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(57) Abstract: A process for producing a supported cobalt-based Fischer-Tropsch synthesis catalyst process includes, in a first activation stage, treating a particulate catalyst precursor, with a reducing gas, at a space velocity, SV1, and at a heating rate, HR1, up to a temperature, T_1, where $80°C \le T_1 \le 180°C$. In a second activation stage, the resultant partially treated precursor is treated with a gas, at a space velocity, SV2, and a heating rate, HR2, where $0 \le HR2 \ge HR1$, for a time, t_1, where t_1 is up to 20 hours. There after, the resultant partially reduced precursor is treated, in a third activation stage, with a reducing gas, at a space velocity, SV3, and at a heating rate, HR3, up to a temperature, T_2. The partially reduced precursor is maintained at T_2 for a time, t_2, where t_2 is from 0 to 20 hours to obtain an activated catalyst. When t_1=0, then SV3>SV1 and HR3≥HR1.

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CATALYSTS

THIS INVENTION relates to catalysts. In particular, the invention relates to a process for activating a catalyst precursor, to obtain a supported cobalt-based Fischer-Tropsch synthesis catalyst, and to a catalyst obtained from the process.

As regards supported cobalt-based Fischer-Tropsch synthesis catalysts, it is well-known that precursors of such catalysts are prepared using a metal precursor and a particulate support. The catalyst precursor preparation involves a number of different catalyst preparation steps. The catalyst precursor is then, in an activation process or step, reduced by using a reducing gas such as hydrogen, to obtain an active Fischer-Tropsch synthesis catalyst.

In known activation processes, ie involving reduction of the catalyst precursor in a flowing hydrogen or hydrogen containing gas stream at elevated temperatures, for supported cobalt-based Fischer-Tropsch synthesis catalyst precursors that the Applicant is aware of, hydrogen reduction is carried out at a temperature in the range 250°C to 500°C, and preferably at low pressures and high linear gas velocities to minimize vapour pressure of any product water which enhances sintering of the reduced metal. It is well known that manipulation of the reduction of cobalt oxide to cobalt metal in different ways influences activity and selectivity of the resultant Fischer-Tropsch synthesis catalyst. In particular, US 4605679 discloses that the activity of a cobalt catalyst can be increased by reduction in hydrogen, then re-oxidising the catalyst followed by re-reduction in hydrogen. In US 5292705, it is shown that hydrogen reduction in the presence of hydrocarbon liquids enhances the initial Fischer-Tropsch synthesis performance of the catalyst. US 5585316 claims that the selectivity of heavier Fischer-Tropsch products is increased if the catalyst is first oxidised and then reduced with carbon monoxide. . EP 1444040 discloses a two stage reduction step with pure hydrogen with a catalyst precursor in which all reducible cobalt oxide species combined can be described by the formula-unit CoO_aH_b (where: a≥1.7 and b>0), resulting in a more economical reduction process without sacrificing Fischer-Tropsch synthesis catalyst activity.

An object of the present invention is to provide a supported cobalt-based Fischer-Tropsch synthesis catalyst having a higher hydrocarbon synthesis activity. Such a catalyst can be obtained with the process of the present invention.

According to the invention, there is provided a process for producing a supported cobalt-based Fischer-Tropsch synthesis catalyst, which process includes

in a first activation stage, treating a particulate supported cobalt-based Fischer-Tropsch synthesis catalyst precursor comprising a catalyst support impregnated with cobalt and containing cobalt oxide, with a hydrogen-containing reducing gas or a nitrogen-containing gas, at a first specific feed gas space velocity, SV1, and at a first heating rate, HR1, until the precursor has reached a temperature, T_1, where 80°C≤ T_1≤180°C, to obtain a partially treated catalyst precursor; and

in a second activation stage, treating the partially treated catalyst precursor with a hydrogen-containing reducing gas, at a second specific feed gas space velocity, SV2, and a second heating rate, HR2, with HR2 increasing/decreasing gradually or in x step increments, where x is an integer, with x≥1, and where 0≤HR2≥HR1, for a time, t_1, where t_1 is from 0 to 20 hours, to obtain a partially reduced catalyst precursor; and thereafter

treating the partially reduced catalyst precursor, in a third activation stage, with a hydrogen-containing reducing gas, at a third specific feed gas space velocity, SV3, and at a third heating rate, HR3, until the partially reduced catalyst precursor reaches a temperature, T_2, and maintaining the partially reduced catalyst precursor at T_2 for a time, t_2, where t_2 is from 0 to

20 hours to obtain an activated supported Fischer-Tropsch synthesis catalyst, provided that when t_1 =0, then SV3>SV1 and HR3≥HR1.

When HR2 increases/decreases in x step increments, HR2 is then an average heating rate defined as the sum of the heating rates for each step increment divided by the number of step increments where a step increment is defined as an increase in the heating rate and x is, as indicated hereinbefore, the number of step increments.

It was surprisingly found that a supported cobalt-based Fischer-Tropsch synthesis catalyst having high intrinsic activity was obtained when the catalyst precursor was subjected to the reduction or activation procedure of the invention.

The treatments in the first, second and third activation stages may, at least in principle, be effected by using any suitable contacting configuration of the catalyst precursor with the reducing gas, such as a fluidized bed of the catalyst precursor particles, with the reducing gas acting as the fluidizing medium; a fixed bed of the catalyst precursor particles through which the reducing gas passes; or the like. However, a fluidized bed configuration is preferred.

The first activation stage commences when the catalyst precursor is first subjected to treatment with the hydrogen-containing reducing gas at the first gas space velocity SV1 with the immediate application of the first heating rate HR1. Preferably, $1 \leq SV1 \leq 35 m^3_n$/kg red. Co/h; more preferably, $3 \leq SV1 \leq 15 m^3_n$/kg red. Co/h. By 'red.Co' or 'reducible cobalt' is meant the cobalt that can be reduced during normal reduction, eg if the catalyst or catalyst precursor contains 20mass% cobalt and 50% of the cobalt can be reduced, then the amount of reducible cobalt is 0.1g/g catalyst or catalyst precursor. The first activation stage continues until the precursor attains the temperature T_1.

Preferably, $0.5°C/min \leq HR1 \leq 10°C/min$; more preferably, $1°C/min \leq HR1 \leq 2°C/min$.

In the first activation stage, T may be ≥90°C. In one embodiment of the invention, $125°C \leq T_1 \leq 170°C$. This embodiment will typically apply to precursors obtained by forming a slurry of a particulate catalyst support, a cobalt compound as an active component precursor, and water; subjecting the catalyst support to impregnation with the cobalt compound; drying the impregnated catalyst support; and calcining the impregnated support.

The second activation stage thus commences when the precursor has attained the temperature T_1, and endures for the time t_1 as hereinbefore described. As regards the second activation stage treatment time t_1, more preferably $1 \leq t_1 \leq 10$ hours, typically $2 \leq t_1 \leq 6$ hours.

In the second activation stage, one of the following applies: the temperature of the precursor increases gradually; at least one heating ramp is employed to increase the precursor temperature; or the precursor is maintained at the temperature T_1. In one embodiment of the invention, the temperature T_1 thus constitutes a holding temperature at which the precursor is held for the treatment time of t_1. However, in another embodiment of the invention, said at least one heating ramp in which the precursor is heated from the temperature T_1 to a temperature T_H where $T_H > T_1$ and $T_H < 200°C$ is employed over treatment time t_1. Naturally, more than one heating ramp can then be employed. Thus, when two heating ramps are employed, x=2; when three heating ramps are employed, x=3; etc. Where more than one heating ramp is employed, the heating rates in the heating ramps will thus differ. For example, if the heating rate in a first heating ramp is 1°C/min, then the heating rate in a second heating ramp can be 2°C/min. Still further, the precursor can, if desired, be held for some time at the temperature attained at the end of a heating ramp, before commencing the next heating ramp.

The third activation stage commences once the time t_1 has elapsed. The precursor thus, at the commencement of the third activation stage and in one embodiment of the invention, will still be at the temperature T_1, ie at a temperature between 80°C and 180°C. However, in another embodiment of the invention, the precursor will, at the commencement of the third activation stage, thus be at the higher temperature, T_H. The third activation stage treatment is thus continued until the temperature in the third treatment stage, ie the temperature of the activated Fischer-Tropsch catalyst, reaches T_2. Preferably, $300°C \leq T_2 \leq 600°C$. More preferably, T_2 may be in the range of 300°C to 500°C, with a typical value of T_2 being in the range of 300°C to 450°C. The catalyst precursor can be maintained at T_2 for 0-20 hours (ie t_2), preferably $0 < t_2 \leq 20$ hours, more preferably $1 \leq t_2 \leq 10$ hours, typically $2 \leq t_2 \leq 6$ hours.

In one embodiment of the invention, SV1, SV2 and/or SV3 may be constant during the treatments in their respective activation stages. However, in another embodiment of the invention, SV1, SV2 and SV3 may vary during the respective activation stages.

In the first activation stage, a hydrogen-containing reducing gas is preferably used, and the gas used in the three activation stages may have the same composition. By hydrogen-containing reducing gas' is meant a hydrogen containing gas mixture comprising $10vol\% < H_2 \leq 100vol\%$, more preferably >90vol% H_2 and <10 vol% inerts, most preferably >97 vol% H_2 and <3vol% inerts. The inerts could be any combination of Ar, He, NH_3 and H_2O, with the preferred dew point of the hydrogen-containing reducing gas being ≤4°C, more preferably ≤-30°C.

In the first activation stage, a nitrogen-containing gas can instead be used. By 'nitrogen-containing gas' is meant a gas mixture comprising >90vol% N_2 and <10vol% other components with the other components being any combination of Ar, He, and H_2O. The preferred dew point of the nitrogen-containing gas is ≤4°C, more preferably ≤-30°C. This nitrogen containing gas does not contain any hydrogen (ie hydrogen=0vol%).

The treatments in the first, second and third activation stages may be effected at the same or different pressures, and may each be effected at about atmospheric pressure, preferably at between 0.6 and 1.3 bar(a).

The particulate supported cobalt-based Fischer-Tropsch synthesis ('FTS') catalyst precursor may be any suitable catalyst precursor requiring activation or reduction to obtain an active Fischer-Tropsch catalyst, and may be that obtained during preparation of a fresh catalyst or from a regenerated catalyst.

Thus, it may be that obtained during preparation of a fresh catalyst, ie obtained by forming a slurry of a particulate catalyst support, a cobalt compound as an active component precursor, and water; subjecting the catalyst support to impregnation with the cobalt compound; drying the impregnated catalyst support; and calcining the impregnated support, to obtain the catalyst precursor, which contains cobalt oxide. The catalyst precursor thus obtained must, however, then still be activated or reduced prior to using it for catalyzing a Fischer-Tropsch reaction, and this reduction or activation is effected in accordance with the method of the present invention. The resultant catalyst is thus an activated Fischer-Tropsch catalyst.

The regenerated catalyst precursor can be obtained from regenerating a spent cobalt Fischer-Tropsch catalyst, that was used in a FTS process for a period of time, by means of any suitable regeneration process, which results in an oxidized catalyst precursor containing supported cobalt oxide.

Any commercially available pre-shaped porous oxide catalyst support, such as alumina (Al_2O_3), silica (SiO_2), titania (TiO_2), magnesia (MgO), SiO_2-Al_2O_3 and zinc oxide (ZnO), may be used. The support preferably has an average pore diameter between 8 and 50 nanometers, more preferably between 10 and 15 nanometers. The support pore volume may be between 0.1 and 1.5mℓ/g, preferably between 0.3 and 0.9mℓ/g.

The support may be a protected modified catalyst support, containing, for example, silicon as modifying component, as generally described in EP

Application No. 99906328.2 (European Publication No. 1058580), which is hence incorporated herein by reference.

More specifically, the protected modified catalyst support may be that obtained by contacting a silicon precursor, eg an organic silicon compound such as tetra ethoxy silane (‘TEOS’) or tetra methoxy silane (‘TMOS’), with the catalyst support, eg by means of impregnation, precipitation or chemical vapour deposition, to obtain a silicon-containing modified catalyst support; and calcining the silicon-containing modified catalyst support, eg in a rotary calciner, at a temperature from 100°C to 800°C, preferably from 450°C to 550°C, and for a period of from 1 minute to 12 hours, preferably from 0.5 hour to 4 hours.

The cobalt loading can be between 5gCo/100g support and 70gCo/100g support, preferably between 20gCo/100g support and 55gCo/100g support.

The cobalt salt may, in particular, be cobalt nitrate, $Co(NO_3)_2.6H_2O$.

The impregnation of the catalyst support may, in principle, be effected by any known method or procedure such as incipient wetness impregnation or slurry impregnation. Thus, the impregnation may generally be effected in the manner described in US 6455462 or in US 5733839, and which are thus incorporated herein by reference thereto.

More specifically, impregnation may be effected by subjecting, at elevated temperature, a slurry comprising the particulate catalyst support, water, and the cobalt salt to a sub-atmospheric pressure environment, which may be down to 5kPa(a), preferably between atmospheric pressure and 10kPa(a); drying the impregnated carrier at elevated temperature and under a sub-atmospheric pressure environment, which may be as hereinbefore described. Still more specifically, the impregnation may be effected by subjecting the slurry, in an initial treatment stage, to treatment at elevated temperature and under a sub-atmospheric pressure environment as hereinbefore described to impregnate the support with the cobalt salt and to dry the impregnated support partially, and thereafter, in a subsequent treatment stage, subjecting the partially dried impregnated support to treatment of elevated temperature and under a sub-atmospheric pressure environment as hereinbefore described, such that the temperature in the subsequent treatment stage exceeds that in the initial treatment stage and/or the sub-atmospheric pressure in the subsequent treatment stage is lower than that in the initial treatment stage, thereby to obtain more vigorous drying of the impregnated support in the subsequent treatment stage than in the initial treatment stage, to obtain a dried impregnated support.

The impregnation may include subjecting the support to two or more impregnation steps, to obtain a desired cobalt loading. Each impregnation step may then include an initial and a subsequent treatment stage as hereinbefore described.

The process may then include, in each of the impregnation steps, controlling the drying rate of the slurry to a specified drying profile.

The support impregnation may thus involve a 2-step slurry phase impregnation process, which is dependent on a desired cobalt loading requirement and the pore volume of the catalyst support.

The support impregnation and drying may typically be effected in a conical vacuum drier with a rotating screw or in a tumbling vacuum drier.

During the cobalt impregnation steps, a water soluble precursor salt of platinum (Pt), palladium (Pd), ruthenium (Ru), rhenium (Re) or mixtures thereof, may be added, as a dopant capable of enhancing the reducibility of the active component.

Calcination of the impregnated and dried material may be done using any method, known to those skilled in the art, for example in a fluidized bed, or a rotary kiln, calciner at 200-400°C. It may, in particular, be effected as described in PCT Patent Application WO 01/39882, which is thus also incorporated herein by reference.

The invention extends also to an activated Fischer-Tropsch catalyst, when obtained by the process of the first aspect of the invention.

The activated Fischer-Tropsch catalyst can be used in a process for producing hydrocarbons, which includes contacting a synthesis gas comprising hydrogen (H_2) and carbon monoxide (CO) at an elevated temperature between 180°C and 250°C and an elevated pressure between 10 and 40 bar with an activated Fischer-Tropsch catalyst as hereinbefore described, using a Fischer-Tropsch reaction of the hydrogen with the carbon monoxide.

The invention will now be described in more detail with reference to the following non-limiting example;

EXAMPLE 1

A particulate supported cobalt-based Fischer-Tropsch synthesis catalyst precursor, which, on activation, produces a 30g Co/100g Al_2O_3 proprietary slurry phase Fischer-Tropsch synthesis catalyst of the Applicant, and which is fully described in WO 01/39882, was investigated.

A representative batch of this pre-reduced catalyst precursor was specifically prepared as follows: Puralox SCCa 2/150, pore volume of 0.48mℓ/g, from SASOL Germany GmbH of Uberseering 40, 22297 Hamburg, Germany was modified with silicon such that the final silicon level was 2.5 Si atoms/nm^2 of support. TEOS (tetra ethoxy silane) was added to ethanol, alumina (1l ethanol/kg alumina) was added to this solution, and the resultant mixture stirred at 60 °C for 30 minutes. Subsequently the solvent was removed under vacuum with a jacket temperature of the drier equipment of 95 °C. The dried modified support was then calcined at 500 °C for 2 hours. A solution of 17.4kg of $Co(NO_3)_2.6H_2O$, 9.6g of $(NH_3)_4Pt(NO_3)_2$, and 11kg of distilled water was mixed with 20.0kg of the above mentioned silica modified gamma alumina support by adding the support to the solution. The slurry was added to a conical vacuum drier and continuously mixed. The temperature of this slurry was increased to 60°C after which a pressure of 20kPa(a) was applied. During the first 3 hours of the drying step, the temperature was increased slowly and reached 95°C after 3 hours. After 3 hours the pressure was decreased to 3-15kPa(a), and a drying rate of 2.5m%/h at the point of incipient wetness was used. The complete impregnation and drying step took 9 hours, after which the impregnated and dried catalyst support was immediately and directly loaded into a fluidised bed calciner. The temperature of the dried impregnated catalyst support was about 75°C at the time of loading into the calciner. The loading took about 1 to 2 minutes, and the temperature inside the calciner remained at its set point of about 75°C. The dried impregnated catalyst support was heated from 75°C to 250°C, using a heating rate of 0.5°C/min and an air space velocity of 1.0 m^3_n/kg $Co(NO_3)_2.6H_2O$/h, and kept at 250°C for 6 hours. To obtain a catalyst with a cobalt loading of 30gCo/100gAl_2O_3, a second impregnation/drying/calcination step was performed. A solution of 9.4kg of $Co(NO_3)_2.6H_2O$, 15.7g of $(NH_3)_4Pt(NO_3)_2$, and 15.1kg of distilled water was mixed with 20.0kg of the catalyst precursor from the first impregnation and calcination, by adding the catalyst precursor to the solution. The slurry was added to a conical vacuum drier and continuously mixed. The temperature of this slurry was increased to 60°C after which a pressure of 20kPa(a) was applied. During the first 3 hours of the drying step, the temperature was increased slowly and reached 95°C after 3 hours. After 3 hours the pressure was decreased to 3-15kPa(a), and a drying rate of 2.5m%/h at the point of incipient wetness was used. The complete impregnation and drying step took 9 hours, after which the treated catalyst support was immediately and directly loaded into the fluidised bed calciner. The temperature of the dried impregnated catalyst support was about 75°C at the time of loading into the calciner. The loading took about 1 to 2 minutes, and the temperature inside the calciner remained at its set point of about 75°C. The dried impregnated catalyst was heated from 75°C to 250°C, using a heating rate of 0.5°C/min and an air space velocity of 1.0 m^3_n/kg $Co(NO_3)_2.6H_2O$/h, and kept at 250°C for 6 hours. A supported cobalt catalyst precursor on an alumina support was thus obtained.

One sample of this precursor, identified as Precursor A, was subjected to the following sequential reduction procedure:

<u>Stage 1</u>
Pure hydrogen reducing gas
SV1= 1050ml/(g cat.h)
HR1=1°C/min
T_1=170°C

<u>Stage 2</u>
t_1=0

<u>Stage 3</u>
Pure hydrogen reducing gas
SV3= 1500 ml/(g cat.h)
HR3= 1°C/min
T_2=425°C
t_2=10 hours (at 425°C)

Thus SV3>SV1 and HR1=HR3.

This reduction or activation procedure was carried out in a fluidized bed reduction unit (20mm internal diameter), at atmospheric pressure, using an undiluted H_2 reducing gas as total feed gas at space velocities as stated above, whilst applying the temperature program as stated for the different stages, and the same undiluted H_2 reducing gas (100vol% H_2) was used in all the activation stages.

Thus, Precursor A was subjected to a sequential reduction/activation procedure in accordance with the invention, to obtain Catalyst A which is thus in accordance with the invention.

During reduction, precursor A was thus transformed into Fischer-Tropsch synthesis ('FTS') Catalyst A. This catalyst was evaluated in a laboratory scale reactor under realistic FTS conditions (230°C, 17.5 bar_g pressure, H_2:CO inlet ratio of 1.9:1 for Catalyst A, and at a synthesis gas conversion of 60 ± 5%).

Table 1: Summary of the FTS runs of Example 1

Catalyst	RIAF
A	1.05

RIAF = Relative Intrinsic Fischer-Tropsch synthesis Activity Factor

From Table 1(RIAF data) it is clear that the activity of Catalyst A in accordance with the invention, is a catalyst with high activity.

The Relative Intrinsic Fischer-Tropsch synthesis Activity Factor ('$RIAF_x$') of a supported cobalt slurry phase catalyst, of which the pre-reduction catalyst precursor has been prepared in strict accordance with a prescribed catalyst preparation procedure X, ie catalyst precursor X, is defined as:

$$RIAF_x = [A_{xi}/A_x] \qquad \ldots\ldots\ldots\ldots\ldots\ldots \qquad (1)$$

where:

a) A_{xi} is the Arrhenius pre-exponential factor of catalyst precursor X, activated according to an arbitrary reduction procedure

b) A_x is the Arrhenius pre-exponential factor of catalyst precursor X, estimated from the 15 hours on stream slurry-phase Continuous Stirred Tank Reactor (CSTR) Fischer-Tropsch synthesis performance under realistic conditions, and having utilized the standard one-step reduction procedure:

Fluidized bed (20mm internal diameter) reduction of 15±5g catalyst precursor A (ie pre-reduction catalyst mass), at atmospheric pressure utilizing an undiluted H_2 reducing gas (purity of 5.0) as total feed at a space velocity of 13700$m\ell_n$ per gram reducible cobalt per hour, whilst applying the following temperature program: heat from 25°C to 425°C at 1°C/min, and hold isothermally at 425°C for 16 hours.

c) The pre-exponential factor A, ie applicable to both A_{xi} and A_x, is defined from the generally accepted cobalt-based Fischer-Tropsch empirical kinetic expression:

$$r_{FT} = [Ae^{(-Ea/RT)}P_{H2}P_{CO}]/[1+KP_{CO}]^2 \ldots\ldots\ldots\ldots\ldots\ldots \quad (2)$$

Thus:

$$A= [r_{FT}\ (1+KP_{CO})^2]/[e^{(-Ea/RT)}P_{H2}P_{CO}] \ldots\ldots\ldots\ldots\ldots\ldots \quad (3)$$

where:

r_{FT} is expressed in terms of the number of moles of CO converted into Fischer-Tropsch synthesis products per unit time per unit mass of the catalyst precursor in its pre-reduction state.

d) x is any catalyst precursor.

CLAIMS:

1. A process for producing a supported cobalt-based Fischer-Tropsch synthesis catalyst, which process includes

in a first activation stage, treating a particulate supported cobalt-based Fischer-Tropsch synthesis catalyst precursor comprising a catalyst support impregnated with cobalt and containing cobalt oxide, with a hydrogen-containing reducing gas or a nitrogen-containing gas, at a first specific feed gas space velocity, SV1, and at a first heating rate, HR1, until the precursor has reached a temperature, T_1, where $80°C \leq T_1 \leq 180°C$, to obtain a partially treated catalyst precursor; and

in a second activation stage, treating the partially treated catalyst precursor with a hydrogen-containing reducing gas, at a second specific feed gas space velocity, SV2, and a second heating rate, HR2, with HR2 increasing/decreasing gradually or in x step increments, where x is an integer, with $x \geq 1$, and where $0 \leq HR2 \geq HR1$, for a time, t_1, where t_1 is from 0 to 20 hours, to obtain a partially reduced catalyst precursor; and thereafter

treating the partially reduced catalyst precursor, in a third activation stage, with a hydrogen-containing reducing gas, at a third specific feed gas space velocity, SV3, and at a third heating rate, HR3, until the partially reduced catalyst precursor reaches a temperature, T_2, and maintaining the partially reduced catalyst precursor at T_2 for a time, t_2, where t_2 is from 0 to 20 hours to obtain an activated supported Fischer-Tropsch synthesis catalyst, provided that when $t_1 = 0$, then $SV3 > SV1$ and $HR3 \geq HR1$.

2. A process according to Claim 1 wherein, in the first activation stage, $0.5°C/m \leq HR1 \leq 10°C/min$.

3. A process according to Claim 2 wherein, in the first activation stage, $1°C/min \leq HR1 \leq 2°C/min$.

4. A process according to any one of Claims 1 to 3 inclusive wherein, in the second activation stage, $1 \leq t_1 \leq 10$ hours.

5. A process according to Claim 4 wherein, in the second activation stage, 2≤ t_1≤6 hours.

6. A process according to any one of Claims 1 to 5 inclusive wherein, in the second activation stage, the precursor is maintained at the temperature T_1.

7. A process according to any one of Claims 1 to 5 inclusive wherein, in the second activation stage, the precursor is heated from the temperature T_1 to a temperature T_H where $T_H > T_1$ and $T_H < 200°C$.

8. A process according to any one of Claims 1 to 7 inclusive wherein, in the third activation stage, $300°C \leq T_2 \leq 600°C$.

9. A process according to any one of Claims 1 to 8 inclusive wherein, in the third activation stage, $1 \leq t_2 \leq 10$ hours.

10. A process according to any one of Claims 1 to 9 inclusive, wherein the space velocity of the gas is constant during the treatments in each of the first, second and third stages.

11. A process according to any one of Claims 1 to 10 inclusive, wherein the treatments in the first, second and third activation stages are each effected at a pressure between 0.6 and 1.3 bar(a).

12. A process according to any one of Claims 1 to 11 inclusive, wherein a hydrogen-containing reducing gas is used in the first activation stage, with the hydrogen-containing reducing gas in each of the activation stages comprising >90vol% H_2 and <10vol% inerts.

13. A process according to Claim 12, wherein the hydrogen-containing gas in each of the activation stages comprises >97vol% H_2 and <3vol% inerts.