

(51) International Patent Classification:
C07C 29/04 (2006.01)(21) International Application Number:
PCT/IN2011/000493(22) International Filing Date:
26 July 2011 (26.07.2011)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
2211/MUM/2010 4 August 2010 (04.08.2010) IN

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Navi Mumbai- 400 709, Maharashtra (IN).(81) Designated States (unless otherwise indicated, for every
kind of national protection available): AE, AG, AL, AM,
AO, AT, AU, AZ, BA, BB, BG, BH, BR, BW, BY, BZ,
CA, CH, CL, CN, CO, CR, CU, CZ, DE, DK, DM, DO,
DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT,
HN, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KN, KP,
KR, KZ, LA, LC, LK, LR, LS, LT, LU, LY, MA, MD,
ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI,
NO, NZ, OM, PE, PG, PH, PL, PT, RO, RS, RU, SC, SD,
SE, SG, SK, SL, SM, ST, SV, SY, TH, TJ, TM, TN, TR,
TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.(84) Designated States (unless otherwise indicated, for every
kind of regional protection available): ARIPO (BW, GH,
GM, KE, LR, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG,
ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ,
TM), European (AL, AT, BE, BG, CH, CY, CZ, DE, DK,
EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU,
LV, MC, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK,
SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ,
GW, ML, MR, NE, SN, TD, TG).**Declarations under Rule 4.17:**

— of inventorship (Rule 4.17(iv))

Published:— without international search report and to be republished
upon receipt of that report (Rule 48.2(g))

(54) Title: METHOD FOR SELECTIVE HYDRATION OF AROMA OLEFINS TO ALCOHOLS IN CONTINUOUS SOLID CATALYST COLUMN REACTOR

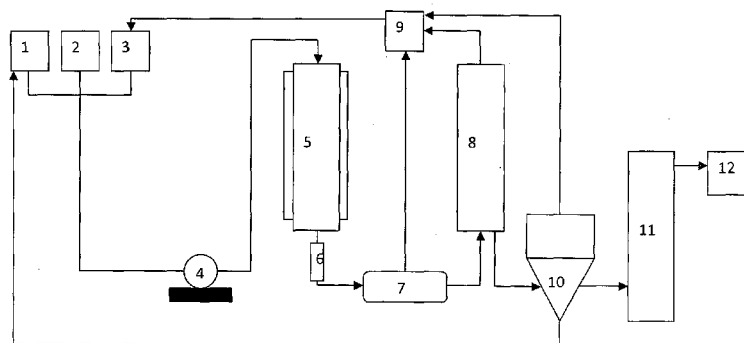


Figure: 2

(57) Abstract: Present invention is related to method for selective hydration of aroma olefins to alcohols in continuous solid catalyst column reactor. The invention discloses the continuous production of unsaturated aroma alcohol and/or ester comprising of C₁₀₋₁₂ carbon chain by selective solid acid catalyzed hydration reaction. According to the process of the invention production of Octa-1-ene-7-ol, 3,7-dimethyl- (Dihydromyrcenol) and/or dihydromyrcenyl acetate can be carried out by passing 1,6-Octadiene, 3,7-dimethyl- (dihydromyrcene) and water continuously through a packed bed column reactor wherein the residence time of column is not more than 90 minutes and reaction temperature not substantially above 100 degree C. The present disclosure solves the problem of effluent treatment and increases the yield of the process due to selectivity in reaction.

“Method for Selective Hydration of Aroma Olefins to Alcohols in Continuous Solid catalyst Column Reactor”

RELATED APPLICATION

The application claims priority from provisional application number: 2211/MUM/2010 entitled
5 “Method for Selective Hydration of Aroma Olefins to Alcohols in Continuous Solid catalyst Column Reactor” and filed on 4th August 2010.

FIELD OF INVENTION

Present invention is related to method for selective hydration of aroma olefins to terpene alcohols in continuous solid catalyst column reactor. The invention discloses the continuous production of
10 unsaturated terpene alcohol and/or ester comprising of C₁₀₋₁₂ carbon chain by selective solid acid catalyzed hydration reaction.

BACKGROUND OF THE INVENTION

Dihydromyrcenol (DHMOL; 2, 6-dimethyl-7-octen-2-ol) is a well known fragrance material with excellent stability, powerful citrus and lime-like odour and is increasingly being used in the fragrance
15 industry.

DHMOL can be produced via the hydration of dihydromyrcene (DHM or 3,7-dimethyl-1,6-octadiene) in the presence of an acidic catalyst using two main methods: indirect hydration and direct
20 hydration.

For indirect hydration different reactants such as carboxylic acid (US Patent No. 2902510, US Patent No. 3487118, US Patent No. 247723) or hydrogen chloride gas (US Patent No. 5105030, US Patent No. 4791222) are used to react with DHM to form intermediate products, which are then hydrolyzed to obtain DHMOL. However, this method suffers from many problems such as lower efficiency of the
25 reaction, acid-base corrosion of equipments and waste pollution.

US patent 5,847,239 discloses a two-step process wherein DHM reacts with carboxylic acid such as acetic acid in the first step to produce dihydromyrcenyl acetate intermediate, followed by transesterification of the intermediate with an alcohol such as 4-tertiary-butylcyclohexanol (PTBCH)
30 to produce DHMOL and 4-tertiary-butylcyclohexyl acetate (PTBCHA) using alkoxides such as sodium methoxide as catalyst. Although this new method has some advantages, there are still many problems like lower efficiency of the reaction and complex process.

The direct hydration of DHM has received an increasing attention with the advantages of simple process and low cost. Based on a turpentine feedstock, DHMOL can be made by hydration of dihydromyrcene (citronellene), and this is currently done commercially using large quantities of strong sulphuric acid. This reaction converts the dihydromyrcene to DHMOL and also produces dilute sulphuric acid as a byproduct stream. Disposal of the waste dilute sulphuric acid in an economical and/or economically acceptable way can present difficulties. For instance DHM was directly hydrated initially using sulfuric acid as catalyst at low temperature (273–278⁰K), and dilute sulfuric acid was produced as a by-product, which raised problems such as corrosion of equipments and pollution (French Pat. 2597861).

Strategies of prior art methods for production of dihydromyrcenol predominantly involve liquid acid catalyzed hydration reaction. All the published literature so far deals with the processes used for manufacturing dihydromyrcenol wherein mineral acids are used as hydration catalyst. Neutralization of mineral acids by addition of aqueous alkali results in phase separation. The organic layer containing all manufacturing products are separated and the aqueous layer saturated with all manufacturing products is taken for effluent treatment. The different manufacturing products include cis-pinene, trans-pinene, dihydromyrcene, dihydromyrcenol, etc.

A possible alternative approach to production of DHMOL is hydration of dihydromyrcene using alternative catalysts of acid clays, zeolites etc. Many attempts at such processes have been reported in literature. Many new types of catalysts such as the Keggin-type heteropoly acids H₃PW₁₂O₄₀ (HPA) (Kozhevnikov I. V., Sinnema A., van der Weerd A. J. A., van Bekkam H., “Hydration and acetoxylation of dihydromyrcene catalyzed by heteropoly acids”, J. Mol. Cat. A: Chem, 1997, 120, 63-70), H-beta zeolite (Botella P., Corma A., Lopez nieto J. M., Valencia S., Lucas M. E., Sergio M., “Selective hydration of dihydromyrcene to dihydromyrcenol over H-beta zeolite. Influence of the microstructural properties and process variables”, Appl. Catal. A: Gen 2000, 203, 251-258), and cation exchange resin R-H modified with metal ion (Lin Y. H., Tan X. F., Xiao S. D., “Study on dihydromyrcene hydration catalyzed by modified cation exchange resins catalyst”, Ion exchange and Adsorption (China), 1998, 14, 450-456) have been applied in the hydration of DHM instead. These catalysts have shown favorable performance in the hydration of DHM, but there still exists some disadvantages including low reactive efficiency, complicated preparation and high cost. The industrial applications of these catalysts are still restrained to a certain extent.

Processes for the preparation of dihydromyrcenol, from various terpenes and terpene derivatives are well known. For example US Patent No. 2902510 contains examples showing the preparation of the alcohol in admixture with other materials. While such prior art processes are capable of producing dihydromyrcenol, the dihydromyrcenol so formed can usually be separated from the reaction mixture

to produce a pure material. However, the conversions from the reactions of the said prior art are quite low, being of the order of 20-22% in a time period of 3-6 days. Thus both time and conversion rates are the limiting steps of the reported invention.

- 5 US patent 3,487,118 discloses preparation of dihydromyrcenol by forming a mixture of dihydromyrcene, formic acid and an acid catalyst and maintaining this mixture at desirably below 40°C for a time period of about 5 hours to form dihydromyrcenol and /or dihydromyrcenol formate. DHMOL is separated from the mixture using any conventional separation techniques. Despite the controlled conditions, the disclosed invention in this patent document reports the production of cyclic materials in the reaction mixture. Also the reported conversions rates are in the range of about 50% only are reported in time period of the range of upto about 5 hours at 5-20°C.

- 15 The mentioned prior art processes involves lots of production drawbacks such as large amount of energy consumption for chilling during addition of mineral acid in presence of water, neutralization of unspent acid catalyst, and final work of the process to isolate pure product which involves lots of water separation and effluent generation.

- Hence, there is a need for a novel process and technology which shall overcome all of these problems associated with the present technology and makes this process viable on production scale with zero or very low generation of effluent.

BRIEF DESCRIPTION OF DRAWINGS

- Figure 1 show the hydration reaction of DHM, wherein one water molecule is added across the double bond having more number of substitutions in presence of the acid catalyst to produce DHMOL.
- 25 Figure 2 shows laboratory scale equipment employed for continuous conversion of Dihydromyrcene (DHM) to Dihydromyrcenol (DHMOL) and/or its ester.

DETAILED DESCRIPTION OF THE INVENTION

DEFINATIONS

- 30 Aroma olefins: The material herein defined as olefins refers to the aroma molecules similar to dihydromyrcene possessing double bonds at different position and can be hydrated using the invention described in the present invention. The olefins are selected from a group comprising but not limited to Myrcene, Ocimene, Cis-pinene, Cis-3-carene, Camphene etc.

- Dihydromyrcene: The material herein designated by the term dihydromyrcene is also known as 2, 6-dimethyl-2-octadiene or citronellene. This material can be supplied to the reaction mixture as a

chemically pure material or as commercially available materials containing approximately 90-95% dihydromyrcene.

Terpene Alcohols: The material herein defined as alcohols from the category of organic compounds possessing isoprene unit which has the molecular formula C_5H_8 . This refers to the aroma molecules similar to Dihydromyrcenol possessing two isoprene units and can be synthesized using the present technology described in the present invention.

The present invention provides a rapid process for preparation of terpene alcohols comprising of C_{10-12} carbon chain, more specifically dihydromyrcenol in good conversions. The invention provides for the synthesis of dihydromyrcenol, a fragrance alcohol, through solid phase catalysis in continuous column bed reactor, whereby the liquid acid catalyst handling and the effluent generated in the process is avoided. The disclosed invention provides a solution to the problem of effluent treatment and increases the yield of the process due to selectivity in reaction.

According to the process of this invention, dihydromyrcenol, an unsaturated aroma alcohol and/or ester is continuously produced by selective solid acid catalyzed hydration reaction. In the present method, a reaction mixture of aroma olefins, more specifically dihydromyrcene, water and a polar solvent is prepared and passed through a catalytic column bed reactor, for a period of time sufficient to form a resultant reaction mixture comprising dihydromyrcenol and/or ester thereof and to minimize the formation of undesired by-products such as citronellal. The catalytic column is maintained at temperatures in the range of about 70-120°C. The pressure of about 4-8 bar of nitrogen is maintained in the catalytic column. The resultant reaction mixture is then passed through a condenser wherein the temperature is maintained below 10°C followed by a flash evaporator. The polar solvent gets flashed partly in the flash evaporator and is collected in tank no. 9 of figure 2. The balance reaction mixture consisting of partly polar solvent, dihydromyrcenol, and unreacted dihydromyrcene along with associated impurities is fed to a distillation column. The polar solvent along with dihydromyrcene and associated impurities such as myrcene, pinane, pinine, camphene, iso-boronyl etc. gets collected from the column top and is stored in tank no. 9 of figure 2. The water and dihydromyrcenol as un-distilled column bottom fraction is fed to a separator for separation of water and non polar fractions of the reaction mixture mainly containing Dihydromyrcenol. In the separator, water and crude Dihydromyrcenol get separated. Water along with traces of Dihydromyrcenol is recycled back to tank no. 1 of figure 2, while the crude Dihydromyrcenol is fed to a final distillation column to obtain pure product of more than 99% purity. The entire system is run as a continuous operation in a closed loop circuit without disturbing the flow of reaction, which avoids the system solvent losses and also air pollution caused by solvent evaporation.

According to the process of the present invention, dihydromyrcene and water, the two immiscible reaction raw materials, were mixed in acetone using the thermodynamic properties of the ternary solvent system. The two immiscible reactants were solubilized in presence of acetone at the calculated temperature (in the range of about 70-120°C) and pressure (in the range of about 4-8bar) to obtain a single phase system. The single phase system thus obtained was contacted with the catalyst. Further, changes in the ternary phase parameters at column outlet flow facilitated product separation as a part of continuous system. Thermodynamic balancing of the three process liquids adds novelty to the system by making it continuous as compared to the batch reactions reported in the prior art process.

The invention is further explained through embodiments and examples by way of illustration only and various changes and modifications in the scope and spirit of the invention will become apparent to one skilled in the art through description. It is also to be understood that the invention is not limited to a particular methodology, protocol or reagents described as those may vary.

The reaction mechanism of the present invention has been illustrated through Figure 1. In a typical hydration reaction of DHM one molecule of water gets added across the double bond having more number of substitutions in presence of the acid catalyst.

In an aspect of the present invention the process of the invention can be operated through apparatus as shown arranged in Figure 2.

Component parts for Figure 2 can be denoted as:

- 1, 2, 3- Reaction mixture vessel
- 4- Mixing pump
- 5- Catalysis column
- 6- Cooling condenser
- 7- Flash evaporator
- 8- Dihydromyrcene distillation column
- 9- Dihydromyrcene and solvent collection tank
- 10- Aqueous-organic separator
- 11- Dihydromyrcenol distillation column
- 12- Pure Dihydromyrcenol collection tank

In another aspect of the present invention the process of the invention can be performed by selecting the solvents from a group comprising of acetone, methanol, dioxane, THF etc. The reaction solvents can be used in combination with 50% water. In presence of methanol as solvent it was found that citronellal (a terpene aldehyde) formation was increased significantly as side product, while there was

no formation of DHMOL. Among the various solvents used, maximum productivity of 1.95 mg/ml of resin per hr was obtained when dioxane was used as a solvent.

In yet another aspect of the invention the residence time is in the range of about 75-150 minutes. Most desirable conversion rates were obtained when the residence time was closer to the range of about 75 minutes.

In a further aspect of this invention the catalytic column bed reactor mentioned in the invention was maintained at 70-120°C temperature and at about 4-8 bar nitrogen pressure. The conversion of the process is in the range of 50-70% more specifically 60% and yield of the process is in the range of 90-98% more specifically 95%. Thus, the preparation of dihydromyrcenol according to the process of this invention gives good conversion of dihydromyrcene to the alcohol.

In still another aspect of the present invention the catalysts employed in carrying out the process of the invention are perhalogenated ion-exchange polymers having substantially halogenated aliphatic backbone with pendent sulphonic or carboxylic groups. The catalyst composition of the process herein comprises a perhalogenated ion-exchange polymer containing sulphonic acid groups supported on an inert carrier having a hydrophobic surface with a mean pore diameter of at least 1000 angstrom.

In another aspect of the present invention the catalysts used in the processes of this invention are pre-activated prior to its application for the said reaction. The method mentioned in the process of invention includes treatment of the catalyst with 0.1-1N mineral acid followed by water washing to reduce the pH of the solution to neutral. The mineral acid used in the process of the invention is sulphuric acid of 98% strength.

In an advantageous aspect of the present invention the reaction yields are significantly higher and range from about 95% to about 98%. These high yields reflect the selectivity of the process which makes it more efficient as compared to the prior art process and viable for commercial application.

In yet another advantageous aspect of the invention, the residence time or the reaction time is not more than 75 minutes in a continuous cycle. The low residence time obtained because of the inventive process parameters yields higher productivity which makes the reaction commercially viable.

In accordance with the present invention, in first embodiment there is provided a process for production of terpene alcohols, the process comprises reacting aroma olefins with a polar solvent and water in a catalytic column for a retention time sufficient to produce a reaction mixture comprising

polar and non polar fractions; separating the said polar and non polar fractions by passing the said reaction mixture through a condenser and subsequently through a separator; distilling the said polar fraction to separate polar solvent and water; and distilling the non-polar fraction to obtain the desired terpene alcohols/aroma olefins and recycle the un-reacted aroma olefins.

- 5 The second embodiment relates to the process for production of terpene alcohols as disclosed in the invention wherein the terpene alcohol is selected from the group comprising of but not limited to linalool, geraniol, citronellol, nerol, α -terpineol, borneol, terpinen-4-ol, limonen-4-ol, dihydromyrcenol, myrcenol, and mixtures thereof.

10 Third embodiment relates to the process for production of terpene alcohols wherein the terpene alcohol is dihydromyrcenol.

Fourth embodiment relates to the process for production of terpene alcohols as disclosed in the invention wherein the aroma olefins is selected from a group comprising but not limited to Myrcene, Ocimene, Cis-pinene, Cis-3-carene, Camphene, dihydromyrcene.

15 Fifth embodiment relates to the process for production of terpene alcohols as disclosed in the invention wherein the aroma olefin is dihydromyrcene.

Sixth embodiment relates to the process for production of terpene alcohols as disclosed in the invention wherein the polar solvent is selected from a group consisting of but not limited to acetone, dioxane, tetrahydrofuran.

20 Seventh embodiment relates to the process for production of terpene alcohols as disclosed in the invention, wherein the polar solvent is acetone.

Eighth embodiment relates to the process for production of terpene alcohols as disclosed in the invention, wherein the ratio of polar solvent and water is in the range of 0.1 to 1.0

25 Ninth embodiment of the present invention relates to the process for production of terpene alcohols as disclosed in the present invention wherein the reaction is performed at a temperature within the range of 70°C to 120°C, more specifically between 85°C to 95°C

Tenth embodiment relates to the process for production of terpene alcohols as disclosed in the present invention wherein the reaction is performed at pressure of 4 to 8 bars, more specifically between 3-4 bars.

30 Eleventh embodiment relates to the process for production of terpene alcohols as disclosed in the present invention, wherein the catalytic column is selected from a group comprising of Polystyrene based cation donors, polymethacrylate based cation donors, silica based cation donors, and metal halide based cation donors in macroporous bead form.

Twelfth embodiment relates to the process for production of terpene alcohols as disclosed in the present invention wherein the catalytic column is polystyrene based cation donor.

Thirteenth embodiment relates to the process for production of terpene alcohols as disclosed in the present invention, wherein the retention time is in the range of 75 minutes to 150 minutes.

- 5 Fourteenth embodiment relates to the process for production of terpene alcohols as disclosed in the present invention, wherein the condenser is maintained at a temperature between 5-10°C.

The invention is further elaborated through the following examples which are meant only to clarify the invention and should not be interpreted so as to narrow the scope of the invention.

10

EXAMPLES

EXAMPLE 1

The invention was carried out using the equipment shown in Figure 2, which comprises a reaction system for continuous processing of dihydromyrcene as reaction substrate to produce dihydromyrcenol as reaction product. The equipment includes a catalyst bed 5 of PRR 450 catalyst, a pre-halogenated ion-exchange polymer with styrene DVB backbone and side chain with a pendant sulphonic acid site on the end of the side chain, packed in a jacketed column reactor of optimized dimension. The reactant streams mainly fresh dihydromyrcene, and water was pumped from tank 1, 2, and the recycled mixture of solvent and unreacted dihydromyrcene was pumped from tank 3. All the three streams were pumping to the catalysis column 5 through a mixing pump 4. The reaction assembly including the reactant tanks and column are maintained at optimized temperature and pressure using external heating system and N₂ gas respectively. Out let of the reaction column was throttled into a flash evaporator 7 connected via a cooling condenser 6. The content from the flash evaporator was fed to a distillation column 8 to recover the un-reacted dihydromyrcene and solvent in a transfer tank 9. The bottom collection of the distillation assembly was fed to the aqueous-organic separator 10 for separation of product dihydromyrcenol from water. The crude dihydromyrcenol separated from the separator was finally fed to a factional distillation column 11 for recovery of pure dihydromyrcenol. Pure dihydromyrcenol gets continuously stored in final collection tank 12.

30 EXAMPLE 2

A liquid reaction mixture containing 3.5% of 1,6-Octadiene, 3,7-dimethyl- (dihydromyrcene) and 42.5% water in acetone as solvent was contacted with PRR 450 catalyst a pre-halogenated ion-exchange polymer with styrene DVB backbone and side chain with a pendant sulphonic acid site on the end of the side chain. The said catalyst was packed in a column reactor design especially for the process. The said reaction mixture was passed through the catalyst column. The reaction was carried out continuously at temperature 95°C and pressure at 4 bar. The column reactor was jacketed for

5 maintenance of the said temperature and non-return valve for maintaining the desired pressure. The desired pressure was maintained using N₂ gas cylinder with control flow meter. The reaction mixture of said composition was passed down-flow through the column reactor at the residence time of 60 minutes. The outlet reaction mixture was collected through a coiled condenser maintained at temperature below 5⁰C through a throttle valve and analyzed for the conversion and yield.

EXAMPLE 3

10 Reaction of example 1 is carried out at temperatures 120⁰C and at pressures at 8 bar of nitrogen pressure. The column reactor was jacketed for maintenance of the said temperature and non-return valve for maintaining the desired pressure. The desired pressure was maintained using N₂ gas cylinder with control flow meter. The reaction mixture of said composition was passed down-flow through the column reactor at the residence time of 75 minutes. The outlet reaction mixture was collected through a coiled condenser maintained at temperature below 5⁰ C through a throttle valve and analyzed for the conversion and yield.

EXAMPLE 4

15 Reaction of example 1 is carried out at temperatures 105⁰C and at pressures at 4 bar of nitrogen pressure. The column reactor was packed with PRR 650 catalyst an ion-exchange polymer with methacrylate backbone and side chain with a pendant carboxylic acid site on the end of the side chain. 20 The column reactor was jacketed for maintenance of the said temperature and non-return valve for maintaining the desired pressure. The desired pressure was maintained using N₂ gas cylinder with control flow meter. The reaction mixture of said composition was passed down-flow through the column reactor at the residence time of 75 minutes. The outlet reaction mixture was collected through a coiled condenser maintained at temperature below 5⁰ C through a throttle valve and analyzed for the conversion and yield. 25

EXAMPLE 5

30 Reaction of example 1 is carried out at temperatures 105⁰C and at pressures at 4 bar of nitrogen pressure. The column reactor was packed with PRR 550 catalyst an ion-exchange polymer with styrene DVB backbone and side chain with a pendant carboxylic acid site on the end of the side chain. The column reactor was jacketed for maintenance of the said temperature and non-return valve for maintaining the desired pressure. The desired pressure was maintained using zero air gas cylinder with control flow meter. The reaction mixture of said composition was passed down-flow through the column reactor at the double residence time of 150 minutes. The outlet reaction mixture was collected 35 through a coiled condenser maintained at temperature below 5⁰ C t hrough a throttle valve and analyzed for the conversion and yield.

EXAMPLE 6

Reaction of example 1 is carried out at temperatures 95⁰C and at pressures at 4 bar of nitrogen pressure. The column reactor was jacketed for maintenance of the said temperature and non-return valve for maintaining the desired pressure. The desired pressure was maintained using N₂ gas cylinder with control flow meter. The reaction mixture of said composition was prepared in 1,4 dioxane as reaction solvent and passed through the column reactor at the residence time of 75 minutes. The outlet reaction mixture was collected through a coiled condenser maintained at temperature below 5⁰ C through a throttle valve and analyzed for the conversion and yield.

EXAMPLE 7

Reaction of example 1 is carried out at temperatures 95⁰C and at pressures at 4 bar of nitrogen pressure. The column reactor was jacketed for maintenance of the said temperature and non-return valve for maintaining the desired pressure. The desired pressure was maintained using N₂ gas cylinder with control flow meter. The reaction mixture of said composition was prepared in diacetone alcohol as reaction solvent and passed through the column reactor at the residence time of 75 minutes. The outlet reaction mixture was collected through a coiled condenser maintained at temperature below 5⁰ C through a throttle valve and analyzed for the conversion and yield.

EXAMPLE 8

Reaction of example 1 is carried out at temperatures 95⁰C and at pressures at 4 bar of nitrogen pressure. The column reactor was jacketed for maintenance of the said temperature and non-return valve for maintaining the desired pressure. The desired pressure was maintained using N₂ gas cylinder with control flow meter. The reaction mixture of said composition was prepared in tetrahydrofuran as reaction solvent and passed through the column reactor at the residence time of 75 minutes. The outlet reaction mixture was collected through a coiled condenser maintained at temperature below 5⁰ C through a throttle valve and analyzed for the conversion and yield.

EXAMPLE 9

Reaction of example 1 is carried out at temperatures 95⁰C and at pressures at 4 bar of nitrogen pressure. The column reactor was jacketed for maintenance of the said temperature and non-return valve for maintaining the desired pressure. The desired pressure was maintained using N₂ gas cylinder with control flow meter. The reaction mixture of said composition using myrcene as the source of olefin was prepared in acetone and passed through the column reactor at the residence time of 75 minutes. The outlet reaction mixture was collected through a coiled condenser maintained at temperature below 5⁰ C through a throttle valve and analyzed for the conversion and yield.

EXAMPLE 10

Reaction of example 1 is carried out at temperatures 95°C and at pressures at 4 bar of nitrogen pressure. The column reactor was jacketed for maintenance of the said temperature and non-return valve for maintaining the desired pressure. The desired pressure was maintained using N_2 gas cylinder
5 with control flow meter. The reaction mixture of said composition using ocemene as the source of olefin was prepared in acetone and passed through the column reactor at the residence time of 75 minutes. The outlet reaction mixture was collected through a coiled condenser maintained at temperature below 5°C through a throttle valve and analyzed for the conversion and yield.

We Claim:

1. A process for production of terpene alcohols, the process comprises
 - a. preparing a reaction mixture of aroma olefins, water and a polar solvent;
 - b. passing the said reaction mixture through a solid acid catalytic column for a retention
5 time sufficient to produce a resultant reaction mixture, the said resultant reaction mixture comprising polar and non polar fractions;
 - c. separating the said polar and non polar fractions by passing the said reaction mixture through a condenser and subsequently through a separator;
 - d. distilling the said polar fraction to separate polar solvent and water; and
 - 10 e. distilling the non-polar fraction to obtain the desired terpene alcohols and recycle the un-reacted aroma olefins.
2. The process of claim 1 wherein the terpene alcohol is selected from the group comprising linalool, geraniol, citronellol, nerol, α -terpineol, borneol, terpinen-4-ol, limonen-4-ol, dihydromyrcenol, myrcenol, and mixtures thereof.
- 15 3. The process of claim 1 wherein the terpene alcohol is dihydromyrcenol.
4. The process of claim 1 wherein the aroma olefins is selected from a group comprising Myrcene, Ocimene, Cis-pinene, Cis-3-carene, Camphene, dihydromyrcene.
5. The process of claim 1 wherein the aroma olefin is dihydromyrcene.
6. The process of claim 1 wherein the polar solvent is selected from a group comprising of acetone,
20 dioxane, tetrahydrofuran.
7. The process of claim 1 wherein the polar solvent is acetone.
8. The process of claim 1 wherein the ratio of polar solvent and water is in the range of 0.1 to 1.0.
9. The process of claim 1 wherein the reaction is performed at a temperature within the range of 70°C to 120°C, more specifically between 85°C to 95°C.
- 25 10. The process of claim 1 wherein the reaction is performed at pressure of 4 to 8 bars, more specifically between 3-4 bars.
11. The process of claim 1 wherein the said catalytic column is selected from a group comprising polystyrene based cation donors, polymethacrylate based cation donors, silica based cation donors, and metal halide based cation donors in macroporous bead form.
- 30 12. The process of claim 1 wherein the said catalytic column is polystyrene based cation donor.
13. The process of claim 1 wherein the retention time is in the range of 75 minutes to 150 minutes.

14. The process of claim 1 wherein the condenser is maintained at a temperature between 5-10°C.

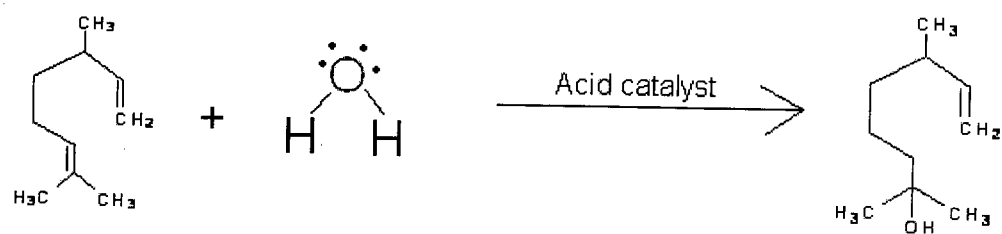


Figure: 1

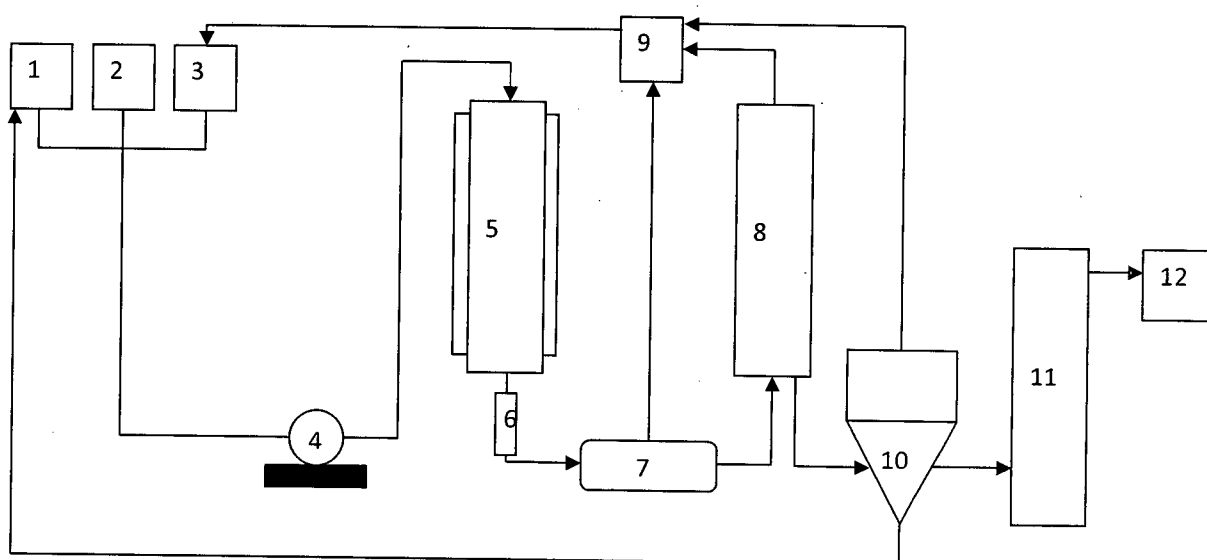


Figure: 2