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BATCH PROCESS FOR PREPARING A POLYETHER ALCOHOL
USING A DOUBLE METAL CYANIDE CATALYST

Field of the invention

The present invention relates to a process for preparing a polyether alcohol, to the polyether alcohol obtainable by
5 said process, to a process for preparing a polyurethane foam using said polyether alcohol, to the polyurethane foam obtainable by said process, and to a shaped article comprising said polyurethane foam.

Background of the invention

10 Polyether alcohols, such as polyether polyols, are commonly used for the manufacture of polyurethane foams, such as flexible polyurethane foams, which have found extensive use in a multitude of industrial and consumer applications. Polyether alcohols are also frequently referred to as
15 polyoxyalkylene alcohols. Polyether alcohols are typically obtained by reacting a starter compound or initiator having one active hydrogen atom or a plurality of active hydrogen atoms, such as glycerol, with one or more alkylene oxides, such as ethylene oxide and propylene oxide. Known suitable
20 catalysts for this reaction comprise composite metal cyanide complex catalysts, which are frequently also referred to as double metal cyanide (DMC) catalysts.

Advantages associated with DMC-catalysed production of polyether alcohols is that it is faster and more efficient
25 than the traditional process using potassium hydroxide (KOH) as catalyst. Further, the DMC-catalysed process is more environmentally friendly and has a decreased carbon (CO₂) footprint. When the DMC-catalysed process is run as a batch process, the DMC catalyst is first activated. It is known to
30 combine, at the beginning of a batch process, a small amount

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of alkylene oxide, for example propylene oxide, with a reactive compound, such as some polyether alcohol from a previous batch, in the presence of the DMC catalyst. After some time, the DMC catalyst is activated, as shown by a drop
5 in alkylene oxide pressure. Then at some point after activation, more alkylene oxide and the initiator may be added continuously to produce the desired polyether alcohol.

WO201703748 discloses a process for making a polyether polyol product, comprising: a) activating a DMC catalyst
10 complex in the presence of i) an alkoxyated starter having a hydroxyl equivalent weight of 50 to 100% of the hydroxyl equivalent weight of the polyether polyol product and ii) up to 10 weight-%, based on the weight of the alkoxyated starter, of 1,2-propylene oxide; b) continuously feeding an
15 alkylene oxide containing at least 50 weight-% 1,2- propylene oxide to the activated DMC catalyst under polymerization conditions, wherein starting at the beginning of step b), a low equivalent weight starter is fed continuously to the activated DMC catalyst under polymerization conditions, the
20 feed of low molecular weight starter is continued until 80 to 95% of the alkylene oxide feed in step b) has been completed and the feed of low molecular weight starter is then discontinued while continuing the alkylene oxide feed, wherein the total weight of low molecular weight starter
25 added in step b) is 0.2 to 25% of the total weight of alkylene oxide added in step b).

It is an object of the present invention to provide a batchwise polyether alcohol production process comprising a separate initial DMC catalyst activation step, wherein the
30 final polyether alcohol product has a relatively low polydispersity and viscosity, which may be demonstrated by a relatively small fraction of high molecular weight polyether

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alcohol. A relatively low polydispersity is indicative of a relatively narrow molecular weight distribution.

Summary of the invention

Surprisingly it was found that the above-mentioned object
5 may be achieved by a batch polyether alcohol production
process, wherein a polyether alcohol having a hydroxyl number
of greater than 115 mg KOH/g is prepared by reacting one or
more starter compounds with one or more alkylene oxides in
the presence of a composite metal cyanide complex catalyst (a
10 double metal cyanide (DMC) catalyst) and wherein alkylene
oxide and starter compound are continuously added,
characterized in that in a separate initial DMC catalyst
activation step more than 10 wt.% of an alkylene oxide is
added, based on the weight of a starter mixture comprising a
15 starter compound and the catalyst.

Accordingly, the present invention relates to a batch
process for preparing a polyether alcohol P having a hydroxyl
number of greater than 115 mg KOH/g by reacting starter
compound S₁ and starter compound S₂, which starter compounds
20 have one active hydrogen atom or a plurality of active
hydrogen atoms, with one or more alkylene oxides in the
presence of a composite metal cyanide complex catalyst,
comprising

a) forming a starter mixture comprising starter compound
25 S₁ and the catalyst and activating the catalyst by adding
more than 10 wt.% of an alkylene oxide, based on the weight
of the starter mixture before adding the alkylene oxide,
followed by

b) continuously adding an alkylene oxide; and
30 c) continuously adding starter compound S₂;

wherein

starter compound S₁ has (I) a nominal functionality which
equals the nominal functionality of polyether alcohol P and a

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hydroxyl number which is within 10% of the hydroxyl number of polyether alcohol P and/or (II) an equivalent weight of from 10 to 10,000 g/mol;

5 starter compound S₂ has an equivalent weight of from 10 to 300 g/mol;

no alkylene oxide is added between steps a) and b), and the continuous addition of alkylene oxide in step b) is not interrupted before the total weight of alkylene oxide needed to prepare polyether alcohol P has been added.

10 Further, the present invention relates to a polyether alcohol obtainable by the above-mentioned process.

The present invention also relates to a process for preparing a polyurethane foam comprising reacting a polyether alcohol and a polyisocyanate in the presence of a blowing
15 agent, wherein the polyether alcohol is a polyether alcohol obtained or obtainable by the above-mentioned process.

Further, the present invention relates to a polyurethane foam obtainable by the above-mentioned process for preparing a polyurethane foam, and to a shaped article comprising a
20 polyurethane foam obtained or obtainable by said process.

Detailed description of the invention

While the processes and compositions of the present invention may be described in terms of "comprising", "containing" or "including" one or more various described
25 steps and components, respectively, they can also "consist essentially of" or "consist of" said one or more various described steps and components, respectively.

In the context of the present invention, in a case where a composition comprises two or more components, these
30 components are to be selected in an overall amount not to exceed 100 wt.%.

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Where upper and lower limits are quoted for a property then a range of values defined by a combination of any of the upper limits with any of the lower limits is also implied.

The term "molecular weight" (or "MW") is used herein to
5 refer to number average molecular weight, unless otherwise specified or context requires otherwise. The number average molecular weight of a polyether alcohol can be measured by gel permeation chromatography (GPC) or vapor pressure osmometry (VPO).

10 The term "hydroxyl (OH) value" or "hydroxyl (OH) number" is used herein to refer to the milligrams of potassium hydroxide equivalent to the hydroxyl content in one gram of polyether alcohol determined by wet method titration. Hence, said OH value or number is expressed in mg KOH/g. The
15 hydroxyl number may be determined according to ASTM D4274.

The term "equivalent weight" (or "EW") is used herein to refer to the weight of polyether alcohol per reactive site. The equivalent weight is 56,100 divided by the hydroxyl value of the polyether alcohol.

20 The term "functionality" or "hydroxyl (OH) functionality" of a polyether alcohol refers to the number of hydroxyl groups per molecule of polyether alcohol. The nominal functionality of a polyether alcohol is the same as that of its starter compound (initiator). Unless indicated otherwise,
25 functionality refers to the actual average functionality which may be lower than the nominal functionality and is determined by the number average molecular weight of the polyether alcohol divided by the equivalent weight of the polyether alcohol.

30 The term "primary hydroxyl content" (or "PHC") is used herein to refer to the relative proportion (in %) of primary hydroxyl groups in a polyether alcohol based on total number of hydroxyl groups including primary and secondary hydroxyl

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groups. The primary hydroxyl content may be determined according to ASTM D4273.

The terms "ethylene oxide content" and "propylene oxide content", respectively, in relation to a polyether alcohol
5 refer to those parts of the polyether alcohol which are derived from ethylene oxide and propylene oxide, respectively. Said contents may also be referred to as oxyethylene content and oxypropylene content, respectively. Further, said contents are based herein on total alkylene
10 oxide weight. The ethylene oxide content may be determined according to ASTM D4875.

The process of the present invention is a batch process. In a batch process, the desired product which in the present invention is polyether alcohol P, is not continuously
15 prepared in a reactor but is prepared during a certain period of time in the reactor, after which at least part of the product is recovered, after which a new batch can be started.

In the present invention, polyether alcohol P has a hydroxyl number of greater than 115 mg KOH/g, suitably
20 greater than 120 mg KOH/g. The hydroxyl number of polyether alcohol P may be at least 120 mg KOH/g or at least 130 mg KOH/g or at least 140 mg KOH/g or at least 160 mg KOH/g or at least 180 mg KOH/g or at least 200 mg KOH/g or at least 220 mg KOH/g. Further, the hydroxyl number of polyether alcohol P
25 may be at most 500 mg KOH/g or at most 450 mg KOH/g or at most 400 mg KOH/g or at most 350 mg KOH/g or at most 300 mg KOH/g or at most 280 mg KOH/g.

Further, in the present invention, polyether alcohol P contains ether linkages (or ether units). Further, said
30 polyether alcohol may additionally contain ester linkages (or ester units) and/or carbonate linkages (or carbonate units). It is preferred that said polyether alcohol does not contain ester linkages (or ester units). Further, it is preferred

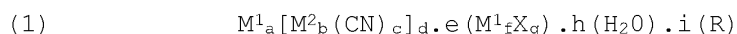
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that said polyether alcohol does not contain carbonate linkages (or carbonate units). Still further, said polyether alcohol may consist of ether linkages.

Still further, in the present invention, polyether
5 alcohol P contains one or more hydroxyl groups. Thus, polyether alcohol P may be a polyether monol or a polyether polyol. A monol is an alcohol containing one hydroxyl group, whereas a polyol is an alcohol containing two or more hydroxyl groups.

10 In step a) of the present process, a starter mixture comprising starter compound S₁ and a composite metal cyanide complex catalyst is formed. Step a) is performed before steps b) and c) are performed. Preferably, in step a), the starter mixture is formed in a reactor. Alternatively, said starter
15 mixture may be formed outside the reactor after which the reactor is charged with the thus obtained starter mixture.

Thus, in the process of the present invention, a composite metal cyanide complex catalyst is used. Composite metal cyanide complex catalysts are frequently also referred
20 to as double metal cyanide (DMC) catalysts. A composite metal cyanide complex catalyst is typically represented by the following formula (1):



wherein each of M¹ and M² is a metal, X is a halogen
25 atom, R is an organic ligand, and each of a, b, c, d, e, f, g, h and i is a number which is variable depending upon the atomic balances of the metals, the number of organic ligands to be coordinated, etc.

In the above formula (1), M¹ is preferably a metal
30 selected from Zn(II) or Fe(II). In the above formula, M² is preferably a metal selected from Co(III) or Fe(III). However, other metals and oxidation states may also be used, as is known in the art.

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In the above formula (1), R is an organic ligand and is preferably at least one compound selected from the group consisting of an alcohol, an ether, a ketone, an ester, an amine and an amide. As such an organic ligand, a water-soluble one may be used. Specifically, one or more compounds selected from tert-butyl alcohol, n-butyl alcohol, iso-butyl alcohol, tert-pentyl alcohol, isopentyl alcohol, N, N-dimethyl acetamide, glyme (ethylene glycol dimethyl ether), diglyme (diethylene glycol dimethyl ether), triglyme (triethylene glycol dimethyl ether), ethylene glycol mono-tert-butylether, iso-propyl alcohol and dioxane, may be used as organic ligand(s). The dioxane may be 1,4-dioxane or 1,3-dioxane and is preferably 1,4-dioxane. Most preferably, the organic ligand or one of the organic ligands in the composite metal cyanide complex catalyst is tert-butyl alcohol.

Further, as an alcohol organic ligand, a polyol, preferably a polyether polyol may be used. More preferably, a poly(propylene glycol) having a number average molecular weight in the range of from 500 to 2,500 Dalton, preferably 800 to 2,200 Dalton, may be used as the organic ligand or one of the organic ligands. Most preferably, such poly(propylene glycol) is used in combination with tert-butyl alcohol as organic ligands. The composite metal cyanide complex catalyst can be produced by known production methods.

In the present invention, starter compound S₁ meets one or both of the following two requirements (I) and (II):

(I) starter compound S₁ has a nominal functionality which equals the nominal functionality of polyether alcohol P and a hydroxyl number which is within 10% of the hydroxyl number of polyether alcohol P and/or

(II) starter compound S₁ has an equivalent weight of from 10 to 10,000 g/mol.

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Thus, in the present invention, compound S₁ may meet requirement (I) only or may meet requirement (II) only or may meet both requirements (I) and (II).

Under requirement (I), starter compound S₁ has a hydroxyl
5 number which is within 10% of the hydroxyl number of
polyether alcohol P. This means that under requirement (I),
the hydroxyl number of starter compound S₁ does not differ by
more than 10% from the hydroxyl number of polyether alcohol
P. Preferably, under requirement (I), starter compound S₁ has
10 a hydroxyl number which is within 8%, more preferably within
6%, more preferably within 4%, more preferably within 2%,
most preferably within 1% of the hydroxyl number of polyether
alcohol P. Further, starter compound S₁ may have a hydroxyl
number which equals the hydroxyl number of polyether alcohol
15 P.

Requirement (I) is met in a case wherein a portion of
polyether alcohol P as prepared in a previous batch in
accordance with the process of the present invention, is used
as starter compound S₁ in a next batch in accordance with the
20 process of the present invention. Thus, starter compound S₁
may comprise the same product as the final targeted product
(polyether alcohol P). Up to 50 wt.% or up to 40 wt.% or up
to 30 wt.% or up to 20 wt.% or up to 10 wt.% or up to 5 wt.%
of the total weight of polyether alcohol P as prepared in
25 said previous batch may be used as starter compound S₁ in
said next batch. At the end of a batch process a portion of
said polyether alcohol P thus prepared may be left in a
reactor (generally also referred to as "heel") and be used as
starter compound S₁ in the next batch. It is also possible
30 that said polyether alcohol P thus prepared is first stored
in a separate storage vessel, and that later a portion
thereof is brought back into the reactor and used as starter
compound S₁ in the next batch. Prior to step a) of the

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present process, above-mentioned "heel" may be subjected to a pre-treatment, wherein such pre-treatment may for example comprise stripping using a stripping gas in order to remove light compounds (such as moisture) and/or refining in order to remove or neutralize any non-DMC catalyst (such as KOH) used in a previous batch.

Under requirement (II), starter compound S₁ has an equivalent weight of from 10 to 10,000 g/mol. Under said requirement (II), starter compound S₁ may have an equivalent weight of at least 40 g/mol or at least 45 g/mol or at least 50 g/mol or at least 55 g/mol or at least 65 g/mol or at least 80 g/mol or at least 100 g/mol or at least 120 g/mol or at least 140 g/mol or at least 160 g/mol or at least 180 g/mol or at least 200 g/mol. Further, under said requirement (II), starter compound S₁ may have an equivalent weight of at most 8,000 g/mol or at most 6,000 g/mol or at most 4,000 g/mol or at most 3,000 g/mol or at most 2,700 g/mol or at most 2,400 g/mol or at most 2,200 g/mol or at most 2,000 g/mol or at most 1,500 g/mol or at most 1,000 g/mol or at most 500 g/mol or at most 450 g/mol or at most 400 g/mol or at most 350 g/mol or at most 300 g/mol or at most 280 g/mol or at most 250 g/mol.

Further, under requirement (II), starter compound S₁ may have a functionality of from 1 to 8, preferably of from 2 to 6, more preferably of from 2 to 4, more preferably of from 2.5 to 3.5, most preferably of from 2.7 to 3.3.

A case wherein only requirement (II) is met, and requirement (I) is not met, is a case wherein in a previous batch another polyether alcohol (another grade) is prepared, which has a nominal functionality which is different from that of the desired polyether alcohol P to be made in the next batch and/or which has a hydroxyl number which differs by more than 10% from the hydroxyl number of said polyether

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alcohol P. When such grade change is carried out, a portion of the other polyether alcohol as prepared in a previous batch, may be used as starter compound S_1 in a next batch wherein polyether alcohol P is prepared in accordance with the process of the present invention. Thus, starter compound S_1 may comprise a product which is different from the final targeted product (polyether alcohol P). Up to 50 wt.% or up to 40 wt.% or up to 30 wt.% or up to 20 wt.% or up to 10 wt.% or up to 5 wt.% of the total weight of said other polyether alcohol as prepared in said previous batch may be used as starter compound S_1 in said next batch. At the end of a batch process a portion of said other polyether alcohol thus prepared may be left in a reactor (generally also referred to as "heel") and be used as starter compound S_1 in the next batch wherein polyether alcohol P is prepared. It is also possible that said other polyether alcohol thus prepared is first stored in a separate storage vessel, and that later a portion thereof is brought back into the reactor and used as starter compound S_1 in the next batch wherein polyether alcohol P is prepared. Prior to step a) of the present process, above-mentioned "heel" may be subjected to a pre-treatment, wherein such pre-treatment may for example comprise stripping using a stripping gas in order to remove light compounds (such as moisture) and/or refining in order to remove or neutralize any non-DMC catalyst (such as KOH) used in a previous batch.

The amount of starter compound S_1 used in step a) of the present process, on the basis of the total weight of final product (polyether alcohol P) in the reactor, may vary within wide ranges. Said proportion may be of from 1 to 80 wt.%, or 3 to 70 wt.%, or 5 to 60 wt.%, or 7 to 50 wt.%, or 8 to 40 wt.%. Said proportion is related to the so-called "build ratio" which in the present specification is defined as the

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ratio of the total weight of final product in the reactor to the weight of starter compound S₁.

Starter compound S₁ may consist of one starter compound which meets one or both of requirements (I) and (II).

5 Alternatively, starter compound S₁ may consist of a mixture of two or more starter compounds, suitably two starter compounds, each of which meets one or both of requirements (I) and (II). In the latter case, for example, one of the starter compounds may meet requirements (I) and (II) whereas
10 another starter compound may only meet requirement (II). Further, additionally, in step a) of the present process, one or more starter compounds other than starter compound S₁, may be used in forming the starter mixture. Preferably, in the present invention, starter compounds not meeting any one of
15 requirements (I) and (II) are not used in step a).

In the present invention, step b) may start before step c) or steps b) and c) may start simultaneously or step c) may start before step b). It is preferred that step b) starts before step c). In the present invention, polyether alcohol P
20 is prepared in a reactor. However, as mentioned above, step a) may be performed within the reactor or, alternatively, may be performed outside the reactor after which the reactor is charged with the thus obtained starter mixture. Steps b) and c) are performed within the reactor, meaning that in step b)
25 alkylene oxide is continuously added to the reactor, and in step c) starter compound S₂ is continuously added to the reactor.

In step a) of the present process, a starter mixture comprising starter compound S₁ and a composite metal cyanide
30 complex catalyst is formed. In step a), starter compound S₁ may be combined with composite metal cyanide complex catalyst as described above, wherein said catalyst to be combined with starter compound S₁ preferably comprises fresh composite

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metal cyanide complex catalyst. Within the present specification, a "fresh" catalyst means a not-activated catalyst which has not been used as a catalyst in a chemical process before, in specific a not-activated catalyst which
5 has not been exposed to alkylene oxide before. The fresh catalyst is, however, suitable to be used as a catalyst in a chemical process, which means that it is a final catalyst obtained as the product in a catalyst preparation process, and not any intermediate catalyst or catalyst precursor.
10 Within the present specification, a "used" catalyst means a catalyst which has been used as a catalyst in a chemical process before, in specific a catalyst which has been exposed to alkylene oxide before.

Above-mentioned fresh composite metal cyanide complex
15 catalyst which is preferably used in step a) should be distinguished from any composite metal cyanide complex catalyst that may be present in starter compound S_1 before forming, in said step a), a starter mixture comprising starter compound S_1 and a composite metal cyanide complex
20 catalyst which preferably comprises fresh composite metal cyanide complex catalyst. Composite metal cyanide complex catalyst present in starter compound S_1 before step a), may originate from a previous batch wherein a composite metal cyanide complex catalyst is also used in preparing a
25 polyether alcohol, a portion of which polyether alcohol may subsequently be used as starter compound S_1 in a next batch wherein polyether alcohol P is prepared in accordance with the process of the present invention. Hence, starter compound S_1 may comprise a used composite metal cyanide complex
30 catalyst. Further, preferably, starter compound S_1 does not comprise a fresh composite metal cyanide complex catalyst.

Hence, in the present invention, polyether alcohol P is prepared in the presence of a composite metal cyanide complex

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catalyst which comprises (i) composite metal cyanide complex catalyst used in step a) to form a starter mixture comprising starter compound S₁ and said catalyst, which catalyst (i) is activated in step a), as further discussed below, and
5 preferably comprises fresh catalyst, and (ii) optionally composite metal cyanide complex catalyst present in starter compound S₁ before step a), which catalyst (ii) may be activated in step a), as further discussed below, and may comprise used catalyst.

10 In the present invention, starter compound S₂ is added in step c). In the present invention, polyether alcohol P is prepared in a reactor. Step a) may be performed partially (i.e. catalyst activation only) or completely within the reactor or, alternatively, may be performed partially (i.e.
15 formation of starter mixture before catalyst activation) or completely outside the reactor after which the reactor is charged with the thus obtained starter mixture. Steps b) and c) are performed within the reactor, meaning that in step b) alkylene oxide is continuously added to the reactor, and in
20 step c) starter compound S₂ is continuously added to the reactor.

Further, in the present invention, no alkylene oxide is added between steps a) and b). This means that in the present invention, alkylene oxide is only added in steps a) and b).
25 Further, in the present invention, the continuous addition of alkylene oxide in step b) is not interrupted before the total weight of alkylene oxide needed to prepare polyether alcohol P has been added. This means that in the present invention, the continuous addition of alkylene oxide in step b) is not
30 discontinued temporarily but is only stopped once the total weight of alkylene oxide needed to prepare polyether alcohol P has been added.

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In the present invention, in step a), an alkylene oxide is added to the starter mixture comprising starter compound S₁ and the composite metal cyanide complex catalyst, in order to activate said catalyst. Further, in the present invention, 5 the amount of alkylene oxide added in step a) is of from more than 10 wt.%, based on the weight of the starter mixture before adding the alkylene oxide (i.e. the alkylene oxide added to activate the catalyst). Said amount of alkylene oxide added in step a) may be of from more than 10 wt.% to 40 10 wt.% or of from 11 to 35 wt.% or of from 12 to 30 wt.% or of from 13 to 25 wt.% or of from 14 to 20 wt.%, based on the weight of the starter mixture before adding the alkylene oxide. Any alkylene oxide may be added in step a) to achieve such catalyst activation. The alkylene oxide added in step a) 15 may comprise one or more of propylene oxide, ethylene oxide and butylene oxide, preferably propylene oxide and/or butylene oxide, most preferably only propylene oxide. Further, as mentioned above, no alkylene oxide is added between steps a) and b). Still further, (i) completing the 20 addition of alkylene oxide in step a) and (ii) starting the continuous alkylene oxide addition in step b) do not immediately follow each other. For step b) is only started after the catalyst is activated in step a). Such catalyst activation can be confirmed by a drop in the pressure of 25 alkylene oxide in the reactor. Such pressure drop indicates that alkylene oxide reacts away and hence the catalyst is activated.

As mentioned above, in the present invention, alkylene oxide is only added in steps a) and b), and the continuous 30 addition of alkylene oxide in step b) is only stopped once the total weight of alkylene oxide needed to prepare polyether alcohol P has been added. Said alkylene oxide added in step b) may comprise one or more of propylene oxide,

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ethylene oxide and butylene oxide, preferably propylene oxide and ethylene oxide, most preferably only propylene oxide.

Polyether alcohol P prepared in the process of the present invention, comprises polyether chains preferably
5 containing propylene oxide content, optionally butylene oxide content and optionally ethylene oxide content.

The propylene oxide content of polyether alcohol P may be at least 10% or at least 20 wt.% or at least 30 wt.% or at least 40% or at least 50 wt.% or at least 60 wt.% or at least
10 70 wt.% or at least 80 wt.% or at least 90 wt.% or at least 95 wt.% or at least 99 wt.%. Further, the propylene oxide content of polyether alcohol P may be at most 100 wt.% or at most 90 wt.% or at most 80 wt.% or at most 70 wt.% or at most 60 wt.% or at most 50 wt.% or at most 40 wt.% or at most 30
15 wt.% or at most 20 wt.%.

The ethylene oxide content of polyether alcohol P may be 0 wt.% or at least 3 wt.% or at least 5 wt.% or at least 10 wt.% or at least 12 wt.% or at least 15 wt.% or at least 20 wt.% or at least 30 wt.% or at least 40% or at least 50 wt.%
20 or at least 60 wt.% or at least 70 wt.% or at least 80 wt.% or at least 90 wt.%. Further, the ethylene oxide content of polyether alcohol P may be at most 90 wt.% or at most 80 wt.% or at most 70 wt.% or at most 60 wt.% or at most 50 wt.% or at most 40 wt.% or below 30 wt.% or at most 25 wt.% or at
25 most 20 wt.% or at most 15 wt.% or at most 12 wt.%.

The polyether chains of the polyether alcohol P may comprise no ethylene oxide content but may comprise only propylene oxide and/or butylene oxide content, suitably only propylene oxide content.

30 Further, polyether alcohol P may comprise primary hydroxyl groups. The primary hydroxyl content of the polyether alcohol P may be 0% or at least 1% or at least 3% or at least 5% or at least 10% or at least 20% or at least

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30%. Further, the primary hydroxyl content of polyether alcohol P may be at most 90% or at most 80% or at most 70% or at most 60% or at most 50% or at most 40% or at most 30% or at most 20% or at most 15% or at most 10% or at most 5%.

5 Further, polyether alcohol P may have a functionality of from 0.8 to 8, preferably of from 1 to 8, more preferably of from 2 to 6, more preferably of from 2 to 4, more preferably of from 2.5 to 3.5, most preferably of from 2.7 to 3.3.

10 Preferably, in the beginning of step b), the addition rate of alkylene oxide is increased till a target addition rate is reached which is then preferably maintained till the end of step b).

In the present invention, starter compound S₂ has an equivalent weight of from 10 to 300 g/mol. Preferably, 15 starter compound S₂ has an equivalent weight of from 10 to 250 g/mol, more preferably 10 to 200 g/mol, more preferably 10 to 150 g/mol, more preferably 10 to 100 g/mol, more preferably 10 to 80 g/mol, more preferably 10 to 70 g/mol, more preferably 10 to 60 g/mol, more preferably 20 to 50 20 g/mol, more preferably 25 to 40 g/mol, most preferably 30 to 35 g/mol.

Further, preferably, starter compound S₂ is a polyfunctional alcohol, generally containing from 1 to 8 or 2 to 6 or 2 to 4 hydroxyl groups. Examples of such alcohols 25 comprise n-butanol, allyl alcohol, glycols, glycerol, pentaerythritol, trimethylolpropane, triethanolamine, sorbitol, mannitol and sucrose. Preferably, starter compound S₂ is selected from the group consisting of glycols, glycerol, pentaerythritol, trimethylolpropane, 30 triethanolamine, sorbitol and mannitol. Advantageously, monopropylene glycol (MPG), glycerol or a combination of both may be used as starter compound S₂.

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Preferably, in the beginning of step c), the addition rate of starter compound S₂ is increased till a target addition rate is reached which is then preferably maintained till the end of step c).

5 Further, in the present invention, it is preferred that once above-mentioned target addition rates for the alkylene oxide and starter compound S₂ have been reached, the weight ratio of the addition rate of the alkylene oxide to the addition rate of starter compound S₂ is of from 2:1 to 10:1
10 or of from 3:1 to 8:1. In specific, it is preferred that the latter weight ratio is smaller than the weight ratio before said target addition rates have been reached, in which earlier stage the weight ratio of the addition rate of the alkylene oxide to the addition rate of starter compound S₂
15 may be of from 8:1 to 30:1 or of from 10:1 to 20:1.

In the present invention, it is preferred that step c) is stopped before step b) is stopped. In particular, step c) may be stopped once of from 5 to 99% or of from 10 to 99% or of from 15 to 99% or of from 20 to 99% or of from 25 to 95% or
20 of from 30 to 90% or of from 50 to 85% or of from 60 to less than 80% or of from 65 to 79% or of from 70 to 79% or of from 75% to 79% or of from 76 to 79% of the total weight of alkylene oxide needed to prepare polyether alcohol P has been added in step b).

25 Further, in the present invention, it is preferred that the total amount of starter compound S₂ added in step c) may be of from 0.1 to 25 wt.% or of from 0.5 to 25 wt.% or of from 2 to 25 wt.% or of from 5 to 25 wt.% or of from 6 to 22 wt.% or of from 10 to 18 wt.%, based on the sum of the total
30 amount of starter compound S₂ added in step c) and the total amount of alkylene oxide added in step b).

In the present invention, the continuous addition of alkylene oxide in step b) may be completed with a space time

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yield (STY) of from 100 to 600 kg/m³/hr, which STY is expressed in kilogram of polyether alcohol P being produced per cubic meter per hour of alkylene oxide addition time in step b), or of from 150 to 500 kg/m³/hr or of from 150 to 400
5 kg/m³/hr or of from 150 to 350 kg/m³/hr or of from 180 to 300 kg/m³/hr or of from 200 to 270 kg/m³/hr or of from 200 to less than 250 kg/m³/hr or of from 220 to less than 250 kg/m³/hr or of from 230 to 245 kg/m³/hr.

Further, the present invention relates to a polyether
10 alcohol obtainable by the above-mentioned process.

The present invention also relates to a process for preparing a polyurethane foam comprising reacting a polyether alcohol and a polyisocyanate in the presence of a blowing agent, wherein the polyether alcohol is a polyether alcohol
15 obtained or obtainable by the above-mentioned batch process and wherein the polyether alcohol is preferably a polyether polyol as described above.

Further, the present invention relates to a process for preparing a polyurethane foam comprising preparing a
20 polyether alcohol P having a hydroxyl number of equal to or lower than 115 mg KOH/g in accordance with the above-mentioned batch process, followed by reacting the polyether alcohol and a polyisocyanate in the presence of a blowing agent.

25 In the above-mentioned process for preparing a polyurethane foam, the polyether alcohol is reacted with a polyisocyanate in the presence of a blowing agent.

The polyisocyanate may comprise an aromatic polyisocyanate or an aliphatic polyisocyanate, preferably an
30 aromatic polyisocyanate.

The aromatic polyisocyanate may for example comprise tolylene diisocyanate (TDI) or polymeric TDI, xylylene diisocyanate, tetramethylxylylene diisocyanate, methylene

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diphenyl diisocyanate (MDI) or polymeric MDI (i.e. polymethylene polyphenyl isocyanate), or a modified product thereof. Preferably, the aromatic polyisocyanate comprises tolylene diisocyanate (TDI), i.e. non-polymeric TDI. The TDI
5 may be a mixture of 80 wt.% of 2,4-TDI and 20 wt.% of 2,6-TDI, which mixture is sold as "TDI-80".

Further, the aliphatic polyisocyanate may for example comprise hexamethylene diisocyanate, dicyclohexylmethane diisocyanate, lysine diisocyanate or isophorone diisocyanate,
10 or a modified product thereof.

Further, the polyisocyanate may comprise any mixture of two or more of the polyisocyanates mentioned above. For example, the polyisocyanate may comprise a mixture of TDI and MDI, in particular a mixture wherein the weight ratio of
15 TDI:MDI varies from 10:90 to 90:10.

The blowing agent may comprise a chemical blowing agent and/or a physical (non-chemical) blowing agent. Within the present specification, by "chemical blowing agent" reference is made to a blowing agent that may only provide a blowing
20 effect after it has chemically reacted with another compound. In case the blowing agent comprises a chemical blowing agent, said chemical blowing agent preferably comprises water. Water reacts with isocyanate groups of the polyisocyanate, thereby releasing carbon dioxide which causes the blowing to occur.

25 However, other suitable blowing agents, such as for example, acetone, gaseous or liquid carbon dioxide, halogenated hydrocarbons, aliphatic alkanes and alicyclic alkanes may be employed additionally or alternatively.

Due to the ozone depleting effect of fully chlorinated,
30 fluorinated alkanes (CFC's) the use of this type of blowing agent is generally not preferred, although it is possible to use them. Halogenated alkanes, wherein at least one hydrogen atom has not been substituted by a halogen atom (including

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the so-called HCFC's) have no or less ozone depleting effect and therefore are the preferred halogenated hydrocarbons to be used in physically blown foams. One suitable HCFC type blowing agent is 1-chloro-1,1-difluoroethane. Another
5 suitable halogenated alkane of this type for use as a blowing agent, is methylene chloride (dichloromethane).

The above blowing agents may be used singly or in mixtures of two or more.

The amount of the blowing agent(s) is determined by the
10 desired density of the polyurethane foam to be prepared. For example, a relatively low density can be obtained by using a relatively high amount of the blowing agent(s), and vice versa. A skilled person can readily determine the amount of blowing agent (physical and/or chemical blowing agent) needed
15 to obtain a desired foam density.

Water may be used as a blowing agent in an amount which is at least 0.1 part per hundred parts by weight of polyether alcohol (pphp) or at least 0.5 pphp or at least 1 pphp. Further, water may be used as a blowing agent in an amount
20 which is at most 10 parts per hundred parts by weight of polyether alcohol (pphp) or at most 5 pphp or at most 3 pphp or at most 2 pphp.

In case of halogenated hydrocarbons, aliphatic alkanes and alicyclic alkanes, the amount of the blowing agent may be
25 of from 1 to 50 parts per hundred parts by weight of polyether alcohol (pphp), suitably of from 1 to 30 pphp, more suitably of from 1 to 20 pphp.

Further, preferably, the polyurethane foam which may be prepared is a flexible polyurethane foam. Further, said
30 flexible polyurethane foam is suitably a slabstock foam. Within the present specification, by "slabstock foam" reference is made to a foam that is made by applying a free rise (unconstrained rise) of the foam.

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The isocyanate index (or NCO index) may vary within wide ranges and may be of from 60 to 120. In particular, the isocyanate index may be at most 120, more suitably at most 110, more suitably at most 100, most suitably at most 90.

5 Further, the isocyanate index is preferably higher than 60 and may be at least 70 or at least 80 or at least 90.

Within the present specification, "isocyanate index" is calculated as 100 times the mole ratio of -NCO groups (isocyanate groups) to NCO-reactive groups in the reaction
10 mixture. In other words, the isocyanate index is defined as:
[(actual amount of isocyanate)/(theoretical amount of isocyanate)]*100, wherein the "theoretical amount of isocyanate" equals 1 equivalent isocyanate (NCO) group per 1 equivalent isocyanate-reactive group.

15 Such "isocyanate-reactive groups" as referred to above include for example OH groups from the polyether alcohol and from any water that may be used as a blowing agent. Isocyanate groups also react with water.

Additionally, other components may also be present during
20 the above-mentioned polyurethane foam preparation process, such as one or more polyurethane catalysts, surfactants and/or cross-linking agents.

Polyurethane catalysts are known in the art and include many different compounds. Suitable catalysts include tin-,
25 lead- or titanium-based catalysts, preferably tin-based catalysts, such as tin salts and dialkyl tin salts of carboxylic acids. Specific examples are stannous octoate, stannous oleate, dibutyltin dilaureate, dibutyltin acetate and dibutyltin diacetate. Other suitable catalysts are
30 tertiary amines, such as, for instance, bis(2,2'-dimethylamino)ethyl ether, trimethylamine, triethylamine, triethylenediamine and dimethylethanolamine (DMEA). Examples of commercially available tertiary amine catalysts are those

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sold under the tradenames Niax, Tegoamin and Dabco (all trademarks). The catalyst is typically used in an amount of from 0.01 to 2.0 parts by weight per hundred parts by weight of polyether alcohol (php). Preferred amounts of catalyst are
5 from 0.05 to 1.0 php.

The use of foam stabilisers (surfactants) is well known. Organosilicone surfactants are most conventionally applied as foam stabilisers in polyurethane production. A large variety of such organosilicone surfactants is commercially available.
10 Usually, such foam stabiliser is used in an amount of from 0.01 to 5.0 parts by weight per hundred parts by weight of polyether alcohol (pphp). Preferred amounts of stabiliser are from 0.25 to 2.0 pphp, more preferably of from 0.75 to 1.5 pphp.

15 The use of cross-linking agents in the production of polyurethane foams is also well known. Polyfunctional glycol amines are known to be useful for this purpose. The polyfunctional glycol amine which is most frequently used and is also useful in the preparation of polyurethane foams,
20 especially flexible polyurethane foams, is diethanolamine, often abbreviated as DEOA. A cross-linking agent may be applied in amounts up to 2 parts by weight per hundred parts by weight of polyether alcohol (pphp), but amounts in the range of from 0.01 to 0.5 pphp are most suitably applied.

25 In addition, other well-known auxiliaries, such as colorants, flame retardants and fillers, may also be used during the above-mentioned polyurethane foam preparation process.

Said polyurethane foam preparation process may involve
30 combining the polyisocyanate, the polyether alcohol, the blowing agent, a catalyst and optionally surfactant, crosslinker, flame retardant, colorant and/or filler, in any suitable manner to obtain the polyurethane foam. For example,

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said process may comprise mixing the polyether alcohol, the blowing agent, a catalyst and any other optional component(s) except the polyisocyanate, and then adding the polyisocyanate.

5 Further, the above-mentioned polyurethane foam preparation process may comprise forming the foam into a shaped article before it fully sets. Suitably, forming the foam may comprise pouring the liquid mixture containing all components into a mould before gelling is complete.

10 Further, the present invention relates to a polyurethane foam obtainable by the above-mentioned process for preparing a polyurethane foam, and to a shaped article comprising a polyurethane foam obtained or obtainable by said process.

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C L A I M S

1. A batch process for preparing a polyether alcohol P having a hydroxyl number of greater than 115 mg KOH/g by reacting starter compound S₁ and starter compound S₂, which starter compounds have one active hydrogen atom or a plurality of active hydrogen atoms, with one or more alkylene oxides in the presence of a composite metal cyanide complex catalyst, comprising
- 5
- a) forming a starter mixture comprising starter compound S₁ and the catalyst and activating the catalyst by adding more than 10 wt.% of an alkylene oxide, based on the weight of the starter mixture before adding the alkylene oxide, followed by
- 10
- b) continuously adding an alkylene oxide; and
- c) continuously adding starter compound S₂;
- 15
- wherein
- starter compound S₁ has (I) a nominal functionality which equals the nominal functionality of polyether alcohol P and a hydroxyl number which is within 10% of the hydroxyl number of polyether alcohol P and/or (II) an equivalent weight of from
- 20
- 10 to 10,000 g/mol;
- starter compound S₂ has an equivalent weight of from 10 to 300 g/mol;
- no alkylene oxide is added between steps a) and b), and the continuous addition of alkylene oxide in step b) is not interrupted before the total weight of alkylene oxide needed to prepare polyether alcohol P has been added.
- 25
2. The process according to claim 1, wherein in step a) of from more than 10 wt.% to 40 wt.% of the alkylene oxide is added, based on the weight of the starter mixture before adding the alkylene oxide.
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3. The process according to claim 1 or 2, wherein in step a) starter compound S₁ is combined with fresh composite metal cyanide complex catalyst.
- 5
4. The process according to any one of claims 1 to 3, wherein step b) starts before step c).
5. The process according to any one of claims 1 to 4,
- 10 wherein step c) is stopped before step b) is stopped.
6. The process according to claim 5, wherein step c) is stopped once of from 5 to 99% of the total weight of alkylene oxide needed to prepare polyether alcohol P has been added in
- 15 step b).
7. A polyether alcohol obtainable by the process according to any one of claims 1-6.
- 20 8. A process for preparing a polyurethane foam comprising reacting a polyether alcohol and a polyisocyanate in the presence of a blowing agent, wherein the polyether alcohol is a polyether alcohol obtained by the process according to any one of claims 1-6 or the polyether alcohol according to claim
- 25 7.
9. A polyurethane foam obtainable by the process according to claim 8.
10. A shaped article comprising a polyurethane foam obtained by the process according to claim 8 or the polyurethane foam according to claim 9.