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Process for the preparation of a fatty aldehyde.

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The present invention relates to a process for the preparation of a fatty aldehyde which process comprises (a) providing a fatty acid, and (b) contacting the fatty acid with a zerovalent metal catalyst to reduce the fatty acid to the fatty aldehyde, wherein the zerovalent metal catalyst is in the form of solid particles.

Process for the preparation of a fatty aldehyde

The present invention relates to a process for the preparation of a fatty aldehyde, which process avoids the use of fossil resources and/or harsh reaction conditions, as well as the use of the obtained fatty aldehyde as e.g. a flavoring agent or fragrance.

There are currently two main methods for the synthesis of aldehydes, in particular fatty aldehydes. Short-chain fatty aldehydes are usually prepared by hydroformylation. The alkenes used as starting material in the hydroformylation reaction are typically obtained on the basis of crude oil. In general, rather high temperatures and very high pressures are applied in the hydroformylation reaction. Long-chain fatty aldehydes are primarily obtained by dehydration of fatty alcohols. Also this process is typically characterized by the use of fossil starting materials and rather harsh reaction conditions.

For instance, EP 37149 describes a method for the preparation of aldehydes by dehydrogenation of monovalent primary alcohols in the gas phase by means of a copper-containing catalyst. For example, passing a gaseous mixture comprising octanol-1 and hydrogen over a Cu/MgO catalyst for a period of four weeks at a temperature of 265 to 330°C results in an octanol conversion of 58%. The corresponding use of an Ag/Na₂O catalyst is described in SU 789491, and US 4,304,943 describes the conversion of alkyl and aryl hydroxy compounds to aldehydes in the presence of hydrogen using a MnO/NiO/MgO catalyst.

Furthermore, US 2,128,908 relates to a process for preparing short chain aliphatic aldehydes by oxidizing C₃- and C₄-alkanes. For instance, a mixture of propane and butane is oxidized at 425 to 460°C to yield the desired aldehyde (20%) as

well as formaldehyde (15%), methanol (19%) and carboxylic acids (11%).

Alternative methods for the preparation of aldehydes, such as the extraction of aldehydes from natural products, are typically characterized by a very low yield. For example, Palma et al. (J. Agric. Food Chem. 1999, 47, 5044) report on the supercritical fluid extraction of white grape seeds resulting in an average yield of 10.6% for the first fraction which *inter alia* comprises aliphatic aldehydes in addition to fatty acids and sterols. Moreover, Chouchi et al. describe the desorption of bigarade peel oil from a polar adsorbent by supercritical CO₂ yielding a residue fraction comprising 0.22% of n-heptanal.

Hence, there is still a need for a further process for the preparation of fatty aldehydes. In particular, it would be desirable to provide a process for the synthesis of fatty aldehydes which process allows the use of renewable starting materials and, thereby, reducing or avoiding the use of fossil resources. Moreover, the process should allow the use of mild reaction conditions so that rather high temperatures and/or pressures can be avoided. In addition, the fatty aldehyde should be obtainable with a high selectivity.

It has surprisingly been found that the above problems are solved by the process defined below and in the appended claims.

Accordingly, in a first aspect the present invention relates to a process for the preparation of a fatty aldehyde which comprises

(a) providing a fatty acid, and



- (b) contacting the fatty acid with a zerovalent metal catalyst to reduce the fatty acid to the fatty aldehyde, wherein the zerovalent metal catalyst is in the form of solid particles.

It has surprisingly been found that the contacting of a fatty acid with a solid zerovalent metal catalyst allows the synthesis of a fatty aldehyde with a high selectivity in a simple method without the use of excessive reaction temperatures or pressures. Moreover, the fatty acid used as starting material in the process according to the invention can suitably be provided on the basis of natural, renewable resources. Thus, the use of fossil resources can be reduced or even avoided.

The zerovalent metal catalyst is in the form of solid particles, i.e. is a solid catalyst. It can be an unsupported (bulk) catalyst or can comprise a support material on or in which the zerovalent metal is present. Preferably, the zerovalent metal catalyst is a bulk catalyst, i.e. the catalyst is unsupported and essentially consists of or consists of the zerovalent metal. The oxidation state of the metal is zero.

Moreover, it is preferred that the solid particles of the zerovalent metal catalyst have a specific surface area of 0.0001 to 1080 m²/g, preferably 0.0003 to 1 m²/g, more preferably 0.005 to 0.002 m²/g, even more preferably 0.001 to 0.01 m²/g. The specific surface area can suitably be determined by imaging procedures. In particular, the average particle size of the solid particles size is determined via microscopy and on the basis of this parameter the surface area of the particles is calculated assuming a spherical shape of each particle. It has been found that a zerovalent metal catalyst having such a specific surface area is particularly suitable for the

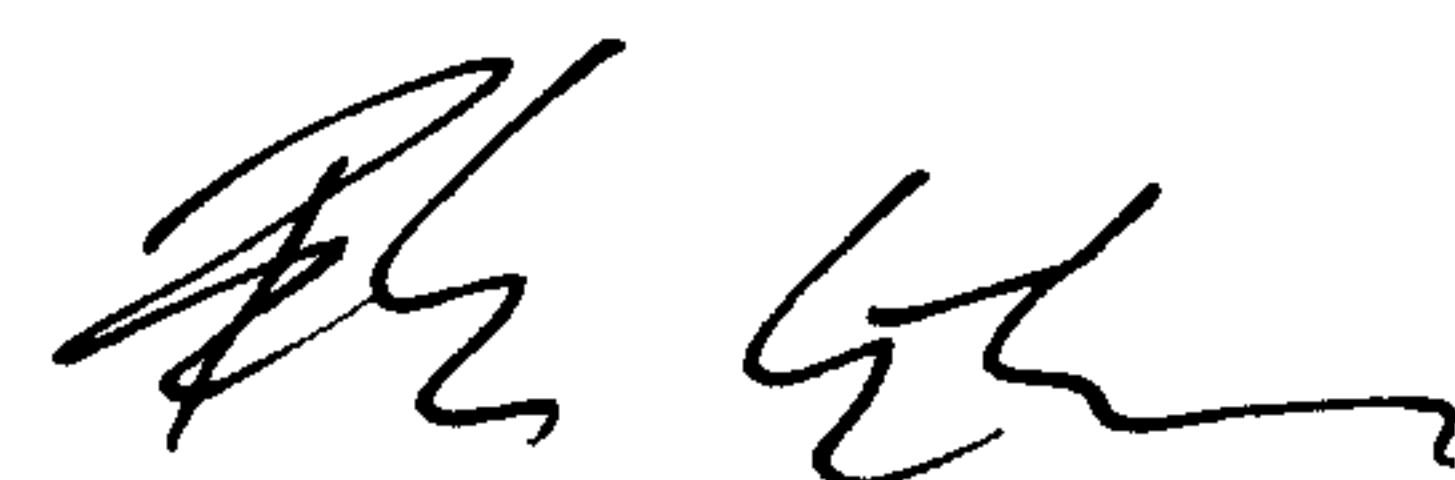


efficient and selective conversion of fatty acids into fatty aldehydes.

It is also preferred that the solid particles have an average particle size of 70 nm to 3000 μm , preferably 5 to 2500 μm , such as 5 to 20 μm or 50 to 150 μm or 500 to 2500 μm , as determined by imaging procedures, such as microscopy, resulting in a number-based average.

The metal of the zerovalent metal catalyst can suitably have a standard electrode potential of less than 1.40 V, preferably less than 1.20 V, more preferably less than 0.80 V. As used herein, the term "standard electrode potential" refers to the standard potential E^0 at a temperature of 25°C, a pressure of 1 atm and an effective ion concentration of 1 mol/L, with the metal in its zerovalent state as reductant and the metal cation in its monovalent (oxidation state = +1), divalent (oxidation state = +2) or trivalent (oxidation state = +3), preferably the metal cation in its divalent form, as oxidant. The metal can be selected from the group consisting of Fe, Cu, Ag, Hg, Au, Pt and Sn and combinations thereof, and preferably is Fe or Cu or Ag and more preferably is Fe. In one embodiment the iron is present in the form of iron filings.

Without wishing to be bound to a particular theory, it is believed that the higher the specific surface area of the catalyst is, the higher the tendency for formation of the corresponding alcohol instead of the desired aldehyde is. The formation of alcohol is probably due to the further reduction of the intermediate aldehyde. Thus, the specific surface area of the catalyst is believed to have an effect on the selectivity of the catalyst. Hence, catalysts having a rather high specific surface area suitably comprise a more noble metal, such as silver. In contrast, catalysts having a rather low specific



surface area suitably comprise a less noble metal, such as iron.

The fatty acid, which is provided in step (a) of the process according to the invention and which is to be reduced to the corresponding aldehyde, can typically comprise an aliphatic carboxylic acid having 1 to 32 carbon atoms, preferably 4 to 28, more preferably 6 to 18 carbon atoms. Moreover, the fatty acid can be saturated or unsaturated and can be branched or unbranched. Preferably, the fatty acid is unbranched.

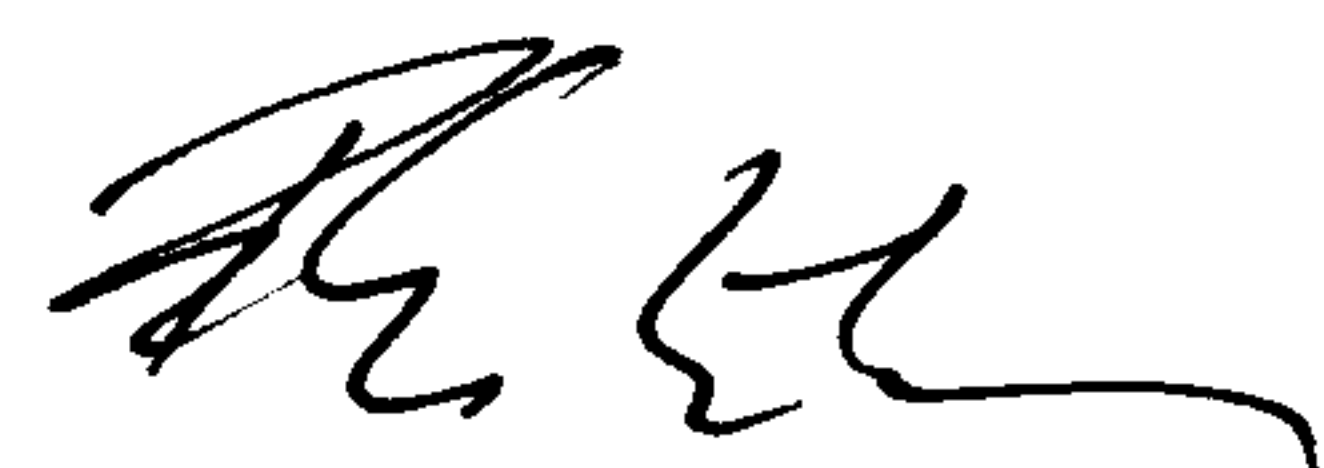
The selective reduction of the fatty acid to yield the corresponding aldehyde does generally not depend on the source of the fatty acid. Thus, synthetic fatty acids as well as fatty acids obtained from natural products or renewable starting materials, like vegetable oils or other natural fatty acid esters, can be provided in step (a). Preferably, the fatty acid provided in step (a) is obtained from natural sources such as renewable starting materials. In particular, it is preferred that in step (a) the fatty acid is provided by

(a1) providing a fatty acid ester, and

(a2) contacting the fatty acid ester with an enzymatic catalyst to hydrolyze the fatty acid ester to the fatty acid.

The enzymatic catalyst is suitably a hydrolase, preferably a hydrolase of subclass EC 3.1 such as subclass 3.1.1 like 3.1.1.1 and 3.1.1.3, more preferably an esterase like a lipase, such as e.g. lipase B *candida antarctica*.

Moreover, it is known that synthetic fatty esters, such as ethyl esters, on the one hand and natural fatty acid esters on the basis of triglyceride, such as vegetable oils, on the other hand show a very similar hydrolyzation behavior. Hence, the

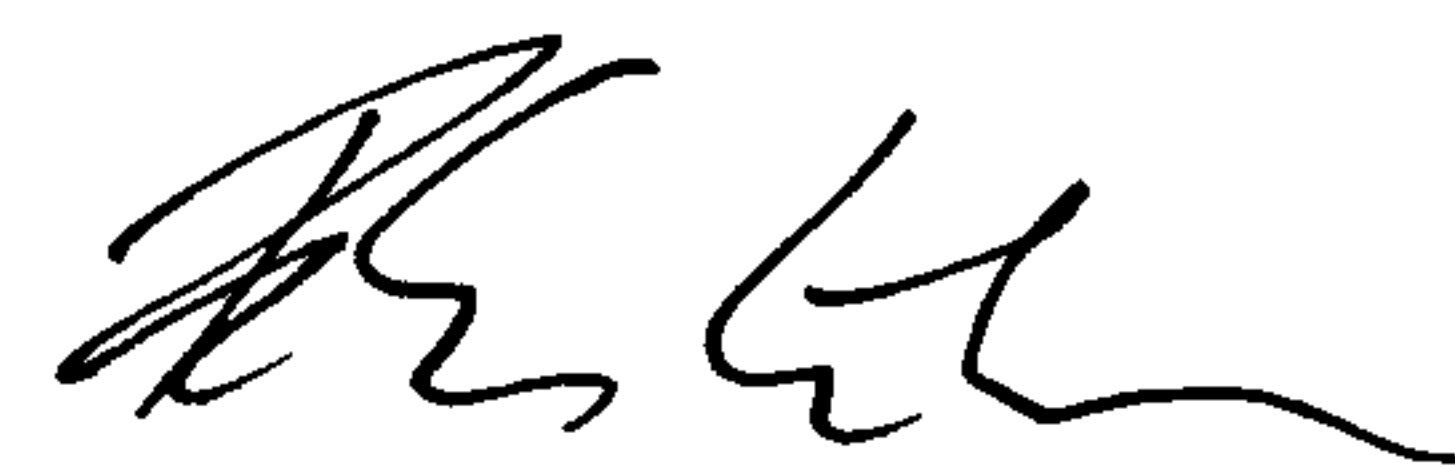


fatty acid ester provided in step (a1) can be both a synthetic fatty acid ester or a natural fatty acid ester, such as a vegetable oil. However, due the general demand for the use of natural starting materials, the fatty acid ester provided in step (a1) is preferably natural fatty acid ester, such as a vegetable oil.

In step (b), the contacting is typically effected at a ratio of the zerovalent metal catalyst relative to the fatty acid of $8.0 \cdot 10^{-5}$ to $3.1 \cdot 10^{-3}$ m² of catalyst per mmol of fatty acid, preferably $1.0 \cdot 10^{-4}$ to $1.0 \cdot 10^{-3}$ m² of catalyst per mmol of fatty acid, and more preferably $2.0 \cdot 10^{-4}$ to $5.0 \cdot 10^{-4}$ m² of catalyst per mmol of fatty acid. It has been found that such a ratio of zerovalent metal catalyst to fatty acid is particularly suitable for the efficient and selective conversion of fatty acids into fatty aldehydes.

Moreover, it is preferred - with a view to avoid the undesired formation of the corresponding alcohols - that in step (b) the contacting is effected at a temperature of 80 to 800°C, preferably 80 to 250°C, more preferably 85 to 250°C, most preferably 90 to 220°C, such as about 90°C or 180 to 220°C; and/or at a pressure of 100 to 1200 mbar, preferably 100 to 300 mbar or about 1000 mbar; and/or for a time of 3 to 180 min, preferably 15 to 120 min, more preferably 20 to 90 min, most preferably 30 to 70 min.

The reducing performance of the metal catalyst can be effected by other conditions as well. For example, the presence of oxygen or water can result in an undesired redox reaction between the metal catalyst and oxygen and/or water so that the amount of metal catalyst available for the reduction the fatty acid becomes too low. Hence, the contacting in step (b) is suitably effected at a concentration of water of less than 10 wt.-%, preferably less than 2 wt.-%, more preferably 0 to less than 1



wt.-%, relative to the total weight of the reaction mixture of step (b). Furthermore, the contacting in step (b) is suitably effected at a concentration of oxygen of less than 20 wt.-%, preferably less than 19 wt.-%, more preferably 0 to 1 wt.-%, such as 0 to 0.1 or 0 to 0.01 wt.-%, relative to the total weight of the reaction mixture of step (b).

In addition, the reduction in step (b) can be carried without the presence of hydrogen and it is preferred that the contacting in step (b) is effected at a concentration of hydrogen of about 0 wt.-%.

The aldehyde obtained in step (b) preferably comprises 1 to 32 carbon atoms, and more preferably comprises hexanal, octanal, decanal, dodecanal or mixtures thereof. Such aldehydes are valuable products which can in particular be used as a flavoring agent, fragrance or as an intermediate product for the production of plastifiers, lubricating agents, polymers of pharmaceuticals.

The invention disclosed herein is exemplified by the following examples which should not be construed to limit the scope of the disclosure.

Examples

Example 1: Preparation of dodecanal

A mixture of 250 mL of ethyl dodecanoate, 250 mL of water and 5g of Novozym 435 (a lipase B acrylic resin from *Candida antarctica*) was stirred at 40°C for 24 hours. Then the mixture was cooled to 0°C for 8 hours. The liquid phase was separated by decantation. The container containing the solid phase comprising dodecanoic acid was equipped with a heating unit and a distilling connecting tube which was held at 18°C. A pressure



of 100 mbar was applied. The temperature of the distillation receiver and the gas phase of the distilling connecting unit was monitored and water was completely removed from the dodecanoic acid by distillation at a temperature of about 60°C.

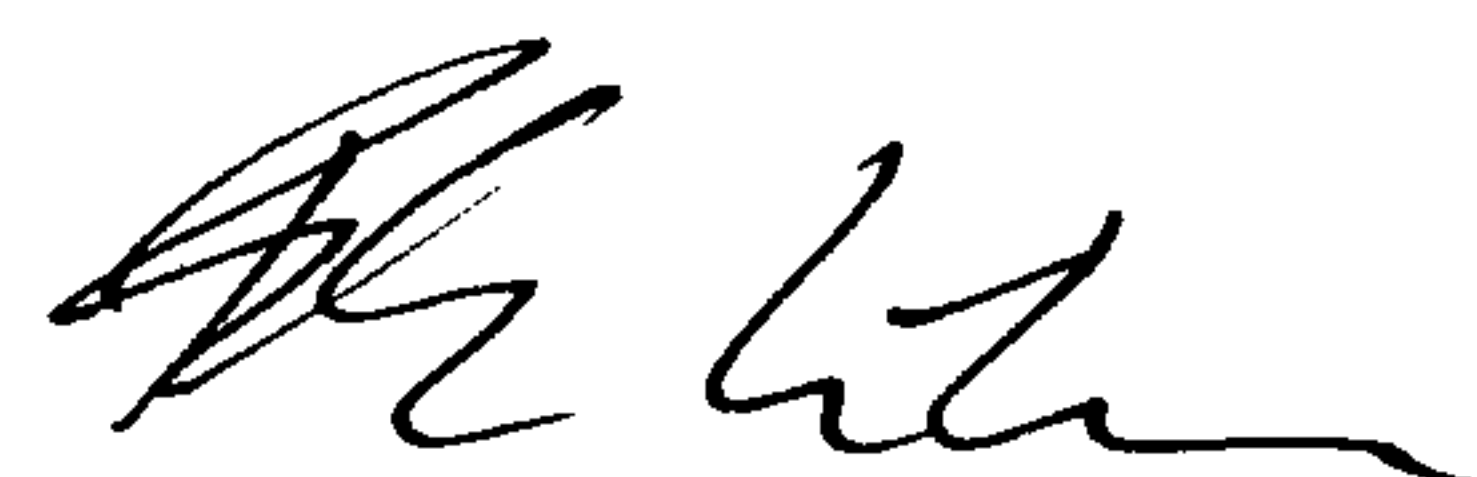
Subsequently, 200 g of iron filings having a particle size of about 500 to 2500 μm and a specific surface area of about 14.7 cm^2/g were added to the dodecanoic acid. The system was rinsed with nitrogen three times and then a nitrogen pressure of 200 mbar was applied. The reaction mixture was heated to a temperature of 200 to 230°C for 35 min. Then the pressure was reduced in intervals of 5 mbar to yield dodecanal in the distillation receiver which dodecanal was identified via gas chromatography using a PerkinElmer Clarus 500 apparatus equipped with flame ionization detector and a SUPELCO SLB-5ms capillary column having dimensions of 30 mm x 0.25 mm x 0.25 μm . Nitrogen was used as mobile phase. For the detection of dodecanal, the temperature was kept at 60°C for 6 minutes and subsequently raised to 200°C at a rate of 12°C/min. Then the temperature was kept at 200°C for 7 minutes.

Example 2: Preparation of hexanal

2 mL (16 mmol) of hexanoic acid were contacted at 90°C for 60 min with the following bulk catalysts A to D. The amount of the bulk catalysts is indicated in Table 1 below. The contacting was performed at ambient pressure under air atmosphere.

Catalyst A:

Iron particles (iron filings) having a particle size of about 500 to 2500 μm and a specific surface area of about 14.7 cm^2/g .



Catalyst B:

Iron particles (iron powder) having a particle size of about 90 μm and a specific surface area of about $8.44 \cdot 10^3 \text{ cm}^2/\text{g}$.

Catalyst C:

Iron particles (iron powder) having a particle size of about 70 nm and a specific surface area of about $1.08 \cdot 10^7 \text{ cm}^2/\text{g}$.

Catalyst D:

Copper particles having a particle size of about 9 μm and a specific surface area of about $7.41 \cdot 10^4 \text{ cm}^2/\text{g}$.

The resulting reaction mixtures were subjected to a Fehling analysis. A blue color was indicative for the presence of aldehydes, i.e. the formation of hexanal.

Table 1:

Catalyst	Amount [g]	Formation of hexanal
A	1.6	+
A	2.2	++
A	4.5	+++
B	1.6	+/-
B	2.0	+
B	2.4	+
C	0.02	+/-
C	0.03	+/-
C	0.07	+/-
D	0.2	+++

+/-: moderate blue Fehling analysis

+: significant blue Fehling analysis

++: intensive blue Fehling analysis

+++ : very intensive blue Fehling analysis

Some of the resulting reaction mixtures were also analyzed via gas chromatography using a PerkinElmer Clarus 500 apparatus



equipped with flame ionization detector and a SUPELCO SLB-5ms capillary column having dimensions of 30 mm x 0.25 mm x 0.25 μ m. Nitrogen was used as mobile phase. For the detection of hexanal, the temperature was kept at 60°C for 6 minutes and subsequently raised to 210°C at a rate of 12°C/min. Then the temperature was kept at 210°C for 1.5 minutes. The reaction mixtures showed a peak at about 6.9 min indicative for hexanal with no peak at about 9.5 min which would be indicative for hexanol.

Example 3: Preparation of hexanal

3 mL (24 mmol) of hexanoic acid were contacted with 0.669 g of silver catalyst having a total surface of 1.27 cm² and heated with a Bunsen burner for about 3 minutes. Subsequently, the reaction mixture was subjected to Fehling analysis which was positive indicating the presence of hexanal.

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Claims

1. Process for the preparation of a fatty aldehyde comprising
 - (a) providing a fatty acid, and
 - (b) contacting the fatty acid with a zerovalent metal catalyst to reduce the fatty acid to the fatty aldehyde, wherein the zerovalent metal catalyst is in the form of solid particles.
2. Process according to claim 1, wherein the zerovalent metal catalyst is a bulk catalyst.
3. Process according to claim 1 or 2, wherein the solid particles have a specific surface area of 0.0001 to 1080 m²/g, preferably 0.0003 to 1 m²/g, more preferably 0.005 to 0.002 m²/g, even more preferably 0.001 to 0.01 m²/g.
4. Process according to claims 1 to 3, wherein the solid particles have an average particle size of 70 nm to 3000 µm, preferably 5 to 2500 µm, such as 5 to 20 µm or 50 to 150 µm or 500 to 2500 µm.
5. Process according to any one of claims 1 to 4, wherein the metal of the zerovalent metal catalyst has a standard electrode potential of less than 1.40 V, preferably less than 1.20 V, more preferably less than 0.80 V.
6. Process according to claim 5, wherein the metal is selected from the group consisting of Fe, Cu, Ag, Hg, Au, Pt and Sn and combinations thereof, and preferably is Fe or Cu and more preferably is Fe.



7. Process according to any one of claims 1 to 6, wherein the fatty acid comprises an aliphatic carboxylic acid having 1 to 32 carbon atoms, preferably 4 to 28, more preferably 6 to 18 carbon atoms.
8. Process according to any one of claims 1 to 7, wherein the fatty acid is saturated or unsaturated and preferably is unbranched.
9. Process according to any one of claims 1 to 8, wherein in step (b) the contacting is effected at a ratio of the zerovalent metal catalyst relative to the fatty acid of $8.0 \cdot 10^{-5}$ to $3.1 \cdot 10^{-3}$ m² of catalyst per mmol of fatty acid, preferably $1.0 \cdot 10^{-4}$ to $1.0 \cdot 10^{-3}$ m² of catalyst per mmol of fatty acid, and more preferably $2.0 \cdot 10^{-4}$ to $5.0 \cdot 10^{-4}$ m² of catalyst per mmol of fatty acid.
10. Process according to any one of claims 1 to 9, wherein in step (b) the contacting is effected

at a temperature of 80 to 800°C, preferably 80 to 250°C, more preferably 85 to 250°C, most preferably 90 to 220°C, such as about 90°C or 180 to 220°C; and/or

at a pressure of 100 to 1200 mbar, preferably 100 to 300 mbar or about 1000 mbar; and/or

for a time of 3 to 180 min, preferably 15 to 120 min, more preferably 20 to 90 min, most preferably 30 to 70 min.
11. Process according to any one of claims 1 to 10, wherein in step (b) the contacting is effected at a concentration of water of less than 10 wt.-%, preferably less than 2 wt.-%, more preferably 0 to less than 1 wt.-%.



12. Process according to any one of claims 1 to 12, wherein in step (b) the contacting is effected at a concentration of oxygen of less than 20 wt.-%, preferably less than 19 wt.-%, more preferably 0 to 1 wt.-%, such as 0 to 0.1 or 0 to 0.01 wt.-%.
13. Process according to any one of claims 1 to 12, wherein in step (b) the contacting is effected at a concentration of hydrogen of about 0 wt.-%.
14. Process according to any one of claims 1 to 13, wherein in step (a) the fatty acid is provided by
 - (a1) providing a fatty acid ester, and
 - (a2) contacting the fatty acid ester with an enzymatic catalyst to hydrolize the fatty acid ester to the fatty acid.
15. Process according to claim 14, wherein the enzymatic catalyst is a hydrolase, preferably a hydrolase of subclass EC 3.1 such as subclass 3.1.1 like 3.1.1.1 and 3.1.1.3, more preferably an esterase like a lipase, such as e.g. lipase *B candida antarctica*.
16. Process according to any one of claims 1 to 15, wherein the fatty aldehyde obtained in step (b) comprises 1 to 32 carbon atoms, and preferably comprises hexanal, octanal, decanal, dodecanal or mixtures thereof.
17. Use of a fatty aldehyde obtained by a process according to any one of claims 1 to 16 as a flavoring agent, fragrance or as an intermediate product for the production of plastifiers, lubricating agents, polymers of pharmaceuticals.



Patentansprüche

1. Verfahren zur Herstellung eines Fetaldehyds, bei dem
 - (a) eine Fettsäure bereitgestellt wird und
 - (b) die Fettsäure mit einem nullwertigen Metallkatalysator in Kontakt gebracht wird, um die Fettsäure zu einem Fetaldehyd zu reduzieren, wobei der nullwertige Metallkatalysator in Form von festen Partikeln vorliegt.
2. Verfahren nach Anspruch 1, wobei der nullwertige Metallkatalysator ein Bulk-Katalysator ist.
3. Verfahren nach Anspruch 1 oder 2, wobei die festen Partikel eine spezifische Oberfläche von 0,0001 bis 1080 m²/g, vorzugsweise 0,0003 bis 1 m²/g, insbesondere 0,005 bis 0,002 m²/g, besonders bevorzugt 0,001 bis 0,01 m²/g, aufweisen.
4. Verfahren nach Ansprüchen 1 bis 3, wobei die festen Partikel eine mittlere Partikelgröße von 70 nm bis 3000 µm, vorzugsweise 5 bis 2500 µm, wie beispielsweise 5 bis 20 µm oder 50 bis 150 µm oder 500 bis 2500 µm, aufweisen.
5. Verfahren nach einem der Ansprüche 1 bis 4, wobei das Metall des nullwertigen Metallkatalysators ein Standardelektrodenpotential von weniger als 1,40 V, vorzugsweise weniger als 1,20 V, insbesondere weniger als 0,80 V, aufweist.
6. Verfahren nach Anspruch 5, bei dem das Metall ausgewählt ist aus der Gruppe bestehend aus Fe, Cu, Ag, Hg, Au, Pt

und Sn und deren Kombinationen, und vorzugsweise Fe oder Cu ist, und insbesondere Fe ist.

7. Verfahren nach einem der Ansprüche 1 bis 6, wobei die Fettsäure eine aliphatische Carbonsäure umfasst, die 1 bis 32, vorzugsweise 4 bis 28, insbesondere 6 bis 18 Kohlenstoffatome aufweist.
8. Verfahren nach einem der Ansprüche 1 bis 7, wobei die Fettsäure gesättigt oder ungesättigt ist, und vorzugsweise unverzweigt ist.
9. Verfahren nach einem der Ansprüche 1 bis 8, wobei in Schritt (b) das Kontaktieren bei einem Verhältnis von nullwertigem Metallkatalysator bezogen auf die Fettsäure von $8,0 \cdot 10^{-5}$ bis $3,1 \cdot 10^{-3}$ m² Katalysator pro mmol Fettsäure, vorzugsweise $1,0 \cdot 10^{-4}$ bis $1,0 \cdot 10^{-3}$ m² Katalysator pro mmol Fettsäure und insbesondere $2,0 \cdot 10^{-4}$ bis $5,0 \cdot 10^{-4}$ mmol Katalysator pro mmol Fettsäure, durchgeführt wird.
10. Verfahren nach einem der Ansprüche 1 bis 9, wobei in Schritt (b) das Kontaktieren

bei einer Temperatur von 80 bis 800°C, vorzugsweise 80 bis 250°C, insbesondere 85 bis 250°C, besonders bevorzugt 90 bis 220°C, wie beispielsweise etwa 90°C oder 180°C oder 220°C, und/oder

bei einem Druck von 100 bis 1200 mbar, vorzugsweise 100 bis 300 mbar oder etwa 1000 mbar, und/oder

für eine Dauer von 3 bis 180 min, vorzugsweise 15 bis 120 min, insbesondere 20 bis 90 min, besonders bevorzugt 30 bis 70 min,

durchgeführt wird.

11. Verfahren nach einem der Ansprüche 1 bis 10, wobei in Schritt (b) das Kontaktieren bei einer Wasserkonzentration von weniger als 10 Gew.-%, vorzugsweise weniger als 2 Gew.-%, insbesondere 0 bis weniger als 1 Gew.-%, durchgeführt wird.
12. Verfahren nach einem der Ansprüche 1 bis 12, wobei in Schritt (b) das Kontaktieren bei einer Sauerstoffkonzentration von weniger als 20 Gew.-%, vorzugsweise weniger als 19 Gew.-%, insbesondere 0 bis 1 Gew.-%, wie beispielsweise 0 bis 0,1 oder 0 bis 0,01 Gew.-%, durchgeführt wird.
13. Verfahren nach einem der Ansprüche 1 bis 12, wobei in Schritt (b) das Kontaktieren bei einer Wasserstoffkonzentration von etwa 0 Gew.-% durchgeführt wird.
14. Verfahren nach einem der Ansprüche 1 bis 13, wobei in Schritt (a) die Fettsäure dadurch bereitgestellt wird, dass
 - (a1) ein Fettsäureester bereitgestellt wird, und
 - (a2) der Fettsäureester mit einem enzymatischen Katalysator in Kontakt gebracht wird, um den Fettsäureester zur Fettsäure zu hydrolysieren.
15. Verfahren nach Anspruch 14, wobei der enzymatische Katalysator eine Hydrolase, vorzugsweise eine Hydrolase der Unterklasse EC 3.1, wie beispielsweise Unterklasse 3.1.1, wie 3.1.1.1 und 3.1.1.3, insbesondere eine Esterase, wie eine Lipase, wie beispielsweise *Candida antarctica* Lipase B, ist.

16. Verfahren nach einem der Ansprüche 1 bis 15, wobei das in Schritt (b) erhaltene Fettaldehyd 1 bis 32 Kohlenstoffatome umfasst, und vorzugsweise Hexanal, Octanal, Decanal, Dodecanal oder deren Mischungen umfasst.
17. Verwendung von einem Fettaldehyd, das durch ein Verfahren nach einem der Ansprüche 1 bis 16 erhalten wurde, als ein Geschmacksstoff, Duftstoff oder als ein Zwischenprodukt für die Herstellung von Weichmachern, Schmiermitteln, Polymeren von Arzneimitteln.