

**CHEMICAL REACTOR WITH KNITTED WIRE MESH FABRIC AS A HOLDING DEVICE FOR PARTICLES**

5 The present invention relates to a chemical reactor for the heterogeneously catalysed reaction of a fluid, having an improved retaining means for inert particles and/or catalyst particles through which fluid flows. It also relates to a method for carrying out chemical reactions by means of such a reactor and the use of wire mesh knitted fabric in chemical reactors.

10 The hydrogenation of nitroaromatic compounds to form the corresponding amines in a fixed-bed reactor which is flowed through axially may take place in a multi-stage process under adiabatic reaction conditions. Such hydrogenation processes take place in periodic cycles, wherein production cycles alternate with regeneration cycles, for example in which carbonaceous deposits are removed by being burnt off.

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Reactors for such reactions are typically cylindrical in shape and are equipped with a wedge wire screen which acts as a substrate for the fixed-bed catalyst (Heterogeneous Catalysis in Industrial Practice, 2<sup>nd</sup> Ed. 1996, page 474). These wedge wire screens must bear the weight of the catalyst as well as the additional load  
20 produced by the pressure loss via the catalyst fill.

Wedge wire screens as described for example in US 2,915,375 are often used. They consist of V-shaped wires arranged in parallel which are in turn supported and fixed so that openings are produced, of which the width is smaller than the particle size of  
25 the material to be supported, said openings widening downwardly so as, if necessary, to carry off downwardly any material infiltrating between the wires and to prevent blocking the wedge wire screen.

Owing to the periodic operation of the reactors, the screen is subjected to thermal  
30 stresses, which lead to damage. In particular this is the case if exothermic reactions are carried out under adiabatic conditions, that is to say the reaction heat which is released is absorbed by the reaction gas and therefore extreme jumps in temperature are produced inside the reactor.

In these cases, the V-shaped wires originally arranged in parallel may become deformed, even after only a few production cycles, until some wires are broken or torn off. Repairs of a screen damaged in this way are expensive and are not  
5 successful in the long term. To minimize this problem, the wedge wire screens are generally fixed on a floatingly mounted support ring which has a clearance to the reactor wall so as to compensate for thermal expansions.

In particular in larger reactors an absolutely flat bearing surface (supporting ring) for  
10 this ring can only be manufactured at substantial expense, if at all. It is also difficult to produce large reactors so as to be precisely round. Owing to these problems, there is often a loss of catalyst such that the catalyst trickles out as a result of damage to the wedge wire screen or through openings between the wedge wire screen and the reactor wall or supporting ring.

15 The problems are intensified further by the use of increasingly smaller catalyst particles, which are generally more efficient but trickle out more easily through small cracks. Lumpy recesses are produced in the surface of the fill owing to the catalyst loss. The reaction gas then preferably flows through the areas with the recesses so  
20 that a non-uniform gas flow is produced, until reaching a bypass of unreacted starting material. Furthermore, the catalyst which has migrated on the rear face of the wedge wire screen may cause damage to apparatuses located therebehind.

In order to avoid a penetration of catalyst on the other side of the wedge wire screen,  
25 the catalyst may be deposited on a complex, multi-layered fill formed of inert particles, wherein the particle diameter in the layer which lies closest to the catalyst is somewhat greater than the particle diameter of the catalyst and then increases with each layer (Fixed-Bed Reactor Design and Diagnostics, 1990, Fig. 1.1, page 4).

30 Rather large inert particles, which cannot trickle through the screen, in the case of relatively small cracks, thus first lie on the wedge wire screen. The drawback of this solution is that a not insignificant part of the reactor is filled with inert material and is no longer available for the actual catalyst, and the introduction of the different

layers into the reactor is demanding. If the inert material is to be re-used when the reactor is re-filled, the material removed from the reactor must be screened in a multi-stage process so as to separate the differently sized particles from one another.

- 5 The object of the invention was therefore to provide a reactor in which a penetration of catalyst onto the rear face of the supporting structure for the catalyst is prevented, even in the case of repeated thermal stressing, wherein the supporting structure is to be cost effective, maintenance costs and complexity are to be low and the process of filling the reactor with catalyst fill is to be simple.

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In accordance with the invention, the object is achieved by a chemical reactor for the heterogeneously catalysed reaction of a fluid, comprising a particle retaining means through which fluid flows, wherein the side of the retaining means arranged upstream in terms of the direction of flow of the fluid comprises a wire mesh knitted  
15 fabric and the average clear mesh size of the wire mesh is less than the mean particle size  $x_{50,3}$ , which is determined by a screen analysis carried out using a vibration screening machine having a screen set under the observance of the provisions of DIN 66165 (version of April 1987).

- 20 For example, *knitted fabrics* are known from the textiles and jewellery industries. Quite generally, a knitted fabric denotes a fabric in which a loop formed by means of a thread or a plurality of threads is looped through another loop, thus forming the "knits". A distinction is to be made from woven fabrics, in which the surface is produced by weaving two thread systems. Knitted fabrics have a greater stretching  
25 ability and resilience than woven fabrics. Knitted fabrics can be divided into machine knit fabrics, warp knitted fabrics and weft knitted fabrics. All three are suitable in principle to carry out the invention. However, machine knit fabrics and/or weft knitted fabrics are more preferably used, since these are more resilient than warp knitted fabrics (which are resilient in any case). In the present invention, a *wire mesh*  
30 *knitted fabric* is used, that is to say the knitted fabric according to the invention is made from wires and/or metal threads. Within the scope of the present invention, the term "wire mesh" includes all types of knitted fabric, that is to say even warp knitted fabrics. The wire mesh is produced using wire processing machines, which are

known *per se* and are conventional for example for applications in the automotive industry, process engineering and environmental engineering (for example wire mesh knitting machines). For example, wire meshes by DHD Technology are suitable for carrying out the present invention.

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Owing to the flexibility of the knitted fabric, it is ensured that this is not damaged with periodic operation at different temperatures. At the same time, the three-dimensional structure of the knitted fabric prevents too many meshes from becoming clogged by particles and the gas flow being impeded.

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Within the meaning of this invention, *particles* are to be understood to mean both *catalyst particles* (for example a noble metal catalyst carried on aluminium oxide balls) and *inert particles* (for example aluminium oxide balls). Particles are preferably used which are at least substantially spherical, that is to say any deviations from perfect sphere geometry are so small that the particles behave like perfect spheres in practice. In principle however, the invention can also be applied to asymmetrical particles. The manufacturer's specifications should be used as the first point of reference for the mean particle size.

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20 A "particle size analysis" is necessary to precisely determine the *mean particle size*. Within the scope of this invention, this occurs by screen analysis. In accordance with the invention the mass-based value (" $x_{50,3}$ ") is used as the mean particle size.

Within the scope of the present invention, in order to determine the mean particle size a representative sample of the particles to be supported is first subjected to a screen analysis and the result is assessed based on mass. The screen analysis is carried out using a vibration screening machine (for example the AS 200 digit model by Retsch), in which the analysis screens are arranged one above the other with increasing mesh size to form a screen set. The selection of the analysis screens (diameter and mesh size) primarily depends on the amount of material to be screened and the expected particle size distribution (preliminary tests may be necessary). The number of screens and the gradations of the nominal opening widths should be

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selected so that, where possible, the entire particle spectrum of the sample is divided into fractions. When carrying out the screen analysis it should be ensured that the maximum amount of material to be screened passes through (optimal screening out efficiency). If necessary (for example if new particles are to be used of which there is  
5 no working experience), suitable screening times and amplitudes have to be determined by way of experiment in preliminary tests. A first point of reference for the amplitude is provided by observing the movement of the material to be screened. This should be neither too weak nor too strong. The optimal screening time is reached when the mass of material passing through the screen changes within a  
10 minute by less than 0.1% of the feed amount (DIN 66165, version of April 1987). A person skilled in the art is familiar with the methods outlined briefly here.

The screen analysis delivers the particle size distribution of the measured particles as its result. The result is preferably illustrated in a graph, in which the mass fraction of  
15 the individual fractions ("p<sub>3</sub>") is plotted in a bar chart, and the cumulative frequency curve ("Q<sub>3</sub>") from the fraction percentages is plotted over the nominal screen opening widths (x). A person skilled in the art can easily calculate the mean particle size  $x_{50,3}$  (that is to say 50% by mass of the particles are smaller than the corresponding value x) either manually or preferably by computer-supported  
20 evaluation programs.

If this first result demonstrates sufficient homogeneity of the sample, the mean particle size  $x_{50,3}$  thus determined is therefore the value required to carry out the invention. *Sufficient homogeneity* is understood in this regard to mean that the  
25 particle size distribution is monomodal and that in each case 0.1% by mass at most of all particles lie in the range between  $\frac{1}{2} x_{50,3}$  and  $\frac{2}{3} x_{50,3}$  or in the range between  $1\frac{1}{2} x_{50,3}$  and  $1\frac{1}{2} x_{50,3}$ . Furthermore, no particles should be smaller than  $\frac{1}{2} x_{50,3}$  or larger than  $1\frac{1}{2} x_{50,3}$ . If this first result does not demonstrate sufficient homogeneity of the sample within the meaning above, the particles to be supported are thus homogenized  
30 by screening on an industrial scale, that is to say dust fractions and fragments which can no longer be used are separated. This is carried out until the aforementioned requirements of homogeneity are met.

For example, the chemical reactor may be a fixed-bed reactor which is flowed through axially, as is used in the hydrogenation of nitrobenzene to form aniline. The fluid to be reacted in the reactor may be liquid, gaseous or supercritical. It may contain one reactant or a mixture of several reactants.

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As will be described below in greater detail, the particles detained by the wire mesh knitted fabric may be both catalyst particles and inert particles.

The type of wire mesh knitted fabric is not specified further to start with. It may be a single-core or multi-core wire which has been knitted in any desired manner. For example, the knitted meshes may thus contain up to 5 round, flat, smooth and/or wavy threads. Knitted materials may be, *inter alia*, steel, high-grade steel such as 1.4571, copper, Monel (2.4360) or aluminium. In addition to wire meshes from flat-bed knitting machines, dual-layer wire mesh webs may also be used, for example, which are formed by pressing together wire mesh tubes produced using circular knitting machines in relation to a folding plane extending through the central axis of the wire mesh tubes.

In accordance with the invention the average clear mesh size of the wire mesh is smaller than the mean particle size  $x_{50,3}$  of the particles which are intended to be received in the retaining means. It denotes the inner spacing (not the spacing from one wire centre to another wire centre) of two adjacent mesh branches at the point of the greatest expansion of a mesh. The ratio between mesh length, that is to say the spacing between two adjacent mesh heads, and mesh width is preferably between 4:1 and 0.5:1, more preferably between 2:1 and 1:1. The average clear mesh width thus denotes the average value of all clear widths of individual meshes of a wire mesh knitted fabric. The average clear mesh width is set during production of the wire mesh knitted fabric by methods known to a person skilled in the art (for example number, spacing and thickness of the needles at the knitting head, number and thickness of the wires, etc.). The use of knitted fabric having irregular mesh sizes (that is to say differences in the mesh sizes which are beyond normal manufacturing tolerances) is not preferable, but is also possible in principle. In this case the clear mesh size of the largest mesh is preferably smaller than the mean particle size  $x_{50,3}$ .

The particle retaining means may, in the simplest case, be formed merely by wire mesh knitted fabric mounted in the reactor. However, it is preferable for the wire mesh knitted fabric to be supported by a support so as to withstand the prevailing mechanical stresses. Both the support and the wire mesh knitted fabric are thus part of the particle retaining means. The catalyst bed is formed from this, together with the catalyst particles.

The position of the wire mesh knitted fabric in the particle retaining means in the reactor through which fluid flows is determined by the function of the knitted fabric to detain particles. It is thus located on the upstream side of the retaining means.

With the reactor according to the invention, it is also advantageous that a simple wedge wire screen can be used as a support. The use of a wedge wire screen, which is costly and complex in terms of manufacture, is not necessary, and a simple grate may be used instead. The knitted fabric is placed over the wedge wire screen or the grate and is fixed if necessary.

The knitted fabric applied now performs the function of catalyst retention. As a result, not only can damage to the wedge wire screen be endured, but the openings in the wedge wire screen can be designed so as to be larger in the first place.

This results in a further advantage: owing to the larger openings it is possible to increase the thickness of the wedge wire screen or grate without running the risk of blockages forming. The rigidity of the structure bearing the knitted fabric is in turn increased so that larger amounts of catalyst can be supported and also higher pressure losses and the loads produced thereby can be withstood. It is even possible to dispense with the fill with inert material, whereby even greater amounts of catalyst can be used.

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Embodiments of the invention will be illustrated hereinafter, wherein the embodiments can be freely combined with one another, unless otherwise clearly indicated by the context.

In one embodiment of the reactor according to the invention the average clear mesh size of the wire mesh is  $\geq 20\%$  to  $\leq 80\%$  of the mean particle size  $x_{50.3}$ . This value is preferably  $\geq 30\%$  to  $\leq 60\%$  and more preferably  $\geq 40\%$  to  $\leq 50\%$ . An undesirable pressure loss during operation of the reactor caused by particles caught in the meshes is thus particularly prevented.

In a further embodiment of the reactor according to the invention the extensibility of the wire mesh knitted fabric under mechanical and/or thermal load parallel to the surface is  $\geq 1\%$ , preferably  $\geq 2\%$ . The extensibility parallel to the surface should not exceed 50%, preferably 25%, preferably 10%. It is thus particularly ensured that the knitted fabric adapts to the thermal expansions of the reactor without becoming damaged and without particles falling through the meshes.

In a further embodiment of the reactor according to the invention the wire diameter of the wire mesh knitted fabric is  $\geq 0.03$  mm to  $\leq 1$  mm. The diameter of the wire from which the knitted fabric is knitted is preferably  $\geq 0.1$  mm to  $\leq 0.5$  mm and more preferably  $\geq 0.2$  mm to  $\leq 0.3$  mm. It is thus particularly ensured that the wire has sufficient stability with simultaneously low weight and large area through which the fluid can flow.

In a further embodiment of the reactor according to the invention, the height of the wire mesh knitted fabric in the particle retaining means is  $\geq 3$  mm to  $\leq 100$  mm. For example, this can be achieved by applying the knitted fabric, folded many times, to a support. The height is preferably  $\geq 5$  mm to  $\leq 80$  mm and more preferably  $\geq 10$  mm to  $\leq 50$  mm. The passage of particles on the upstream side of the knitted fabric is thus prevented particularly reliably and free channels for the circulating fluid always remain.

In a further embodiment of the reactor according to the invention the wire mesh knitted fabric is a wire mesh knitted fabric deformed by mechanical action. For this purpose the knitted fabric may be rolled or passed through between two cylinder rolls or toothed wheels. Knitted fabric deformed in this manner has either a low height so

as to maximize the available space for catalyst in the reactor (rolled), or has a thicker three-dimensional structure (passed through toothed wheels) so that a greater support formed of knitted fabric can be achieved at low material cost. This then experiences a particularly low pressure loss and a has particularly low tendency to block the mesh.

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In a further embodiment of the reactor according to the invention the wire mesh knitted fabric is arranged on a support and the support comprises through-openings in the direction of flow of the fluid. As already illustrated, preferred supports are wedge wire screens and gratings. The support can be floatingly mounted on a supporting ring on the inner face of the reactor wall so as to compensate for the thermal expansion during operation.

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Wire mesh knitted fabric is preferably additionally arranged in this case between the support and the wall of the reactor. A clearance to the passage of particles created for the purposes of compensation of the thermal expansion is thus ensured. This wire mesh knitted fabric is preferably a metal sealing cord made of knitted fabric. It is favourable if it consists of the same material as the knitted fabric located on the support.

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In a particularly preferred embodiment of the reactor according to the invention the wire mesh knitted fabric additionally located between the support and the wall of the reactor is arranged in a U-shaped manner and a resiliently biased further wire mesh knitted fabric is introduced into the recess formed thereby. For example, this may be a metal sealing cord made of knitted fabric.

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The branches of the "U" may each, or together extend upright so as to be adapted to the shape of the reactor wall, for example. However, it is also possible for the branches to take a horizontal course, at least in part. They may lie on a wedge wire screen or grating. Further knitted fabrics can then be laid thereon.

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The type of biasing may be both compression and expansion. The sealing cord is preferably compressed before insertion so that it then expands. The two upwardly protruding sides of the "U" are then pressed against the support and/or a supporting

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ring as well as the reactor wall. Inert or catalyst particles are thus reliably and lastingly prevented from passing through any cracks between the support, supporting ring and reactor wall or bearing surface of the supporting ring. The metal scaling cord is resilient and compensates for the accuracy of the fit between the wedge wire screen and the reactor wall, and for the changes in the width of the clearance between the wedge wire screen and the reactor wall, even during temperature changes.

In a further embodiment of the reactor according to the invention catalyst particles are arranged on the wire mesh knitted fabric in the retaining means. Suitable catalysts are described, *inter alia*, in EP 0 011 090 A1 and comprise:

- (a) 1 to 100 g/l of a support of at least one metal of groups 8 to 12 of the Periodic Table, and
  - (b) 0 to 100 g/l of a support of at least one transition metal of groups 4 to 6 and 12 of the Periodic Table, as well as
  - (c) 0 to 100 g/l of a support of at least one metal of the primary group elements of groups 14 and 15 of the Periodic Table
- on an aluminium oxide support with a BET surface of less than 20 m<sup>2</sup>/g.

In a further embodiment of the reactor according to the invention a layer of particles inert to the fluid is arranged on the wire mesh knitted fabric in the retaining means and catalyst particles are arranged on this layer. Particles which are inert to the fluid are catalytically inactive and do not react with the fluid. If such particles are placed on the knitted fabric as an intermediate layer, it is possible to use smaller and therefore more active catalyst particles.

A further subject matter of the present invention is a method for reacting a fluid, wherein the reaction is carried out in a reactor according to the invention in the presence of heterogeneous catalyst particles, and the catalyst particles are arranged in the retaining means for catalyst particles. Also included is the case in which an

intermediate layer formed of inert particles is located in the catalyst bed. The advantage of the method according to the invention lies in particular in the longer service lives before servicing is again required.

5 In one embodiment of the method according to the invention the reactor is heated, once the reaction has finished and in the presence of oxygen, to a temperature of  $\geq 200\text{ }^{\circ}\text{C}$  to  $\leq 400\text{ }^{\circ}\text{C}$ . It has been found that reactors according to the invention also cope advantageously with this burning off of carbon deposits.

10 In a further embodiment of the method according to the invention, the fluid comprises aromatic nitro compounds and hydrogen. In particular, the hydrogenation of nitrobenzene to form aniline may be involved. The method according to the invention may be operated for longer, until there is an increased nitrobenzene content (nitrobenzene breakthrough) in the end product.

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In a further embodiment of the method according to the invention the reaction is carried out adiabatically. This method is described for example in EP 0 696 573 A1 and EP 0 696 574 A1. The method according to the invention copes advantageously with the jumps in temperature which occur with adiabatic reactions.

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An additional aspect of the present invention is the use of wire mesh knitted fabric as a retaining means for catalyst particles and/or inert particles in chemical reactors. With regard to details and preferred embodiments, reference is made to the explanations regarding the reactor according to the invention in order to avoid repetitions.

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The invention will now be described further with reference to the following drawings and examples, but is not limited thereto. In the drawings:

30 Fig. 1 shows a chemical reactor according to the invention;

Fig. 2 shows a further chemical reactor according to the invention;

Fig. 3 shows a further chemical reactor according to the invention;

Fig. 4 shows a further chemical reactor according to the invention;

5 Fig. 5 shows a further chemical reactor according to the invention;

Fig. 6 shows a further chemical reactor according to the invention;

Fig. 7 shows a detail of the reactor shown in Fig. 6.

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Fig. 1 is a schematic cross-sectional view of a chemical reactor according to the invention, as can be used for example as a fixed-bed reactor, which is flowed through axially, for the hydrogenation of nitrobenzene to form aniline. The direction of flow of a reaction fluid extends vertically in the drawing from top to bottom.

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A supporting ring 20 is formed peripherally on the inner face of the reactor walls 10. A support 30 formed as a wedge wire screen is mounted floatingly on this supporting ring 20. The openings 40 in this wedge wire screen widen in the direction of flow. The wedge wire screen may be defined by a peripheral support ring.

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Wire mesh knitted fabric 50 is located on the support 30, and wire mesh knitted fabric 60 is additionally introduced into the clearance which is formed between the support 30 and the reactor wall 10.

25 The retaining means formed by the support 30 and the wire mesh knitted fabric 50, 60 may receive catalyst particles in the region 70. The wire mesh knitted fabric 50 is located in this case on the side of the retaining means arranged upstream in terms of the direction of flow of a reaction fluid.

30 Fig. 2 shows the same cross-sectional view of a chemical reactor as Fig. 1, with the difference that catalyst particles 80 are now arranged on the wire mesh knitted fabric 50. The average clear mesh size of the wire mesh knitted fabric 50 is dimensioned in this case in such a way that the catalyst particles 80 are detained.

Fig. 3 shows further designs of the chemical reactor described above in Figs 1 and 2. In this case additional wire mesh knitted fabric 60 is inserted in a U-shaped manner into the clearance formed between the support 30 and the reactor wall 10. Further  
5 resiliently biased wire mesh knitted fabric 90, for example a metal sealing cord made of knitted fabric, is inserted into the recess formed hereby. The wire mesh knitted fabric 60 is fixed by the biasing.

A layer formed of inert particles 100 is also located between the layer formed of  
10 catalyst particles 80 and the wire mesh knitted fabric 50.

Fig. 4 shows a device similar to the reactor shown in Fig. 1, in which device the wire mesh knitted fabric 50 lying on the support 30 is formed continuously as far as the reactor wall 10. Additional, round wire mesh knitted fabric 60 is arranged beneath  
15 and also seals the clearance between the support 30 and the reactor wall 10. This design has the advantage that the knitted fabric layer 50 arranged above can be lifted more easily for maintenance and inspection.

The arrangement in Fig. 5 corresponds to that in Fig. 5, with the difference that a  
20 layer of catalyst particles 80 has now been placed on the wire mesh knitted fabric 50, similarly to Fig. 2.

Fig. 6 shows a reactor similar to Fig. 3, wherein the catalyst particles 80 are arranged on a layer of inert particles 100. The space between the support 30 and the reactor  
25 wall 10 is sealed by the additional knitted fabric 60. This knitted fabric 60 is laid in a U-shaped manner, wherein the side of the "U" facing the reactor wall 10 stands upright and the other side of the "U" follows the course of the shape of the support 30, that is to say is first upright and then extends further horizontally on the support 3. The wire mesh knitted fabric 50 is then laid upon this.

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This course is illustrated in Fig. 7 to provide a better overview. This figure corresponds to Fig. 6, wherein only the reactor walls 10, the supporting ring 20, support 30, openings 40 and the additional knitted fabric 60 are shown. As can be

seen in Fig. 6, a resiliently biased wire mesh knitted fabric 90 can again be inserted into the recess formed by the "U" of the additional knitted fabric 60.

### Examples

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A reactor made of high-grade steel which was fed with starting products via an evaporator was used as an experimental system for the exemplary reactions (hydrogenation of nitrobenzene to form aniline under adiabatic reaction conditions). Nitrobenzene was pumped into the evaporator from above using metering pumps.

- 10 The hydrogen was fed from below into the evaporator, heated to approximately 250 °C, so that the nitrobenzene pumped in from above could evaporate in the counterflow. The hydrogen supply was controlled before the evaporator by a mass flow rate controller. The experimental system was operated at 4 bar and the temperature of the reaction gas was brought to 240 °C in a heat exchanger before  
15 entry into the reactor.

- A 400 mm tall fill of the catalyst was placed inside the reaction tube. Details regarding the respective design of the supporting structure, which was replaced before each experiment, can be found in the corresponding descriptions of the  
20 examples. Once exited from the reactor, the reaction product was cooled with water. The non-volatile components were thus condensed out and separated from the gaseous components in a subsequent separator. The liquid components were guided from the separator into a product collection container, where they were collected. A sampling point was located before the collection container, at which point samples of  
25 the product could be taken at regular time intervals. These were examined by gas chromatography for the presence of nitrobenzene. If the nitrobenzene content in the raw product exceeded 500 ppm, the reaction was terminated and the catalyst was regenerated.

- 30 The charge in all example tests was set at  $1 \text{ g}_{\text{nitrobenzene}}/(\text{ml}_{\text{catalyst}} \cdot \text{h})$  and the hydrogen:nitrobenzene ratio was set at approximately 80:1. The catalyst was an aluminium-oxide-supported metal catalyst containing  $9 \text{ g/l}_{\text{support}}$  Pd,  $9 \text{ g/l}_{\text{support}}$  V,  $3 \text{ g/l}_{\text{support}}$  Pb on  $\alpha$ -aluminium oxide (see EP 0 011 090 A1). The mean particle size  $x_{50,3}$

of catalyst particles and, if necessary, of inert particles was determined by screen analysis. Screens having nominal screen opening widths of 1.25 mm, 1.4 mm, 1.6 mm, 1.7 mm and 1.8 mm ("small balls", catalyst particles, particle size between 1 mm and 2 mm) and of 2.5 mm, 3.0 mm, 3.5 mm, 4.0 mm and 4.5 mm ("large balls", inert material, particle sizes between 3.0 mm and 5.0 mm) were used in this case. The respective  $x_{50,3}$  values for catalyst and inert particles were determined to be 1.5 mm and 4 mm.

In order to carry out the reaction, the reactor was inactivated in each case using nitrogen and charged with hydrogen over a period of 48 h at 240 °C. The nitrobenzene feed was then begun and increased over 5 hours to the above-mentioned value.

To carry out the regeneration, the reactor was again inactivated using nitrogen, heated to 270 °C and then an air flow was applied to burn off any coking. This was carried out until it was no longer possible to determine any heat loss and the CO<sub>2</sub> content in the exhaust gas flow had fallen to less than 0.2% (determined by IR photometry).

In each example, eight cycles consisting of reaction and regeneration were first carried out so that the reactor and its contents were heated first by ambient temperature and were then subjected to a number of temperature fluctuations between 200 and 500 °C. In the subsequent cycle (referred to as the test cycle hereinafter) the moment of the nitrobenzene breakthrough was established and the reactor was cooled to ambient temperature after regeneration and opened for inspection.

Comparative example V-1: (Euroslot wedge wire screen, catalyst)

In this example an adiabatic hydrogenation of nitrobenzene to form aniline was carried out in a reactor which was equipped with a self-supporting wedge wire screen for the catalyst bed. The V-profile wire of the wedge wire screen had a width of 1.8

mm and a height of 3.7 mm and the gap width between the V-profile wires was 0.65 mm. The construction of the V-profile wires was normal, that is to say not inverted.

5 The wedge wire screen was formed of parallel segments. Each segment lay on a supporting structure and was screwed to the next support. The joints protruded beyond the screen surface. Corrosion and bending of the profiles and supporting profiles were prevented by using austenitic steel. The outer supporting ring of the screen was mounted floatingly on a bearing ring located on the reactor wall. A clearance of approximately 1 cm was formed between the supporting ring and the  
10 reactor wall in the cold state. The catalyst fixed bed, of which the catalyst particles had a mean particle size  $x_{50,3}$  of 1.5 mm, came to rest on the wedge wire screen.

In the test cycle, nitrobenzene with a concentration of more than 500 ppm was found in the reaction product after just four days of reaction time. During inspection of the  
15 reactor, deep lumps were found in the catalyst bed. V-profile wires of the wedge wire screen had become detached beneath the lumps. The catalyst bed also exhibited a recess along the reactor wall.

The amount of catalyst missing from the catalyst fixed bed was found beneath the  
20 wedge wire screen on the reactor base and in the subsequent apparatus of the system.

Comparative example V-2: (Eurosloth wedge wire screen, inert material, catalyst).

The test was carried out according to that of Example V-1, wherein the supporting  
25 screen was additionally covered with two 5 cm tall layers of inert material. The first layer applied to the screen consisted of aluminium oxide balls measuring 6 mm in diameter, whereas the layer arranged on top consisted of aluminium oxide balls measuring 4 mm in diameter. The catalyst fill was arranged on this second layer, the catalyst particles of which fill had a mean particle size  $x_{50,3}$  of 1.5 mm.

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In the test cycle, nitrobenzene with a concentration of more than 500 ppm was found in the reaction product after approximately 36 days of reaction time. During inspection of the reactor, no lumps were found in the catalyst bed, although damage

to the supporting screen was again observed. The catalyst bed did not exhibit a recess along the reactor wall. Neither catalyst nor inert material was found on the reactor base or in the subsequent apparatus.

- 5    Example according to the invention E-1: (wedge wire screen, wire mesh, inert material, catalyst).

The test was carried out according to that of comparative example V-1, wherein the wedge wire screen was additionally covered by a wire mesh knitted fabric made of  
10    endless wire. The knitted fabric had a wire diameter of 0.23 mm, was knitted twice and folded in three layers. The knitted fabric was produced from high-grade steel and the average clear mesh size was 0.65 mm. The knitted fabric was fixed to the wedge wire screen by means of bolts.

- 15    The knitted fabric was coated with a 5 cm tall layer of inert material formed of aluminium oxide balls which had a mean particle size  $x_{50.3}$  of 4 mm.

The catalyst fixed bed, of which the catalyst particles had a mean particle size  $x_{50.3}$  of 1.5 mm, came to rest on the inert material.

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In the test cycle, nitrobenzene with a concentration of more than 500 ppm was found in the reaction product after approximately 38 days of reaction time. During inspection of the reactor, no lumps were found in the catalyst bed, although damage to the supporting screen was again observed. The catalyst bed did not exhibit a recess  
25    along the reactor wall. Neither catalyst nor inert material was found on the reactor base or in the subsequent apparatus.

- Example according to the invention E-2: (wedge wire screen, wire mesh, inert material, met. sealing cord, cat.).

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The test was carried out according to that of Example E-1, wherein a metal sealing cord formed of wire mesh knitted fabric, having a wire diameter of 0.23 mm and an average clear mesh size of 0.65 mm, was additionally inserted into the clearance

between the outer supporting ring of the wedge wire screen and the reactor wall. The sealing cord was still flexible, even under the reaction conditions, and had a diameter of 1.2 cm so that it was fixed in the clearance by its own spring tension.

- 5 In the test cycle, nitrobenzene with a concentration of more than 500 ppm was found in the reaction product after approximately 38 days of reaction time. During inspection of the reactor, no lumps were found in the catalyst bed, although damage to the supporting screen was again observed. The catalyst bed did not exhibit a recess along the reactor wall. Neither catalyst nor inert material was found on the reactor  
10 base or in the subsequent apparatus.

Example according to the invention E-3: (simple grate, wire mesh, metal sealing cord, catalyst).

- 15 In this example an adiabatic hydrogenation of nitrobenzene to form aniline was carried out in a reactor which was equipped with a simple grate for the catalyst bed. The average mesh size of the grate was 30 x 10 mm. Corrosion and bending of the profiles and supporting profiles were prevented by using austenitic steel. The outer supporting ring of the grate was mounted floatingly on a bearing ring located on the  
20 reactor wall. A clearance of approximately 1 cm was formed between the supporting ring and the reactor wall in the cold state. A metal sealing cord formed of wire mesh knitted fabric having a wire diameter of 0.23 mm and an average clear mesh size of 0.65 mm was inserted into this clearance. The sealing cord was still flexible, even under the reaction conditions and had a diameter of 1.2 cm so that it was fixed in the  
25 clearance by its own spring tension.

- The grate was covered with a wire mesh knitted fabric made of endless wire. The knitted fabric had a wire diameter of 0.23 mm, was knitted twice and folded in three layers. The knitted fabric was produced from high-grade steel and the average clear  
30 mesh size was 0.65 mm. The knitted fabric was fixed to the grate by means of bolts.

The knitted fabrics were covered with a 5 cm tall layer of inert material made of aluminium oxide balls which had a mean particle size  $x_{50,3}$  of 4 mm.

The catalyst fixed bed, of which the catalyst particles had a mean particle size  $x_{50,3}$  of 1.5 mm, came to rest on the inert material.

- 5 In the test cycle, nitrobenzene with a concentration of more than 500 ppm was found in the reaction product after approximately 900 h of reaction time. During inspection of the reactor, no lumps were found in the catalyst bed and the average clear mesh size of the supporting grate was greater than the diameter of the catalyst or inert material particles. The catalyst bed did not exhibit a recess along the reactor wall.
- 10 Neither catalyst nor inert material was found on the reactor base or in the subsequent apparatus.

Example according to the invention E-4: (as Example E-3, but with increased catalyst fill).

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- The test was carried out according to that of Example E-3, but with the difference that the height of the catalyst fill was increased to 50 cm. The same amount of nitrobenzene was reacted per unit of time as in the comparative examples and examples E-1 to E-3. A volume charge of the catalyst of only 0.8
- 20  $\text{g}_{\text{nitrobenzene}}/(\text{ml}_{\text{catalyst}} \cdot \text{h})$  instead of 1  $\text{g}_{\text{nitrobenzene}}/(\text{ml}_{\text{catalyst}} \cdot \text{h})$  was thus provided in this case.

- In the test cycle, nitrobenzene with a concentration of more than 500 ppm was found in the reaction product after approximately 46 days of reaction time. During
- 25 inspection of the reactor, no lumps were found in the catalyst bed, and the average clear mesh size of the supporting grate was greater than the diameter of the catalyst or inert material particles. The catalyst bed did not exhibit a recess along the reactor wall. Neither catalyst nor inert material was found on the reactor base or in the subsequent apparatus.

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The results are summarized in the table below to provide a better overview:

| Example                                        | V-1  | V-2     | E-1     | E-2     | E-3  | E-4  |
|------------------------------------------------|------|---------|---------|---------|------|------|
| Wedge wire screen (S) or grate (G)             | S    | S       | S       | S       | G    | G    |
| Inert material                                 |      | X       | X       | X       |      |      |
| Knitted mesh support                           |      |         | X       | X       | X    | X    |
| Sealing cord on the reactor edge               |      |         |         | X       | X    | X    |
| Nitrobenzene breakthrough after                | 4 d  | 36 d    | 36 d    | 38 d    | 38 d | 46 d |
| Cost of filling the reactor                    | low  | high    | average | average | low  | low  |
| Cost of manufacturing the supporting structure | high | high    | high    | high    | low  | low  |
| Cost of maintaining the supporting structure   | high | average | low     | low     | low  | low  |

**Claims**

1. Chemical reactor for the heterogeneously catalysed reaction of a fluid, comprising a retaining means, through which fluid flows, for particles (80, 100),  
5 **characterized in that** the side of the retaining means arranged upstream in terms of the direction of flow of the fluid comprises wire mesh knitted fabric (50) and the average clear mesh size of the wire mesh (50) is less than the mean particle size  $x_{50.3}$  of the particles (80, 100).
- 10 2. Reactor according to Claim 1, wherein the average clear mesh size of the wire mesh (50) is  $\geq 20\%$  to  $\leq 80\%$  of the mean particle size  $x_{50.3}$  of the particles (80, 100).
3. Reactor according to Claim 1, wherein the extensibility of the wire mesh  
15 knitted fabric (50) under mechanical and/or thermal load parallel to the surface is  $\geq 1\%$ .
4. Reactor according to Claim 1, wherein the wire diameter of the wire mesh knitted fabric (50) is  $\geq 0.03$  mm to  $\leq 1$  mm.  
20
5. Reactor according to Claim 1, wherein the height of the wire mesh knitted fabric (50) in the particle retaining means is  $\geq 3$  mm to  $\leq 100$  mm.
6. Reactor according to Claim 1, wherein the wire mesh knitted fabric (50) is a  
25 wire mesh knitted fabric deformed by mechanical action.
7. Reactor according to Claim 1, wherein the wire mesh knitted fabric (50) is arranged on a support (30) and the support (30) comprises through-openings (40) in the direction of flow of the fluid.  
30
8. Reactor according to Claim 7, wherein wire mesh knitted fabric (60) is additionally arranged between the support (30) and the wall (10) of the reactor.

9. Reactor according to Claim 1, wherein catalyst particles (80) are arranged on the wire mesh knitted fabric (50) in the retaining means.
10. Reactor according to Claim 1, wherein a layer of particles (100) inert to the fluid is arranged on the wire mesh knitted fabric (50) in the retaining means, and catalyst particles (80) are arranged on this layer.
11. Method for reacting a fluid, **characterized in that** the reaction is carried out in a reactor according to any one of Claims 1 to 10 in the presence of heterogeneous catalyst particles (80), and the catalyst particles (80) are arranged in the retaining means for catalyst particles (80).
12. Method according to Claim 11, wherein the reactor is heated, once the reaction has finished and in the presence of oxygen, to a temperature of  $\geq 200^{\circ}\text{C}$  to  $\leq 400^{\circ}\text{C}$ .
13. Method according to Claim 11, wherein the fluid comprises aromatic nitro compounds and hydrogen.
14. Method according to Claim 11, wherein the reaction is carried out adiabatically.
15. Use of wire mesh knitted fabric (50) as a retaining means for catalyst particles (80) and/or inert particles (100) in chemical reactors.