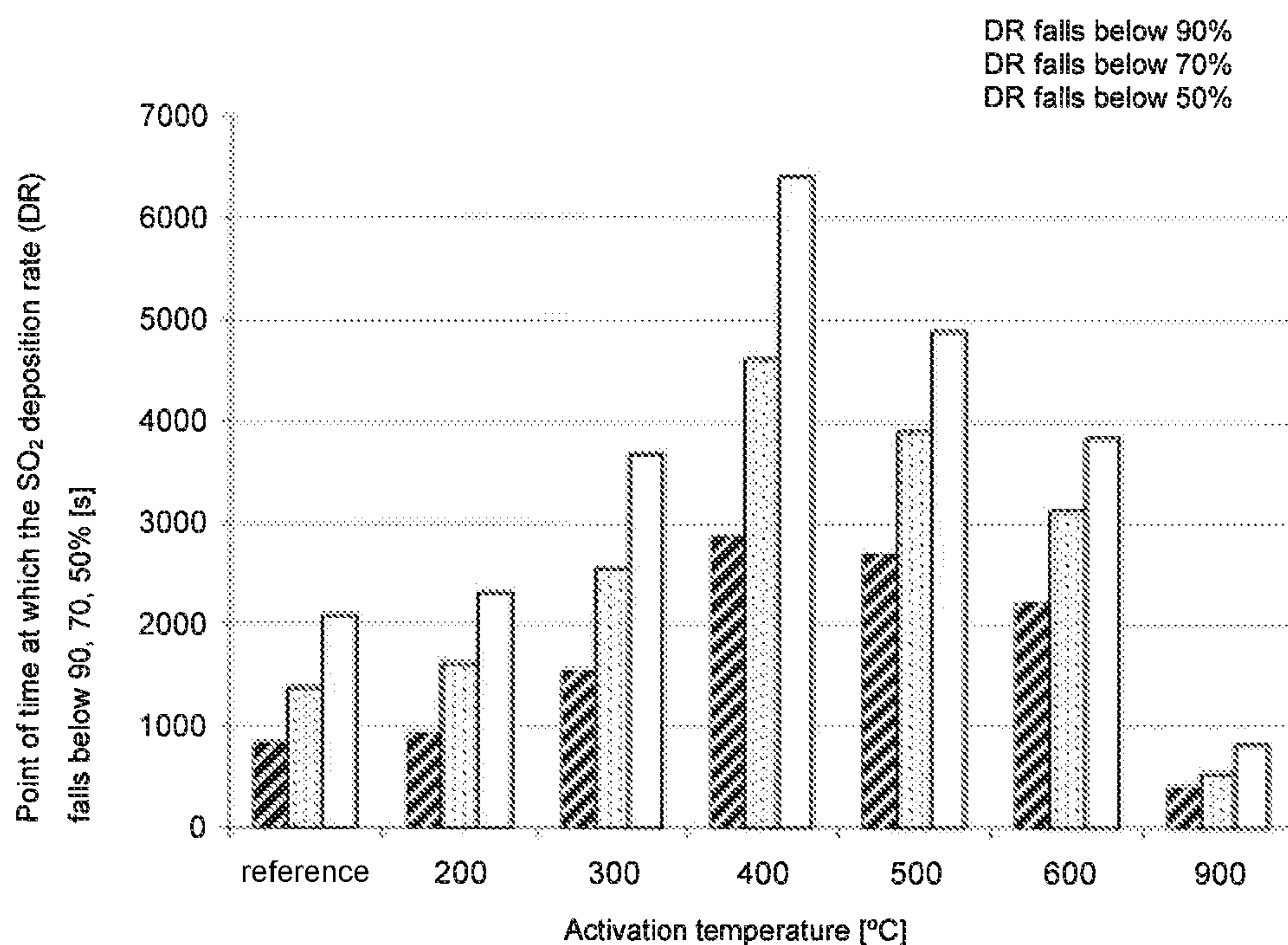


Fig. 2

