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(72) Feltaláló(k):

VAN BROEKHOVEN, Emanuel Hermanus, NL-1141 DN**Monnickendam (NL)****HARTE, Mark Hendrikus, NL-1507 TB Zaandam (NL)****KLAVER, Gijsbertus, NL-1452 VN IJpendam (NL)****NIEMAN, Jan, NL-3607 CB Maarssen (NL)**

(73) Jogosult(ak):

Albemarle Netherlands BV, 1030 BE**Amsterdam (NL)**

(74) Képviselő:

SBGK Szabadalmi Ügyvivői Iroda, Budapest(54) **Alkilezési eljárás ritkaföldfém tartalmú zeoliteket és egy hidrogénező fémet tartalmazó katalizátorral**

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(54) **ALKYLATION PROCESS USING A CATALYST COMPRISING RARE EARTH CONTAINING ZEOLITES AND A HYDROGENATION METAL**

ALKYLIERUNGSVERFAHREN MIT EINEM KATALYSATOR MIT SELTENERDE MIT ZEOLITHEN UND EINEM HYDRIERMETALL

PROCEDE D'ALKYLATION AU MOYEN D'UN CATALYSEUR INCLUANT DES ZEOLITES COMPRENANT DES TERRES RARES ET UN METAL D'HYDROGENATION

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(73) Proprietor: **Albemarle Netherlands BV
1030 BE Amsterdam (NL)**

(72) Inventors:

- **VAN BROEKHOVEN, Emanuel Hermanus
NL-1141 DN Monnickendam (NL)**

- **HARTE, Mark Hendrikus
NL-1507 TB Zaandam (NL)**
- **KLAVER, Gijsbertus
NL-1452 VN IJpendam (NL)**
- **NIEMAN, Jan
NL-3607 CB Maarssen (NL)**

(74) Representative: **Schwabe - Sandmair - Marx
Patentanwälte Rechtsanwalt
Partnerschaft mbB
Joseph-Wild-Straße 20
81829 München (DE)**

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Description

[0001] The term alkylation refers to the reaction of an alkylatable compound, such as an aromatic or saturated hydrocarbon, with an alkylation agent, such as an olefin. The reaction is of interest because it makes it possible to obtain, through the alkylation of isobutane with an olefin containing 2-6 carbon atoms, an alkylate which has a high octane number and which boils in the gasoline range. Unlike gasoline obtained by cracking heavier petroleum fractions such as vacuum gas oil and atmospheric residue, gasoline obtained by alkylation is essentially free of contaminants such as sulfur and nitrogen and thus has clean burning characteristics. Its high anti-knock properties, represented by the high octane number, lessen the need to add environmentally harmful anti-knock compounds such as aromatics or lead. Also, unlike gasoline obtained by reforming naphtha or by cracking heavier petroleum fractions, alkylate contains few if any aromatics or olefins, which offers further environmental advantages.

[0002] The alkylation reaction is acid-catalyzed. Conventional alkylation process equipment makes use of liquid acid catalysts such as sulfuric acid and hydrofluoric acid. The use of such liquid acid catalysts is attended with a wide range of problems. For instance, sulfuric acid and hydrofluoric acid are both highly corrosive, so that the equipment used has to meet severe service requirements. Since the presence of highly corrosive materials in the resulting fuel is objectionable, the remaining acid must be removed from the alkylate. Also, because of the liquid phase separations that must be carried out, the process is complicated and expensive. In addition, there is always the risk that toxic substances such as hydrogen fluoride will be emitted to the environment.

[0003] US 3,851,004 discloses that the activity of zeolitic hydrocarbon alkylation catalysts is maintainable for very long periods at commercially feasible levels by employing a Group VIII metal hydrogenation agent as a constituent of the catalyst composition and periodically hydrogenating the catalyst composition with a solution of hydrogen in a hydrocarbon solvent. The document does not disclose the presence of rare earth metals in the catalyst.

[0004] WO 98/23560 A1 describes a process for alkylating hydrocarbons in which an alkylatable organic compound is reacted with an alkylation agent to form an alkylate in the presence of a catalyst comprising a hydrogenation function and a solid acid constituent, with the catalyst being subjected intermittently to a regeneration step by being contacted with feed containing a saturated hydrocarbon and hydrogen, said regeneration being carried out at 90 % or less of the active cycle of the catalyst, with the active cycle of the catalyst being defined as the time from the start of the feeding of the alkylation agent to the moment when 20 % of the alkylation agent leaves the catalyst-containing reactor section without being converted, not counting isomerisation inside the molecule. The catalyst employed in the process does not contain rare earth.

[0005] The present invention provides an improved alkylation process utilizing a solid-acid catalyst comprising a rare earth containing zeolite and a hydrogenation metal as defined in claim 1.

[0006] The water content of the catalyst ranges from 1.5 wt% to 6 wt%, in one embodiment it ranges from 1.8 wt% to 4 wt%, and in another embodiment it ranges from 2 wt% to 3 wt%. The water content of the catalyst is defined as its water content during use in the alkylation process and is measured by determining the weight loss upon heating the catalyst for two hours at 600 °C (Loss on Ignition, or LOI 600).

[0007] The catalyst further comprises a hydrogenation metal selected from platinum, palladium, and mixtures thereof. The amount of hydrogenation metal will depend on its nature. The catalyst generally will contain in the range of 0.01 to 2 wt% of the metal. In one embodiment it ranges from 0.1 to 1 wt%, calculated as metal and based on the total weight of the catalyst.

[0008] The catalyst further comprises a solid acid. According to the invention, the solid acids are Y-zeolites, including H-Y-zeolites and USY-zeolites. Mixtures of solid acids can also be employed. According to the invention, the solid acid is Y-zeolite with a unit cell size of 24.56 - 24.72 angstroms.

[0009] The solid acid component of the catalyst comprises rare earth, i.e., an element chosen from the lanthanide series. In one embodiment, rare earth ranges from 0.5 wt % to 32 wt %. In another, rare earth ranges from 2 wt % to 9 wt %. In yet another, rare earth ranges from 4 wt% to 6 wt%. All references herein to rare earth wt% are calculated as rare earth oxides on a dry basis (600°C, 1 hour).

[0010] The rare earth element(s) may be exchanged into the solid acid component by conventional means. In one embodiment, the solid acid component is a lanthanum exchanged Y-zeolite.

[0011] During the exchange process of the solid acid component sodium (Na⁺) is removed from the catalyst. The solid acid component contains less than 1.5 wt% Na₂O. In another, less than 1.0 wt% Na₂O. In yet another less than 0.6 wt% Na₂O, all calculated on dry basis (600°C, 1 hour).

[0012] The catalyst may additionally comprise a matrix material. Examples of suitable matrix materials are alumina, silica, titania, zirconia, clays, and mixtures thereof. Matrix materials comprising alumina are generally preferred. In one embodiment, the catalyst comprises 2 wt% to 98 wt% of the solid acid and 98 wt% to 2 wt% of the matrix material, based on the total weight of the solid acid and the matrix material present in the catalyst. In another embodiment, the catalyst comprises 10 wt% to 90 wt% of the solid acid and 90 wt% to 10 wt% of the matrix material, based on the total weight of the solid acid and the matrix material contained in the catalyst. In another embodiment, the catalyst comprises 10 wt%

to 80 wt% of matrix material and balance solid acid. In yet another embodiment, the catalyst comprises 10 wt% to 40 wt% of the matrix material and balance solid acid, based on the total weight of the solid acid and the matrix material contained in the catalyst.

[0013] The catalyst preferably contains no halogen component.

[0014] In one embodiment, the catalyst comprises catalyst particles wherein the ratio between (i) the volume in catalyst pores with a diameter of 40 to 8,000 nm (herein defined as "macropores") and (ii) the specific length of the catalyst particles is in the range of 0.01 to 0.90 ml/(g*mm), and wherein the catalyst has a total pore volume of at least 0.20 ml/g.

[0015] The specific length of a catalyst particle is defined as the ratio between the geometric volume and the geometric surface of the solid part of this catalyst particle. The determination of the geometric volume and the geometric surface is known to the person skilled in the art and can be carried out, e.g., as described in DE 2354558.

[0016] The macropore volume as well as the total pore volume is determined via mercury intrusion on the basis of the Washburn equation covering pores with a diameter of 3.6-8,000 nm.

[0017] In one embodiment, the ratio between the volume in macropores and the specific length is above 0.20 ml/(g*mm), and in another above 0.30 ml/(g*mm). In yet another embodiment, the ratio is above 0.40 ml/(g*mm), but below 0.80 ml/(g*mm).

[0018] In one embodiment, the catalyst has a total pore volume of at least 0.23 ml/g and in another at least 0.25 ml/g.

[0019] In one embodiment, the catalyst particles have a specific length of at least 0.10 mm, in another at least 0.16 mm, and in yet another at least 0.20 mm. In one embodiment, the upper limit of the specific length lies at 2.0 mm, in another at 1.0 mm, and in yet another at 0.6 mm.

[0020] The pore volume in macropores in one embodiment of the catalyst is at least 0.05 ml/g, in another at least 0.08 ml/g. In one embodiment, the upper limit of the pore volume in macropores is below 0.30 ml/g, in another below 0.25 ml/g.

[0021] The particles of the catalyst can have many different shapes, including spheres, cylinders, rings, and symmetric or asymmetric polylobes, for instance tri- and quadrulobes.

[0022] In one embodiment, the catalyst particles have an average particle diameter of at least 0.5 mm, in another embodiment at least 0.8 mm, and in yet another embodiment at least 1.0 mm. In one embodiment, the upper limit of the average particle diameter lies at 10.0 mm, in another at 5.0 mm, and in yet another embodiment at 3.0 mm.

[0023] The catalyst used in the process according to the invention is prepared by adjusting the water content. For example, the solid acid constituent may be mixed with a matrix material, to form carrier particles, followed by calcination of the particles. The hydrogenating function may, e.g., be incorporated into the catalyst composition by impregnating the carrier particles with a solution of a hydrogenation metal component. After impregnation the catalyst may be calcined.

[0024] In one embodiment, the catalyst is reduced at a temperature in the range of 200 to 500°C in a reducing gas such as hydrogen. In another embodiment, the catalyst is reduced at a temperature in the range of 250 to 350°C. The reduction can be performed before adjustment of the water content, after addition of water to the catalyst and/or by using reduction as a way to adjust the water content. In one embodiment, the reduction is performed before adjustment of the water content. In another, the reduction is performed after drying the catalyst in a dry, non-reducing gas (such as nitrogen, helium, air, and the like).

[0025] The water content of the catalyst can be adjusted by various methods as described in PCT/EP2005/000929. Such methods are exemplified below as methods 1, 2, and 3.

Method 1 involves increasing the LOI of a catalyst by exposing the catalyst to water. This can be achieved by exposing the catalyst to a water-containing atmosphere, e.g., air at ambient conditions. Embodiments of this method include exposing a reduced catalyst to water until the desired LOI is reached, exposing an unreduced catalyst to water until an LOI above the desired level is reached, followed by reduction of the catalyst, thereby decreasing the LOI to the desired level, exposing a reduced catalyst to water until an LOT above the desired level is reached, followed by treatment of the catalyst in either an inert or a reducing atmosphere, thereby decreasing the LOI to the desired level, and reducing the catalyst in a hydrogen and water-containing atmosphere.

Method 2 involves decreasing the LOI of an existing catalyst to the desired level by reducing an unreduced catalyst with an LOI above the desired level.

Method 3 involves in-situ water addition by starting the alkylation process with a catalyst having an LOI below the desired level and adding water to the alkylation unit during processing, for instance by adding water to the hydrocarbon feed, by degenerating the catalyst in a water-containing atmosphere and/or by exposing the regenerated catalyst to a water-containing atmosphere.

[0026] A combination of two or more of the above methods may also be employed.

[0027] The hydrocarbon to be alkylate in the alkylation process is isobutane. The alkylation agent is an olefin or mixture of olefins having 2- 6 carbon atoms. In one embodiment, the alkylation process consists of the alkylation of isobutane

with butenes.

[0028] As will be evident to the skilled person, the alkylation process can take any suitable form, including fluidized bed processes, slurry processes, and fixed bed processes. The process can be carried out in a number of beds and/or reactors, each with separate addition of alkylation agent if desirable. In such a case, the process of the invention can be carried out in each separate bed or reactor.

[0029] As mentioned above, water may be added during the process in order to increase the LOI of the catalyst to the desired level. This water can be introduced during the alkylation reaction via, e.g., the hydrocarbon feed or the feed of alkylation agent. Alternatively, the catalyst can be hydrated by using a water-containing atmosphere during the optional (mild) regeneration steps described below, or by contacting the catalyst with water in a separate intermediate hydration step. Similar procedures can be applied to rehydrate the catalyst after its LOI has decreased during processing (i.e. during the alkylation reaction and/or regeneration).

[0030] Suitable process conditions are known to the skilled person. Preferably, an alkylation process as disclosed in WO 98/23560 is applied. The process conditions applied in the present process are summarized in the following Table:

	Temperature range [°C]	Pressure range [bar]	Molar ratio of hydrocarbon to alkylation agent
Preferred	-40 - 250	1 - 100	5:1 - 5,000:1
More preferred	20 - 150	5 - 40	50:1 - 1,000:1
Most preferred	65 - 95	15 - 30	150:1 - 750:1

[0031] Optionally, the catalyst may be subjected to high-temperature regeneration with hydrogen in the gas phase. This high-temperature regeneration may be carried out at a temperature of at least about 150°C, in one embodiment regeneration is carried out at about 150° to about 600°C, and another at about 200° to about 400°C. For details of this regeneration procedure, reference is made to WO 98/23560, and in particular to page 4, lines 12-19. The high-temperature regeneration can be applied periodically during the alkylation process. If as a result of high-temperature regeneration the water content of the catalyst has decreased to below the desired level, the catalyst may be rehydrated during the process in the ways described above.

[0032] In addition to the high-temperature regeneration treatment, a milder regeneration may be applied during the alkylation process, such as described in WO 98/23560, in particular page 9, line 13 through page 13, line 2.

[0033] During the alkylation process, the catalyst is subjected intermittently to a regeneration step by being contacted with a feed containing a hydrocarbon and hydrogen, with said regeneration being carried out at 90% or less of the active cycle of the catalyst, at 60% or less in another embodiment, at 20% or less in yet another embodiment, and at 10% or less in another embodiment. The active cycle of the catalyst is defined herein as the time from the start of the feeding of the alkylation agent to the moment when, in comparison with the alkylation agent added to the catalyst-containing reactor section, 20% of the alkylation agent leaves the catalyst-containing reactor section without being converted, not counting isomerization inside the molecule.

[0034] The preparation of a catalyst for the present invention comprises the steps of: a) calcining solid acid-containing particles at a temperature in the range of 400 to 500°C; b) incorporating platinum and/or palladium into the calcined particles to form noble metal-containing particles; and c) calcining the noble metal-containing particles at a temperature in the range of 350 to 500°C.

[0035] Performance in alkylation reactions of catalysts of the present invention can be further improved if the calcination steps before and after incorporation of the hydrogenation component are both conducted in a specific temperature window.

[0036] The solid acid-containing particles are calcined in step a) at a temperature in the range of 400 to 500°C, in another embodiment in the range of 450 to 500°C, and in yet another embodiment in the range of 460 to 500°C. The heating rate ranges from 0.1 to 100°C/min, and in one embodiment from 0.5°C to 50°C/min, and in another embodiment from 1 to 30°C/min. Calcination is conducted for 0.01 to 10 hrs, and in one embodiment for 0.1 to 5 hrs, and in another embodiment for 0.5 to 2 hrs. It may be conducted in an air and/or inert gas (e.g. nitrogen) flow. In one embodiment this gas flow is dry.

[0037] In another embodiment, the solid acid-containing particles are dried before being calcined. This drying may be conducted at a temperature of 110 to 150°C.

[0038] The calcination can be performed in any equipment, such as a fixed bed reactor, a fluidized bed calciner, and a rotating tube calciner.

[0039] Platinum and/or palladium metal is then incorporated into the calcined solid acid-containing particles in step b). In one embodiment, this is preformed by impregnation or competitive ion exchange of the solid acid-containing particles using a solution comprising said noble metal ions and/or their complexes and (optionally) NH₄⁺ ions.

[0040] Said noble metals are platinum, palladium, and combinations thereof. In another embodiment, at least one of the noble metals is platinum. Suitable noble metal salts include nitrates, chlorides, and ammonium nitrates of the noble metals or their complexes (e.g. NH_3 complexes).

[0041] The resulting noble metal-containing particles are then calcined at a temperature in the range of 350-500°C in step c). In one embodiment, the particles are calcined at 400 to 500°C, and in another from 450 to 500°C. This temperature may be reached by heating the particles by 0.1 to 100°C/min to the desired final value between 350 and 500°C. In one embodiment, they are heated by 0.5 to 50°C/min, in another by 1 to 30°C/min. Calcination may be conducted for 0.01 to 10 hrs, and in one embodiment for 0.1 to 5 hrs, and in another for 0.5 to 2 hrs. Calcination may be conducted in an air and/or inert gas (e.g. nitrogen) flow. In one embodiment this gas flow is dry.

[0042] Optionally, a separate drying step is applied between steps (b) and (c). Alternatively, the noble metal-containing particles are dried during the calcination step. Also optionally, a dwell of 15-120 minutes is introduced at a temperature of 200 to 250°C.

[0043] After calcination step (c), the resulting catalyst particles may be reduced at a temperature range of 200 to 500°C, in one embodiment from 250 to 350°C, in a reducing gas such as hydrogen.

EXAMPLES

Performance of catalyst comprising rare earth ions compared to reference catalyst without rare earth (RE) ions:

[0044] The reference standard Y-zeolite without rare earth ions was prepared via a conventional route, i.e., sodium-Y-zeolite (NaY) was prepared (SAR 5.5) followed by ion exchange with NH_4^+ -ions (remaining Na_2O typically about 4 wt%), steaming at about 575 to about 625°C resulting in an a_0 of about 24.53-24.57Å, a second ion exchange with NH_4^+ -ions (remaining Na_2O typically 1 wt%), further steaming at about 500 to about 550°C resulting in an a_0 of about 24.44-24.52 Å, acid leaching with either H_2SO_4 or HCl at a temperature of about 80°C to increase the bulk-SAR (SAR is defined as the ratio of SiO_2 and Al_2O_3 (mol/mol) present in the zeolite material) from about 6 to about 12 (Na_2O typically drops to about 0.2 wt%), and drying.

[0045] Zeolite of the invention are prepared according to similar procedures using the same starting materials, however NH_4^+ - as well as rare earth ions are used in the first exchange step and the steaming temperature is 400 to 500°C. At this low steaming temperature, less non-framework alumina is formed and acid leaching is not required. So after the first steam treatment, only exchange with NH_4^+ -ions is required and then the zeolite is dried. However, multiple steaming and ion exchange with NH_4^+ -ions steps may be employed if required to achieve appropriate a_0 and Na_2O content. In one embodiment, Na_2O ranges from 0.2 to 1 wt%, a_0 ranges from 24.58-24.68 angstrom and rare earth ranges from 2 to 9 wt %.

[0046] In another embodiment, Na_2O content is lower than 0.7 wt%, a_0 ranges from 24.60-24.66 angstrom and rare earth ranges from 4 to 6 wt %.

[0047] Also a zeolite with about 15 wt% rare earth was prepared. In this case the procedure of the invention was followed but no NH_4^+ ions were added in the first exchange step.

[0048] The tested alkylation catalysts had the following compositions and properties: from 60 to 80% of the above-described zeolite, from 20 to 40% alumina, from 0.15 to 0.5% platinum, the average particle length ranges from 2 to 6 mm, the average length/diameter ratio ranges from 1 to 7.5, the particle diameter ranges from 0.5 to 3 mm, and the side crush strength ranges from 1.5 to 10 lbs/mm.

General Test Procedure:

[0049] A fixed-bed recycle reactor as described in WO 9823560, which is herein incorporated by reference in its entirety, having a diameter of 2 cm was filled with a 1:1 volume/volume mixture of 38.6 grams of catalyst extrudates (on dry basis, i.e. the actual weight corrected for the water content) and carborundum particles (60 mesh). At the center of the reactor tube a thermocouple of 6 mm in diameter was arranged. The reactor was flushed with dry nitrogen for 30 minutes (21 N1/hour). Next, the system was tested for leakages at elevated pressure, after which the pressure was set to 21 bar and the nitrogen flow to 21 N1 /hour. The reactor temperature was then raised to 275°C at a rate of 1°C/min, at 275°C nitrogen was replaced by dry hydrogen and the catalyst was reduced at 275°C.

[0050] Alternatively, in case of high temperature regeneration of the same catalyst sample between runs, after draining and flushing the reactor with hydrogen to remove hydrocarbons while maintaining the alkylation reaction temperature, hydrogen flow was set to 21 N1/hour and the reactor temperature was then raised to 275°C at a rate of 1°C/min, and the catalyst was regenerated at 275°C.

[0051] After 2 hours, the reactor temperature was lowered to the reaction temperature of about 75°C. During cooling down water was added to the hydrogen flow to obtain an LOI of the catalyst of about 2-4 wt% (the LOI of the catalyst is defined as the catalyst's weight loss after heating for two hours at 600°C).

[0052] The hydrogen stream was stopped with the attaining of the reaction temperature. Isobutane containing about 2.5-3 wt% alkylate (added to accelerate deactivation rate, composition of the alkylate added is similar to alkylate produced by the process at the conditions described) and about 1 mol% of dissolved hydrogen was supplied to the reactor at a rate of about 4.0 kg/hour. About 95-98% of the isobutane/alkylate mixture was fed back to the reactor. About 2-5% was drained off for analysis. Such an amount of isobutane/alkylate mixture was supplied to the reactor as to ensure a constant quantity of liquid in the system. When the system had stabilized, hydrogen addition was stopped and such an amount of cis-2-butene was added to it as to give a cis-2-butene-WHSV of 0.16 or higher. The overall rate of flow of liquid in the system was maintained at about 4.0 kg/h. The weight ratio of isobutane to cis-2-butene at the reactor inlet was about 500 - 650. The pressure in the reactor amounted to about 21 bar. Total alkylate concentration of the hydrocarbon recycle flow (from added and produced alkylate) was maintained at about 6.5 -7.5 wt% during the test by controlling the drain off flow to analyses.

[0053] Each time after 1 hour of reaction, the catalyst was regenerated by being washed with isobutane/alkylate mixture for 5 minutes, followed by 50 minutes of regeneration through being contacted with a solution of 1 mole% of H₂ in isobutane/alkylate mixture, and then being washed with isobutane/alkylate mixture for another 5 minutes (total washing and regeneration time 1 hour). After this washing step, alkylation was started again.

[0054] The temperature during the washing steps, the regeneration step, and the reaction step was the same.

[0055] The process was conducted as above and the catalytic performance was measured as a function of time.

[0056] The performance was characterized by the olefin conversion per reactor pass and the research octane number (RON). The RON was determined as described on pages 13 and 14 of WO 9823560, the only exception being that the RON contribution of total C₉+ (excl. 2,2,5-trimethylhexane) was estimated to be 84 instead of 90.

[0057] Olefin conversion per reactor pass is the weight fraction (as a percentage) of olefins that is converted between the inlet - and the outlet of the catalyst bed, not counting isomerization within the olefin molecules.

[0058] Figures 1, 2 and 3 shows the results of accelerated deactivation tests of the catalysts.

[0059] Figure 1 shows that at the same test conditions (whsv = 0.16) the new catalysts (with various amounts of RE on zeolite) maintains a higher conversion level of olefins than the reference (no RE) catalyst. Figure 2 shows that the new catalysts permits a higher whsv (0.20) with similar deactivation rates compared to the reference catalyst. Thus, the new catalysts can be operated at higher whsv and accordingly less catalyst is required to produce a fixed quantity of alkylate, i.e., less catalyst is required at constant capacity of an alkylation plant. Figure 3 shows that the new catalysts produce alkylate with at least the same RON as is obtained with the reference catalyst. The catalyst with about 5 wt% RE on zeolite combines the highest stability with a relatively high RON of alkylate produced.

Claims

1. A process for alkylating hydrocarbons wherein isobutane is reacted with an alkylation agent selected from C₂-C₆ alkenes to form an alkylate in the presence of a catalyst comprising a hydrogenation metal and a rare earth containing solid acid constituent with the catalyst being subjected intermittently to a regeneration step by being contacted with a feed containing a saturated hydrocarbon and hydrogen, said regeneration being carried out at 90% or less of the active cycle of the catalyst, with the active cycle of the catalyst being defined as the time from the start of the feeding of the alkylation agent to the moment when, in comparison with the entrance of the catalyst-containing reactor section, 20% of the alkylation agent leaves the catalyst-containing reactor section without being converted, not counting isomerization inside the molecule, wherein said catalyst is regenerated before there is any substantial decrease of activity of said catalyst, wherein the hydrogenation metal is selected from platinum, palladium and mixtures thereof,

wherein the solid acid constituent is a Y-zeolite prepared by a process comprising the steps of: preparing a sodium zeolite, ion exchanging the sodium zeolite with rare earth ions and optionally NH₄⁺ ions to reduce Na₂O to about 3 - 6 wt%, steaming the zeolite at 400 to 500°C such that a₀ ranges from 24.56 to 24.72 Å, ion exchanging with NH₄⁺ ions to reduce Na₂O to below 1.5 wt%, and drying,

and wherein the water content of the catalyst ranges from 1.5 wt.-% to 6 wt.-%, measured by determining the weight loss upon heating the catalyst for two hours at 600 °C (loss on Ignition LOI600).

2. The process of claim 1 wherein the Y-zeolite is prepared by the following process:

a process comprising the steps of preparing a sodium zeolite, ion exchanging the sodium zeolite with rare earth ions and optionally NH₄⁺ ions to reduce Na₂O to 3.5 - 4.5 wt%, steaming the zeolite at 400 to 500°C such that a₀ ranges from 24.58 to 24.68 Å, ion exchanging with NH₄⁺ ions to reduce Na₂O to below 1.0 wt%, and drying; preferably by the following process:

a process comprising the steps of: preparing a sodium zeolite, ion exchanging the sodium zeolite with rare earth ions and optionally NH_4^+ ions to reduce Na_2O to 3.5 - 4.5 wt%, steaming the zeolite at 400 to 500°C such that a_0 ranges from 24.60 to 24.66 Å, ion exchanging with NH_4^+ ions to reduce Na_2O to below 0.7 wt%, and drying.

3. The process of any one of claims 1 or 2 wherein the alkylation agent comprises C3-C5 alkenes, preferably wherein the alkylation agent is butene or a mixture of butenes.
4. The process of claim 1 or 2 wherein the regeneration is carried out at i) 60% or less of the active cycle of the catalyst or ii) 20% or less of the active cycle of the catalyst.
5. The process of claim 1 or 2 wherein the catalyst has a particle diameter ranging from 0.75 to 2 mm.
6. The process of claim 1 or 2 wherein the catalyst comprises a hydrogenation metal on a carrier comprising i) 2-98 wt. % of matrix material, preferably comprising alumina ii) 20-80 wt. % of matrix material or iii) 20-50 wt. % of matrix material and the balance solid acid constituent.
7. The process of claim 1 or 2 wherein the hydrogenation metal is present in an amount of 0.01-2 wt. %, calculated as metal.
8. The process of claim 1 or 2, wherein the rare earth is lanthanum.
9. The process of any one of claims 1 to 8, wherein the solid acid component comprises rare earth in the range from 2 to 9 wt. %.
10. The process of claim 1 or 2, wherein the saturated hydrocarbon employed in the regeneration is isobutane, wherein the regeneration temperature and/or the regeneration pressure do not differ by i) more than 50% from the reaction temperature, expressed in Celsius, and the reaction pressure, respectively; ii) by more than 20% from the reaction temperature, expressed in Celsius, and the reaction pressure, respectively; or iii) the regeneration is carried out at substantially the same temperature and/or pressure as the reaction.
11. The process of claim 1 or 2 wherein the length of the regeneration step is 0.1 to 10 times as long as the length of the reaction step, preferably wherein the length of the regeneration step is 0.5-2 times as long as the length of the reaction step.
12. The process of claim 1 or 2 wherein the regeneration step, is preceded by a washing step with a saturated hydrocarbon essentially in the absence of hydrogen and alkylation agent, is followed by a washing step with a saturated hydrocarbon essentially in the absence of hydrogen and alkylation agent, or both.
13. The process of claim 1 or 2 wherein the catalyst is periodically subjected to a high temperature regeneration with hydrogen in the gas phase at 150-400°C.
14. The process of claim 13 wherein the catalyst is subjected to a high temperature regeneration with hydrogen in the gas phase after every 50 regenerations with saturated hydrocarbon and hydrogen.
15. The process of claim 1 or 2 wherein the catalyst is prepared by a) calcining Y-zeolite containing particles at a temperature in the range of 460-500 °C, b) incorporating a platinum and/or palladium into the calcined particles to form noble metal-containing particles, and c) calcining the noble metal-containing particles at a temperature in the range of 450-500 °C, wherein the Group VIII noble metal is selected from platinum, palladium and mixtures thereof.
16. The process according to claim 1 or 2 wherein:

the catalyst comprises from 1.8 to 4 wt%, preferably from 2 to 3 wt% of water, measured as the loss on ignition at 600°C.
17. The process according to claim 16, wherein the catalyst is prepared by adding water to a dry catalyst comprising solid acid and hydrogenation metal before use in the alkylation process.

Patentansprüche

1. Verfahren zum Alkylieren von Kohlenwasserstoffen, wobei Isobutan mit einem Alkylierungsmittel, ausgewählt aus C2-C6-Alkenen, umgesetzt wird, um ein Alkylat zu bilden, in der Gegenwart eines Katalysators, umfassend ein Hydrierungsmetall und einen festen, Seltene Erden enthaltenden Säurebestandteil, wobei der Katalysator intermittierend einem Regenerierungsschritt ausgesetzt wird, wobei mit einer Zufuhr, enthaltend einen gesättigten Kohlenwasserstoff und Wasserstoff, in Kontakt gebracht wird, wobei die Regenerierung bei 90 % oder weniger des aktiven Zyklus des Katalysators durchgeführt wird, wobei der aktive Zyklus des Katalysators als die Zeit vom Beginn der Zuführung des Alkylierungsmittels bis zu dem Moment, wenn, verglichen mit dem Eingang des katalysatorenhaltenden Reaktorabschnitts, 20 % des Alkylierungsmittels den katalysatorenhaltenden Reaktorabschnitt verlassen, ohne umgewandelt zu sein, wobei Isomerisierung in dem Molekül nicht berücksichtigt wird, definiert ist, wobei der Katalysator regeneriert wird, bevor jedwede wesentliche Abnahme an Aktivität des Katalysators auftritt, wobei das Hydrierungsmetall aus Platin, Palladium und Gemischen davon ausgewählt ist, wobei der feste Säurebestandteil ein Y-Zeolith ist, der hergestellt wird durch ein Verfahren, umfassend die Schritte von: Herstellen eines Natrium-Zeolithen, Ionenaustauschen des Natrium-Zeolithen mit Seltene-Erden-Ionen und gegebenenfalls NH_4^+ -Ionen, um Na_2O auf etwa 3 - 6 Gew.-% zu verringern, Dampfbehandeln des Zeolithen bei 400 bis 500°C, so dass a_0 in einem Bereich von 24,56 bis 24,72 Å liegt, Ionenaustauschen mit NH_4^+ -Ionen, um Na_2O auf unter 1,5 Gew.-% zu verringern, und Trocknen, und wobei der Wassergehalt des Katalysators in einem Bereich von 1,5 Gew.-% bis 6 Gew.-% liegt, gemessen durch Bestimmen des Gewichtsverlusts nach Erwärmen des Katalysators zwei Stunden lang bei 600°C (Glühverlust LOI600).
2. Verfahren nach Anspruch 1, wobei der Y-Zeolith durch das folgende Verfahren hergestellt wird:
 - ein Verfahren, umfassend die Schritte von: Herstellen eines Natrium-Zeolithen, Ionenaustauschen des Natrium-Zeolithen mit Seltene-Erden-Ionen und gegebenenfalls NH_4^+ -Ionen, um Na_2O auf 3,5 - 4,5 Gew.-% zu verringern, Dampfbehandeln des Zeolithen bei 400 bis 500°C, so dass a_0 in einem Bereich von 24,58 bis 24,68 Å liegt, Ionenaustauschen mit NH_4^+ -Ionen, um Na_2O auf unter 1,0 Gew.-% zu verringern, und Trocknen; bevorzugt durch das folgende Verfahren:
 - ein Verfahren, umfassend die Schritte von: Herstellen eines Natrium-Zeolithen, Ionenaustauschen des Natrium-Zeolithen mit Seltene-Erden-Ionen und gegebenenfalls NH_4^+ -Ionen, um Na_2O auf 3,5 - 4,5 Gew.-% zu verringern, Dampfbehandeln des Zeolithen bei 400 bis 500°C, so dass a_0 in einem Bereich von 24,60 bis 24,66 Å liegt, Ionenaustauschen mit NH_4^+ -Ionen, um Na_2O auf unter 0,7 Gew.-% zu verringern, und Trocknen.
3. Verfahren nach einem der Ansprüche 1 oder 2, wobei das Alkylierungsmittel C3-C5-Alkene umfasst, wobei das Alkylierungsmittel bevorzugt Buten oder ein Gemisch von Butenen ist.
4. Verfahren nach Anspruch 1 oder 2, wobei die Regenerierung bei i) 60 % oder weniger des aktiven Zyklus des Katalysators oder ii) 20 % oder weniger des aktiven Zyklus des Katalysators durchgeführt wird.
5. Verfahren nach Anspruch 1 oder 2, wobei der Katalysator einen Teilchendurchmesser im Bereich von 0,75 bis 2 mm aufweist.
6. Verfahren nach Anspruch 1 oder 2, wobei der Katalysator ein Hydrierungsmetall auf einem Träger, umfassend i) 2-98 Gew.-% Matrixmaterial, bevorzugt umfassend Aluminium, ii) 20-80 Gew.-% Matrixmaterial oder iii) 20-50 Gew.-% Matrixmaterial, wobei der Rest fester Säurebestandteil ist, umfasst.
7. Verfahren nach Anspruch 1 oder 2, wobei das Hydrierungsmetall in einer Menge von 0,01-2 Gew.-%, berechnet als Metall, vorhanden ist.
8. Verfahren nach Anspruch 1 oder 2, wobei die Seltene Erde Lanthan ist.
9. Verfahren nach einem der Ansprüche 1 bis 8, wobei der feste Säurebestandteil Seltene Erden im Bereich von 2 bis 9 Gew.-% umfasst.
10. Verfahren nach Anspruch 1 oder 2, wobei der in der Regenerierung verwendete, gesättigte Kohlenwasserstoff

Isobutan ist, wobei die Regenerierungstemperatur und/oder der Regenerierungsdruck nicht um i) mehr als 50 % von der Reaktionstemperatur, ausgedrückt in Celsius, beziehungsweise dem Reaktionsdruck; ii) mehr als 20 % von der Reaktionstemperatur, ausgedrückt in Celsius, beziehungsweise dem Reaktionsdruck abweichen; oder iii) die Regenerierung bei im Wesentlichen der gleichen Temperatur und/oder dem gleichen Druck wie die Umsetzung durchgeführt wird.

11. Verfahren nach Anspruch 1 oder 2, wobei die Dauer des Regenerierungsschritts 0,1-bis 10-mal so lang wie die Dauer des Reaktionsschritts ist, wobei die Länge des Regenerierungsschritts bevorzugt 0,5-2-mal so lang wie die Dauer des Reaktionsschritts ist.

12. Verfahren nach Anspruch 1 oder 2, wobei dem Regenerierungsschritt ein Waschschrift mit einem gesättigten Kohlenwasserstoff im Wesentlichen in der Abwesenheit von Wasserstoff und Alkylierungsmittel vorausgeht, ein Waschschrift mit einem gesättigten Kohlenwasserstoff im Wesentlichen in der Abwesenheit von Wasserstoff und Alkylierungsmittel nachfolgt, oder beides.

13. Verfahren nach Anspruch 1 oder 2, wobei der Katalysator periodisch einer Hochtemperaturregenerierung mit Wasserstoff in der Gasphase bei 150-400°C ausgesetzt wird.

14. Verfahren nach Anspruch 13, wobei der Katalysator einer Hochtemperaturregenerierung mit Wasserstoff in der Gasphase nach jeweils 50 Regenerierungen mit gesättigtem Kohlenwasserstoff und Wasserstoff ausgesetzt wird.

15. Verfahren nach Anspruch 1 oder 2, wobei der Katalysator hergestellt wird durch a) Kalzinieren von Y-Zeolith-haltigen Teilchen bei einer Temperatur im Bereich von 460-500°C, b) Einbringen eines Platins und/oder Palladiums in die kalzinieren Teilchen, um edelmetallhaltige Teilchen zu bilden, und c) Kalzinieren der edelmetallhaltigen Teilchen bei einer Temperatur im Bereich von 450-500°C, wobei das Gruppe VIII-Edelmetall aus Platin, Palladium und Gemischen davon ausgewählt ist.

16. Verfahren gemäß Anspruch 1 oder 2, wobei:

der Katalysator 1,8 bis 4 Gew.-%, bevorzugt 2 bis 3 Gew.-% Wasser, gemessen als der Glühverlust bei 600°C, umfasst.

17. Verfahren gemäß Anspruch 16, wobei der Katalysator durch Geben von Wasser zu einem trockenen Katalysator, umfassend feste Säure und Hydrierungsmetall, vor einer Verwendung in dem Alkylierungsverfahren hergestellt wird.

Revendications

1. Procédé d'alkylation d'hydrocarbures, dans lequel de l'isobutane réagit avec un agent d'alkylation choisi parmi des alcènes en C2 à C6 pour former un alkylate en présence d'un catalyseur comportant un métal d'hydrogénation et un constituant acide solide contenant un élément des terres rares, le catalyseur étant soumis par intermittence à une étape de régénération en étant mis en contact avec une charge contenant un hydrocarbure saturé et de l'hydrogène, ladite régénération étant mise en oeuvre à 90 % ou moins du cycle actif du catalyseur, le cycle actif du catalyseur étant défini comme le temps à partir du début de l'introduction de l'agent d'alkylation jusqu'au moment où, par rapport à l'entrée du tronçon de réacteur contenant le catalyseur, 20 % de l'agent d'alkylation quitte le tronçon de réacteur contenant le catalyseur sans être converti, sans compter l'isomérisation à l'intérieur de la molécule, dans lequel ledit catalyseur est régénéré avant qu'il y ait une quelconque baisse substantielle de l'activité dudit catalyseur, dans lequel le métal d'hydrogénation est choisi parmi le platine, le palladium et des mélanges de ceux-ci, dans lequel le constituant acide solide est une Y-zéolite préparée par un procédé comportant les étapes consistant à : préparer une zéolite de sodium, échanger des ions de la zéolite de sodium avec des ions de terres rares et facultativement des ions NH_4^+ pour réduire Na_2O jusqu'à environ 3 à 6 % en poids, traiter à la vapeur la zéolite à 400 à 500 °C de telle sorte que a_0 varie de 24,56 à 24,72 Å, échanger des ions avec des ions NH_4^+ pour réduire Na_2O jusqu'à moins de 1,5 % en poids, et sécher, et dans lequel la teneur en eau du catalyseur varie de 1,5 % en poids à 6 % en poids, mesurée en déterminant la perte de poids en chauffant le catalyseur pendant deux heures à 600 °C (perte par calcination LOI600).

2. Procédé selon la revendication 1, dans lequel la Y-zéolite est préparée par le procédé suivant :

EP 2 106 394 B1

un procédé comportant les étapes consistant à : préparer une zéolite de sodium, échanger des ions de la zéolite de sodium avec des ions de terres rares et facultativement des ions NH_4^+ pour réduire Na_2O jusqu'à 3,5 à 4,5 % en poids, traiter à la vapeur la zéolite à 400 à 500 °C de telle sorte que a_0 varie de 24,58 à 24,68 Å, échanger des ions avec des ions NH_4^+ pour réduire Na_2O à moins de 1,0 % en poids, et sécher, de préférence par le procédé suivant :

un procédé comportant les étapes consistant à :

préparer une zéolite de sodium, échanger des ions de la zéolite de sodium avec des ions de terres rares et facultativement des ions NH_4^+ pour réduire Na_2O jusqu'à environ 3,5 à 4,5 % en poids, traiter à la vapeur la zéolite à 400 à 500 °C de telle sorte que a_0 varie de 24,60 à 24,66 Å, échanger des ions avec des ions NH_4^+ pour réduire Na_2O à moins de 0,7 % en poids, et sécher.

3. Procédé selon l'une quelconque des revendications 1 ou 2, dans lequel l'agent d'alkylation comporte des alcènes en C3 à C5, de préférence dans lequel l'agent d'alkylation est un butène ou un mélange de butènes.
4. Procédé selon la revendication 1 ou 2, dans lequel la régénération est mise en oeuvre à i) 60 % ou moins du cycle actif du catalyseur ou ii) 20 % ou moins du cycle actif du catalyseur.
5. Procédé selon la revendication 1 ou 2, dans lequel le catalyseur a un diamètre de particules variant de 0,75 à 2 mm.
6. Procédé selon la revendication 1 ou 2, dans lequel le catalyseur comporte un métal d'hydrogénation sur un porteur comportant i) 2 à 98 % en poids de matériau de matrice, de préférence comportant de l'alumine, ii) 20 à 80 % en poids de matériau de matrice ou iii) 20 à 50 % en poids de matériau de matrice et le reste étant le constituant acide solide.
7. Procédé selon la revendication 1 ou 2, dans lequel le métal d'hydrogénation est présent en une quantité de 0,01 à 2 % en poids, calculée comme un métal.
8. Procédé selon la revendication 1 ou 2, dans lequel l'élément des terres rares est le lanthane.
9. Procédé selon l'une quelconque des revendications 1 à 8, dans lequel le composant acide solide comporte un élément des terres rares dans l'intervalle de 2 à 9 % en poids.
10. Procédé selon la revendication 1 ou 2, dans lequel l'hydrocarbure saturé employé dans la régénération est l'isobutane, dans lequel la température de régénération et/ou la pression de régénération ne diffèrent pas i) de plus de 50 % par rapport à la température de réaction, exprimée en Celsius, et à la pression de réaction, respectivement, ii) de plus de 20 % par rapport à la température de réaction, exprimée en Celsius, et à la pression de réaction, respectivement, ou iii) la régénération est mise en oeuvre sensiblement à la même température et/ou à la même pression que la réaction.
11. Procédé selon la revendication 1 ou 2, dans lequel la durée de l'étape de régénération est de 0,1 à 10 fois aussi longue que la durée de l'étape de réaction, de préférence dans lequel la durée de l'étape de régénération est de 0,5 à 2 fois aussi longue que la durée de l'étape de réaction.
12. Procédé selon la revendication 1 ou 2, dans lequel l'étape de régénération est précédée d'une étape de lavage avec un hydrocarbure saturé pratiquement en l'absence d'hydrogène et d'agent d'alkylation, et suivie d'une étape de lavage avec un hydrocarbure saturé pratiquement en l'absence d'hydrogène et d'agent d'alkylation, ou les deux.
13. Procédé selon la revendication 1 ou 2, dans lequel le catalyseur est périodiquement soumis à une régénération à haute température avec l'hydrogène en phase gazeuse à 150 à 400 °C.
14. Procédé selon la revendication 13, dans lequel le catalyseur est soumis à une régénération à haute température avec l'hydrogène en phase gazeuse après chaque 50 régénérations avec l'hydrocarbure saturé et l'hydrogène.
15. Procédé selon la revendication 1 ou 2, dans lequel le catalyseur est préparé a) en calcinant des particules contenant une Y-zéolite à une température dans l'intervalle de 460 à 500 °C, b) en incorporant du platine et/ou du palladium dans les particules calcinées pour former des particules contenant un métal noble, et c) en calcinant les particules

EP 2 106 394 B1

contenant du métal noble à une température dans l'intervalle de 450 à 500 °C, dans lequel le métal noble du groupe VIII est choisi parmi le platine, le palladium et des mélanges de ceux-ci.

16. Procédé selon la revendication 1 ou 2, dans lequel :

5 le catalyseur comporte de 1,8 à 4 % en poids, préférence de 2 à 3 % en poids d'eau, mesurée comme la perte par calcination à 600 °C.

10 17. Procédé selon la revendication 16, dans lequel le catalyseur est préparé en ajoutant de l'eau à un catalyseur sec comportant un acide solide et un métal d'hydrogénation avant utilisation dans le procédé d'alkylation.

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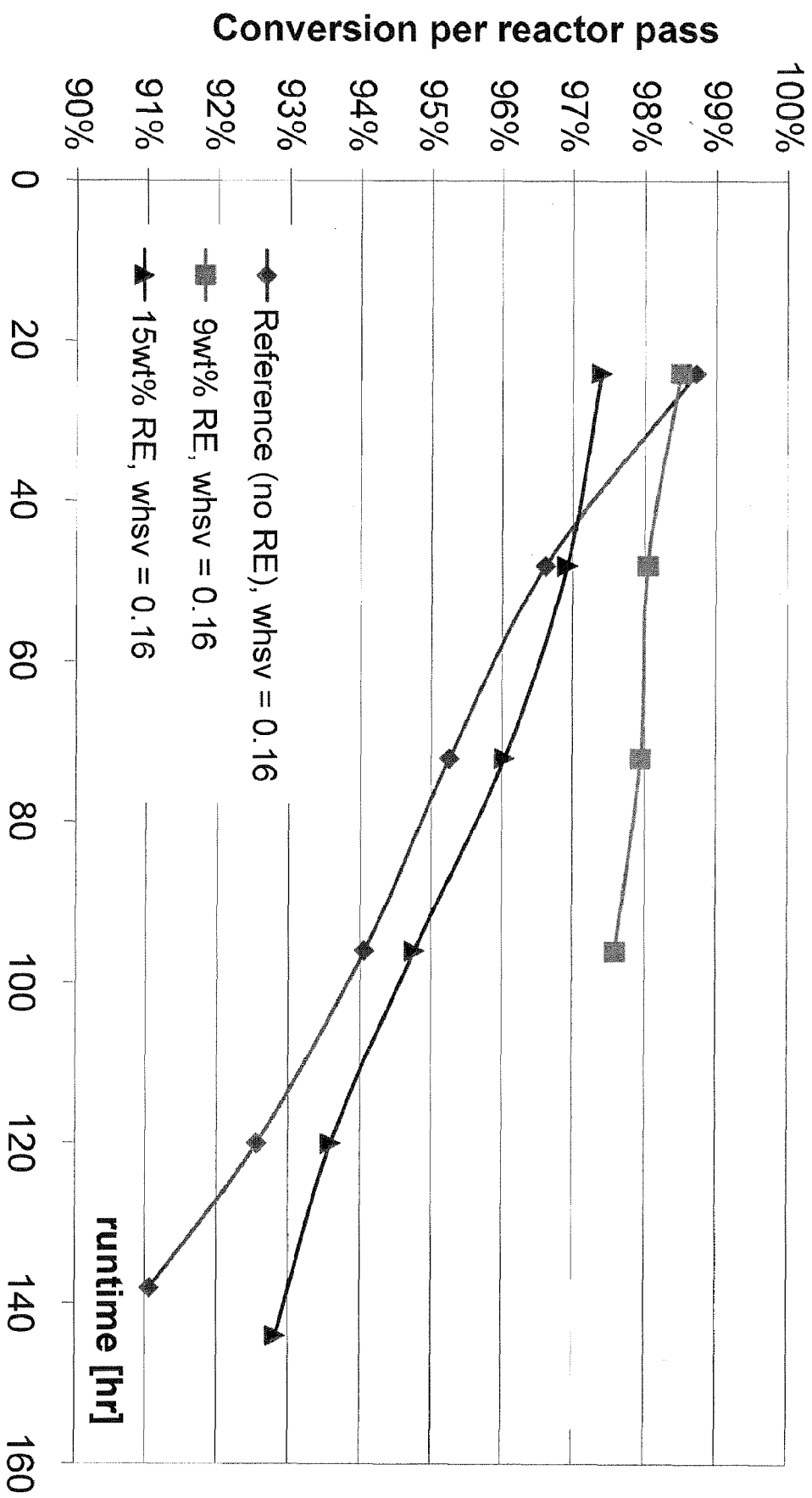
Figure 1 Conversion related to wt% RE on zeolite

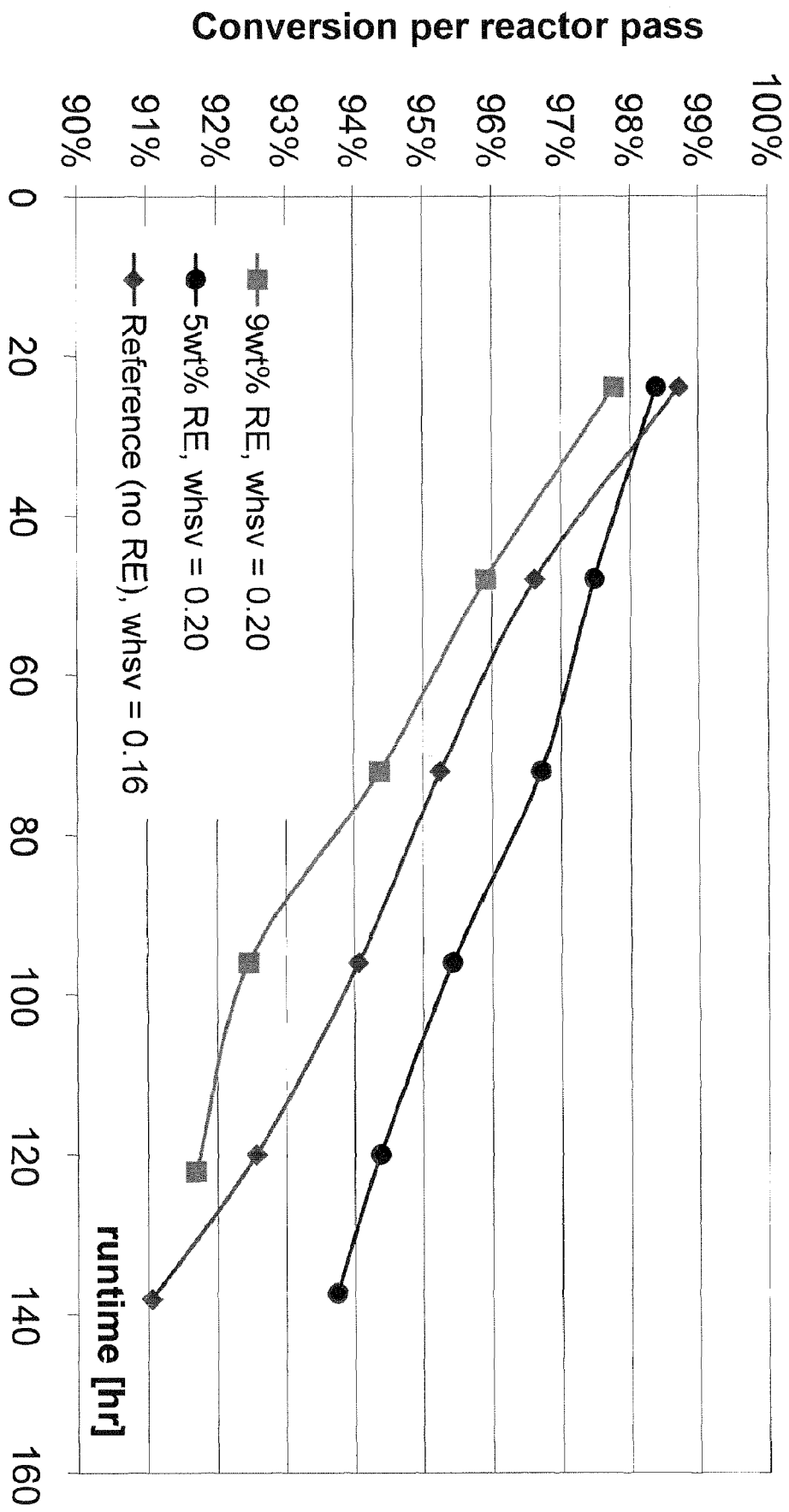
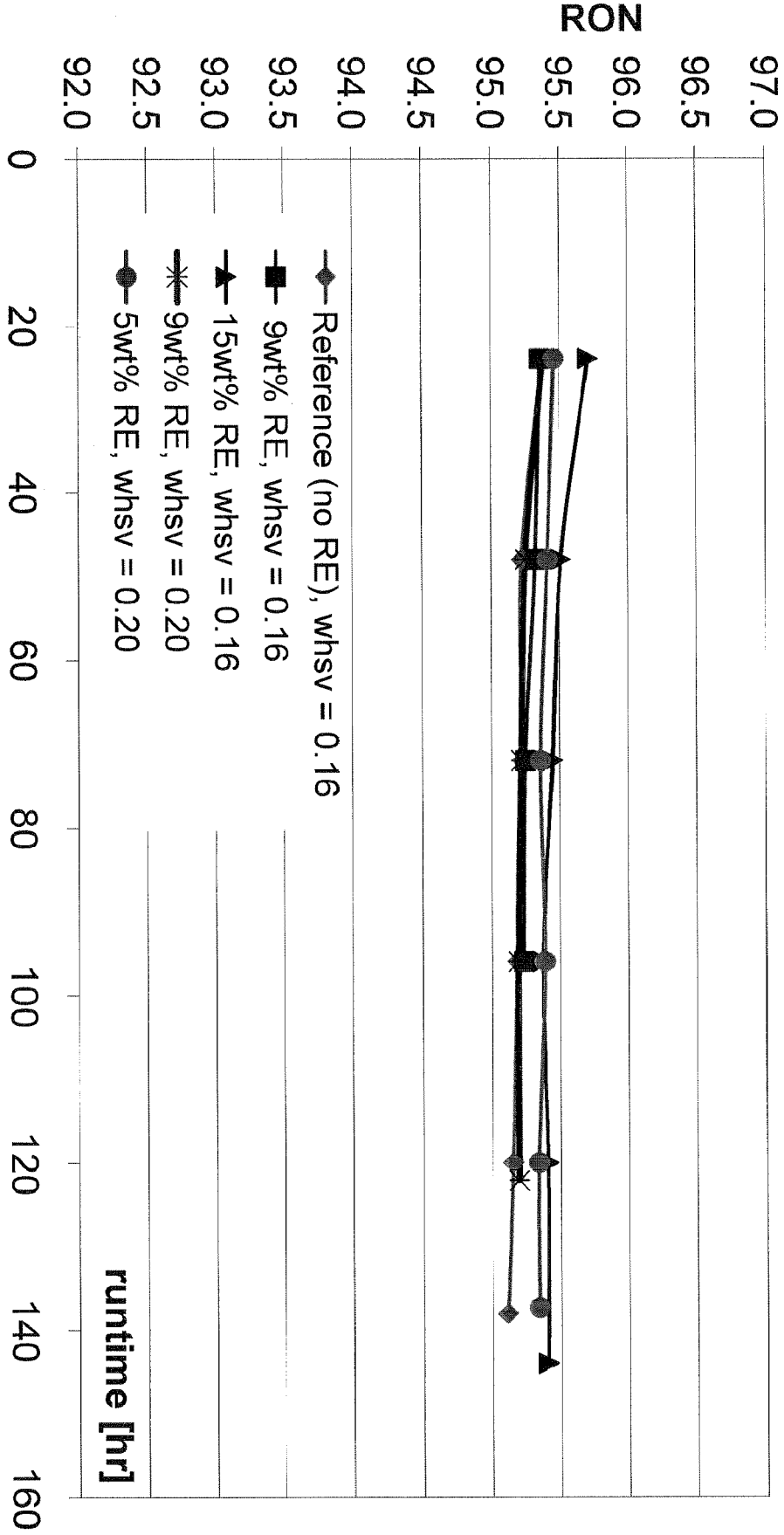
Figure 2 Conversion related to wt% RE on zeolite

Figure 3 RON related to percentage RE on zeolite



REFERENCES CITED IN THE DESCRIPTION

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Alkilezési eljárás ritkaföldfém tartalmú zeoliteket és egy hidrogénező fémet tartalmazó katalizátorral

SZABADALMI IGÉNYPONTOK

1. Eljárás szénhidrogének alkilezésére, melynek során izobutánt egy, a 2-6 szénatomos alkének közül kiválasztott alkilezőszerrel reagáltatunk, alkilát előállítására, hidrogénező fémet és ritkaföldfém tartalmú szilárd sav alkotóelemet tartalmazó katalizátor jelenlétében, a katalizátort időszakosan regenerálási lépésnek vetjük alá, betáplált telített szénhidrogénnel és hidrogénnel való érintkeztetés révén, az említett regenerálást a katalizátor aktív ciklusának 90%-ánál vagy kevesebbnél végezzük, a katalizátor aktív ciklusát úgy definiáljuk, mint az alkilezőszer betáplálásának kezdetétől addig a pillanatig tartó idő, amikor a katalizátor-tartalmú reaktor szekcióba való belépéssel összehasonlítva, az alkilezőszer 20%-a elhagyja a katalizátor tartalmú reaktor szekciót átalakulás nélkül, nem számítva a molekulán belüli izomerizációt, és ahol az említett katalizátort regeneráljuk, mielőtt az említett katalizátor aktivitása számottevő mértékben csökkenne, ahol a hidrogénező fémet a platina, palládium és ezek keverékei közül választjuk ki, ahol a szilárd sav alkotórész egy Y-zeolit, a következő lépéseket tartalmazó eljárással előállítva: nátrium-zeolitot állítunk elő, a nátrium-zeolitot ritkaföldfém ionokkal és adott esetben NH_4^+ ionokkal ioncseréljük az Na_2O mennyiség körülbelül 3-6 tömeg%-ra való csökkentésére, a zeolitot 400-500°C hőmérsékleten gőzöljük, úgy, hogy a_0 értéke 24,56-24,72 Å tartományban legyen, NH_4^+ ionokkal ioncserével a Na_2O tartalmat 1,5 tömeg% alá csökkentjük, és megszáritjuk, és ahol a katalizátor víztartalma 1,5 tömeg% - 6 tömeg% között van, amit a katalizátort tömegvesztésének meghatározásával mérünk 2 óra hosszat 600°C-on végzett hevítés után (gyulladásos veszteség LOI600).
2. Az 1. igénypont szerinti eljárás, ahol az Y-zeolitot az alábbi eljárással állítjuk elő: nátrium-zeolitot állítunk elő, a nátrium-zeolitot ritkaföldfém ionokkal és adott esetben NH_4^+ ionokkal ioncseréljük az Na_2O mennyiség 3,5-4,5 tömeg%-ra való csökkentésére, a zeolitot 400-500°C hőmérsékleten gőzöljük, úgy, hogy a_0 értéke 24,58-24,68 Å tartományban legyen, NH_4^+ ionokkal ioncserével a Na_2O tartalmat 1,0 tömeg% alá csökkentjük, és megszáritjuk;



előnyösen pedig a következő eljárással:

nátrium-zeolitot állítunk elő, a nátrium-zeolitot ritkaföldfém ionokkal és adott esetben NH_4^+ ionokkal ioncseréljük az Na_2O mennyiség 3,5-4,5 tömeg%-ra való csökkentésére, a zeolitot 400-500°C hőmérsékleten gőzöljük, úgy, hogy a₀ értéke 24,60-24,66 Å tartományban legyen, NH_4^+ ionokkal ioncserével a Na_2O tartalmat 0,7 tömeg% alá csökkentjük, és megszáritjuk.

3. Az 1. vagy 2. igénypont szerinti eljárás, ahol az alkilezőszer 3-5 szénatomos alkéneket tartalmaz, előnyösen, ahol az alkilezőszer butén vagy butének elegye.
4. Az 1. vagy 2. igénypont szerinti eljárás, ahol a regenerálást i) a katalizátor aktív ciklusának 60%-ánál vagy kevesebbnél vagy ii) a katalizátor aktív ciklusának 20 %-ánál vagy kevesebbnél végezzük.
5. Az 1. vagy 2. igénypont szerinti eljárás, ahol a katalizátor szemecseátmérője 0,75-2 mm.
6. Az 1. vagy 2. igénypont szerinti eljárás, ahol a katalizátor tartalmaz egy hidrogénező fém hordozón, mely tartalmaz i) 2-98 tömeg%, előnyösen alumíniumoxidot tartalmazó mátrix-anyagot, ii) 20-80 tömeg% mátrix-anyagot vagy iii) 20-50 tömeg% mátrix-anyagot és a maradék szilárd sav alkotórész.
7. Az 1. vagy 2. igénypont szerinti eljárás, ahol a hidrogénező fém 0,01-2 tömeg% mennyiségben van jelen, fémként számolva.
8. Az 1. vagy 2. igénypont szerinti eljárás, ahol a ritkaföldfém lantán.
9. Az 1-8. igénypontok bármelyike szerinti eljárás, ahol a szilárd sav komponens ritkaföldfém 2-9 tömeg% mennyiségben tartalmaz.
10. Az 1. vagy 2. igénypont szerinti eljárás, ahol a regeneráláshoz alkalmazott szénhidrogén izobután, ahol a regenerálási hőmérséklet és/vagy regenerálási nyomás nem különbözik i) több mint 50%-kal a reakció hőmérséklettől, Celsius fokban kifejezve, illetőleg a reakció nyomástól; ii) több mint 20%-kal a reakció hőmérséklettől, Celsius fokban kifejezve, illetőleg a reakció nyomástól; vagy iii) a regenerálást lényegében azonos hőmérsékleten és/vagy nyomáson végezzük, mint a reakciót.
11. Az 1. vagy 2. igénypont szerinti eljárás, ahol a regenerálási eljárás hossza 0,1-10-szerese a reakciólépés hosszának, előnyösen, ahol a regenerálási lépés hossza 0,5-2-szerese a reakciólépés hosszának.
12. Az 1. vagy 2. igénypont szerinti eljárás, ahol a regenerálási lépést egy mosási lépéssel végezzük telített szénhidrogénnel, lényegében hidrogén és alkilezőszer nélkül, melyet egy

telített szénhidrogénnel való mosási lépés követ, lényegében hidrogén és alkilezőszer nélkül, vagy mindkettő.

13. Az 1. vagy 2. igénypont szerinti eljárás, ahol a katalizátort periodikusan magas hőmérsékletű regenerálásnak vetjük alá, hidrogénnel a gáz fázisban, 150-400°C hőmérsékleten.
14. A 13. igénypont szerinti eljárás, ahol a katalizátort magas hőmérsékletű regenerálásnak vetjük alá, hidrogénnel a gáz fázisban, minden 50 telített szénhidrogénnel és hidrogénnel végzett regenerálás után.
15. Az 1. vagy 2. igénypont szerinti eljárás, ahol a katalizátort úgy állítjuk elő, hogy Y-zeolitot tartalmazó szemcséket kalcinálunk 460-500°C hőmérsékleten, b) a kalcinált szemcsékbe nemesfém-tartalmú szemcsék képzésére platinát vagy palládiumot ágyazunk, és c) a nemesfém-tartalmú szemcséket 450-500°C hőmérsékleten kalcináljuk, ahol a VIII csoport-béli nemesfémet a platina, palládium és keverékeik közül választjuk ki.
16. Az 1. vagy 2. igénypont szerinti eljárás, ahol:
a katalizátor 1,8-4 tömeg%, előnyösen 2-3 tömeg% vizet tartalmaz, gyulladáshoz veszteségként mérve 600°C-on.
17. A 16. igénypont szerinti eljárás, ahol a katalizátort úgy készítjük, hogy szilárd savat és hidrogénező fémet tartalmazó száraz katalizátorhoz az alkilezési lépésben való alkalmazás előtt vizet adunk.