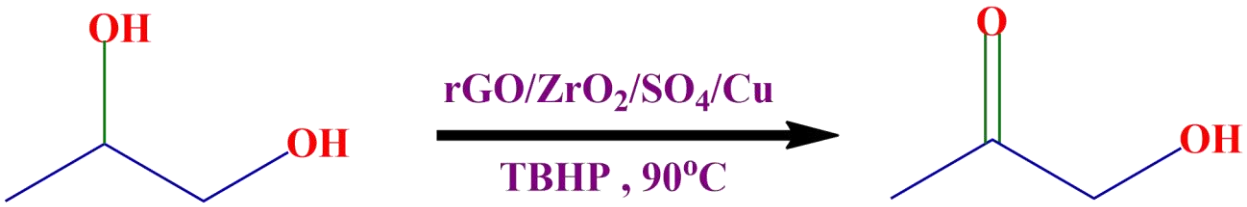
Total sheets: 8

Sheet: 1

Figure 1.



Propylene glycol (PG)

Hydroxyacetone (HA)

Signature

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Ms. DIVYA SRIDHARAN,
Patent Agent (IN/PA-3292)

Figure 2.

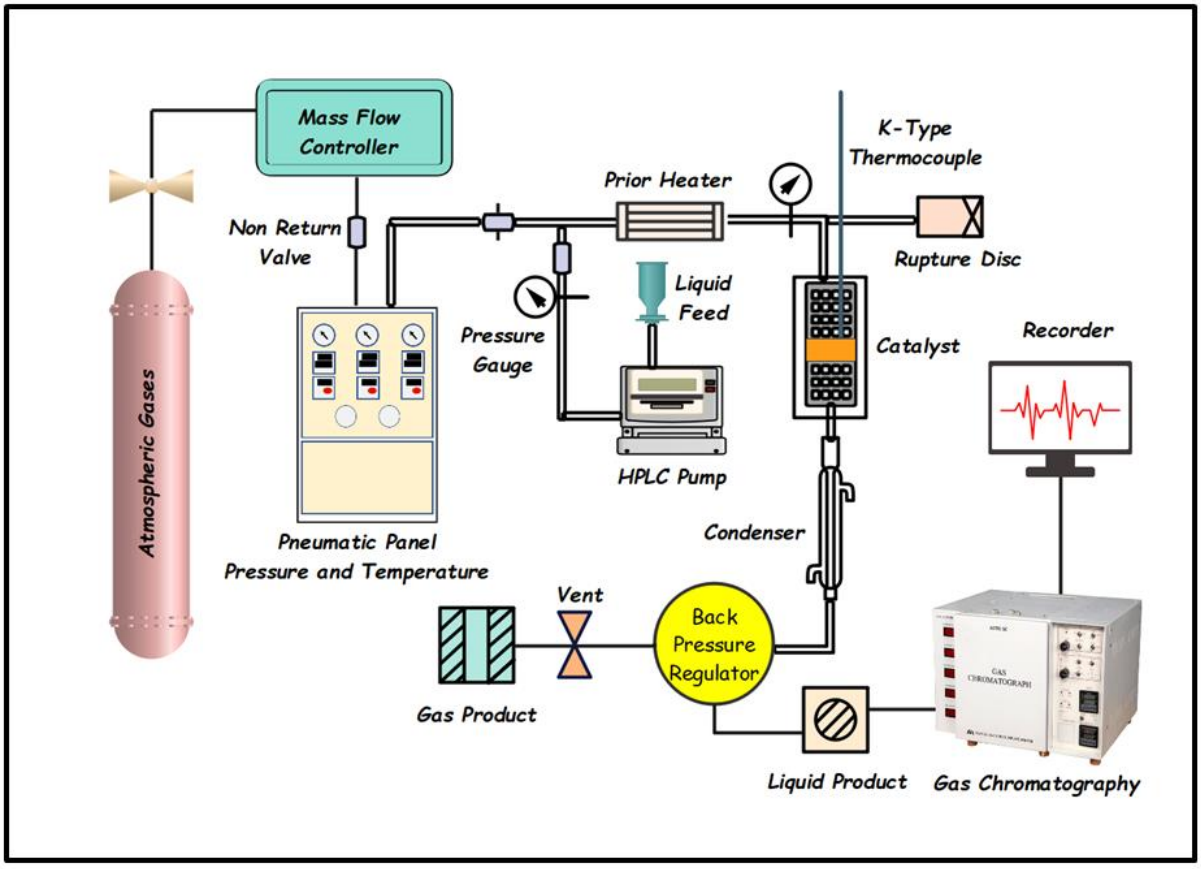

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Figure. 3.

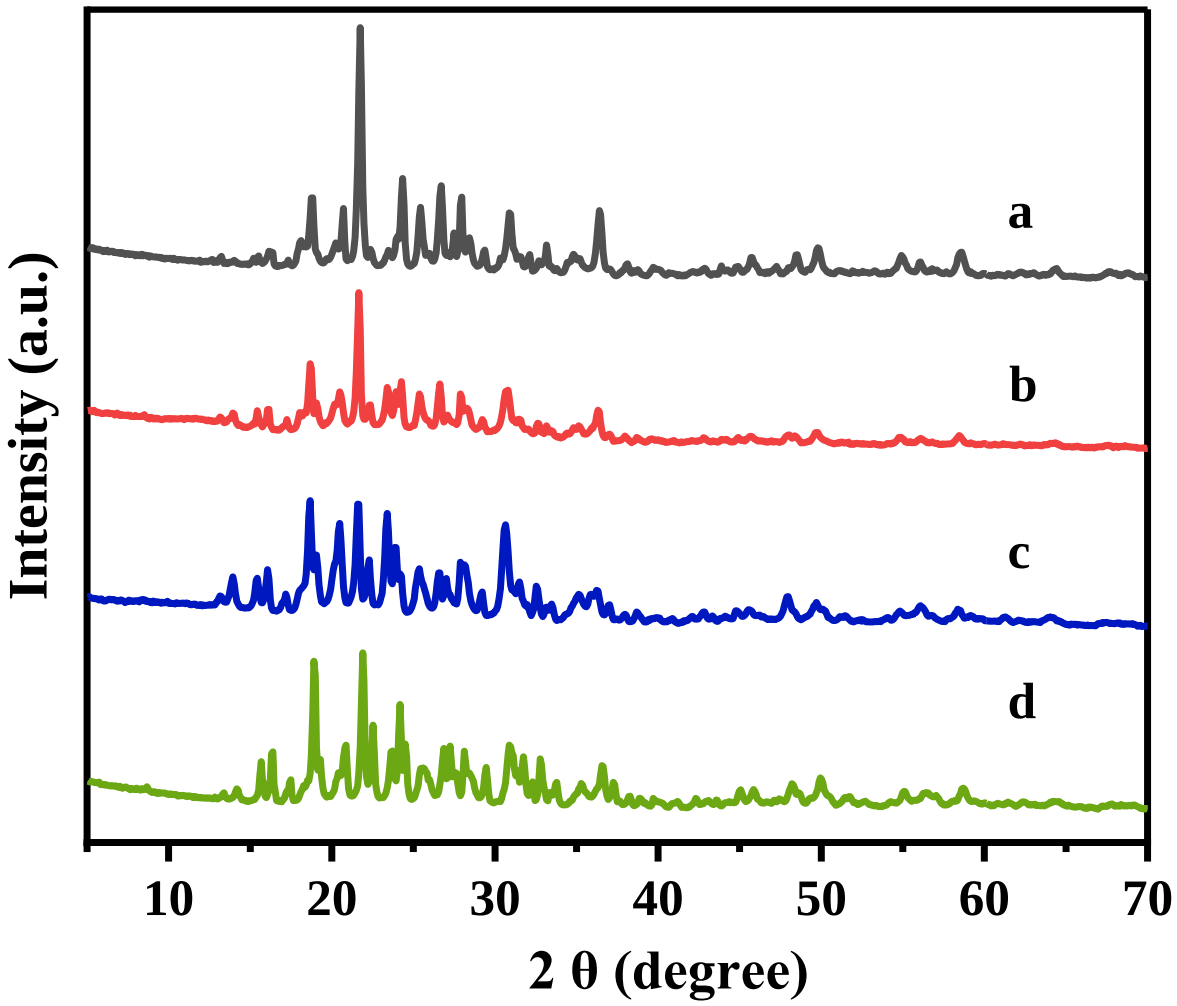

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Figure. 4.

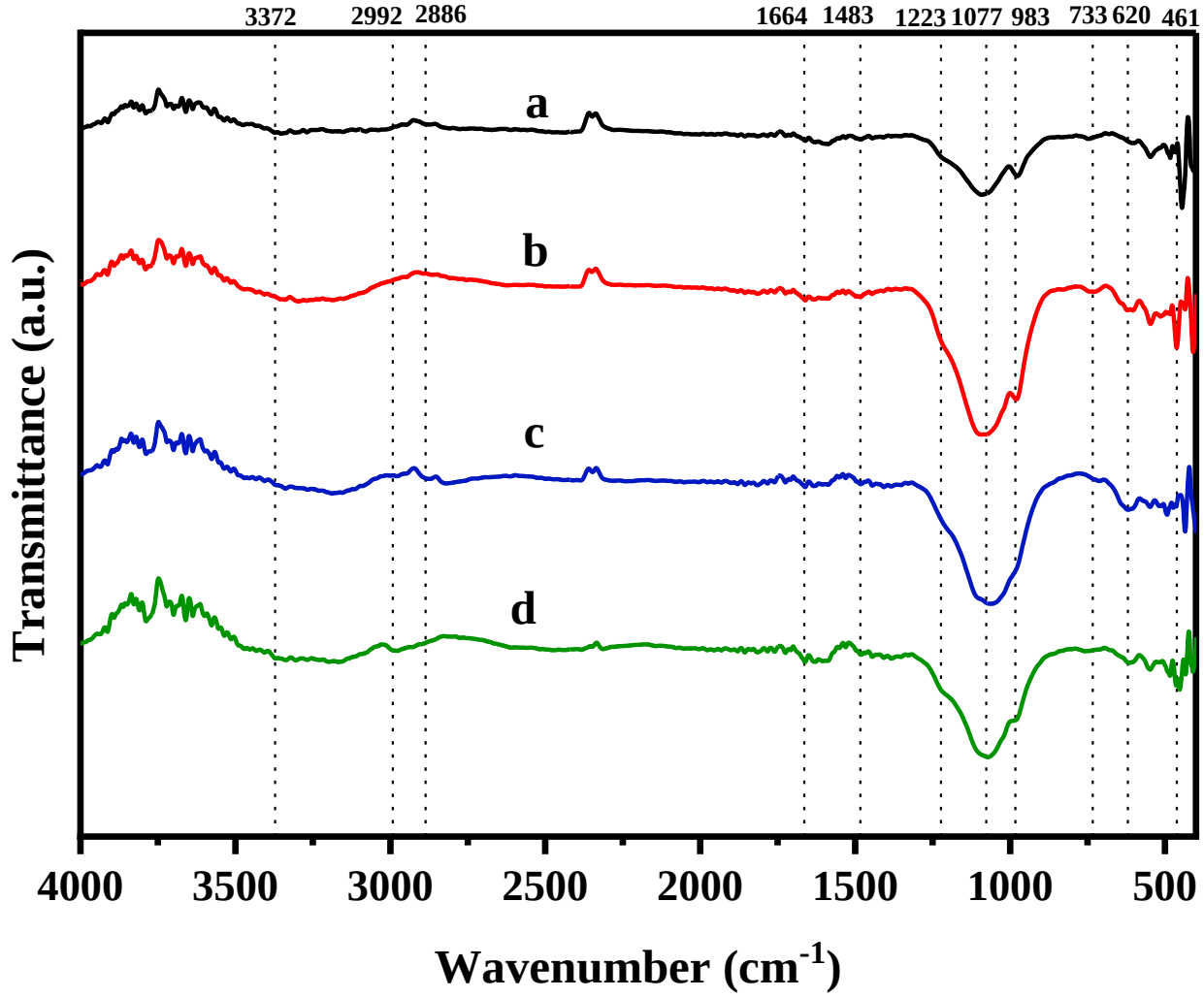

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Figure. 5.

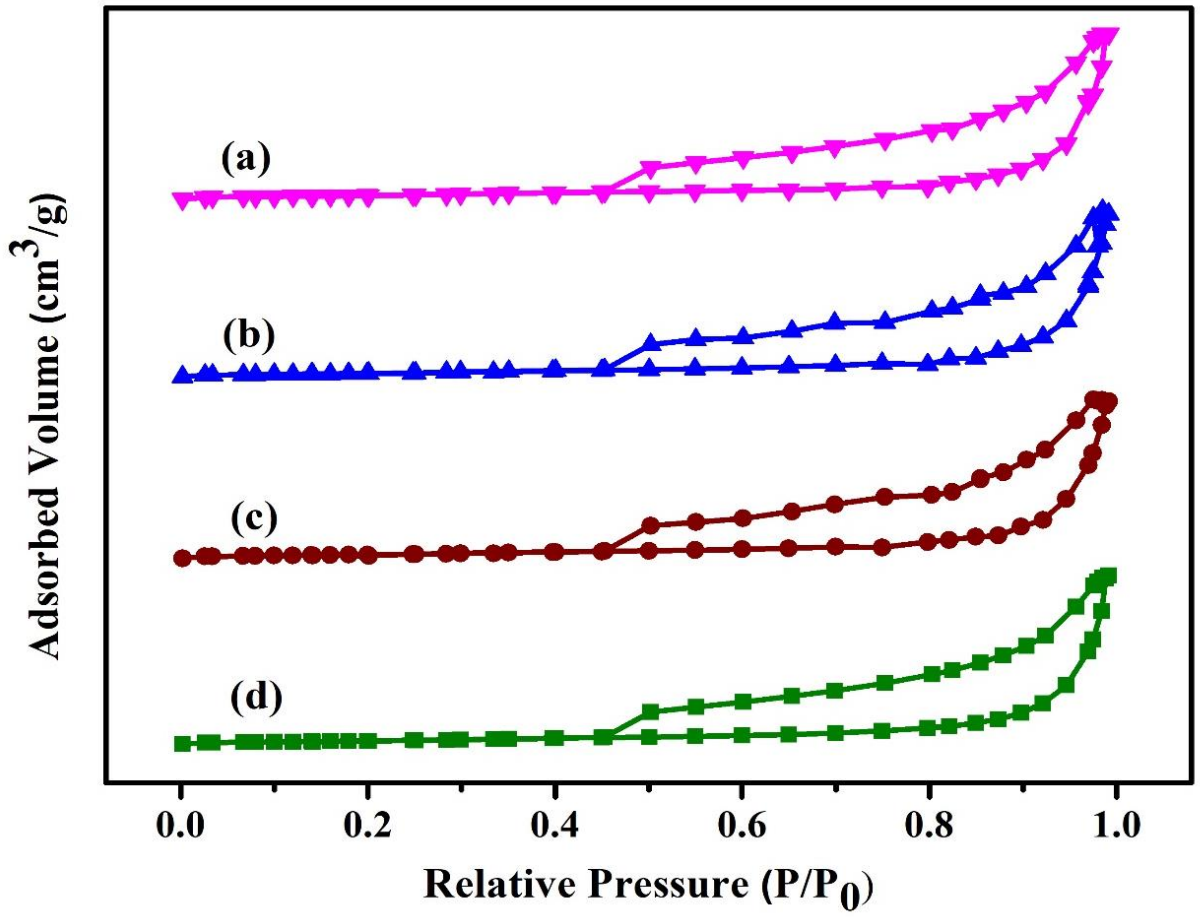

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Figure. 6.

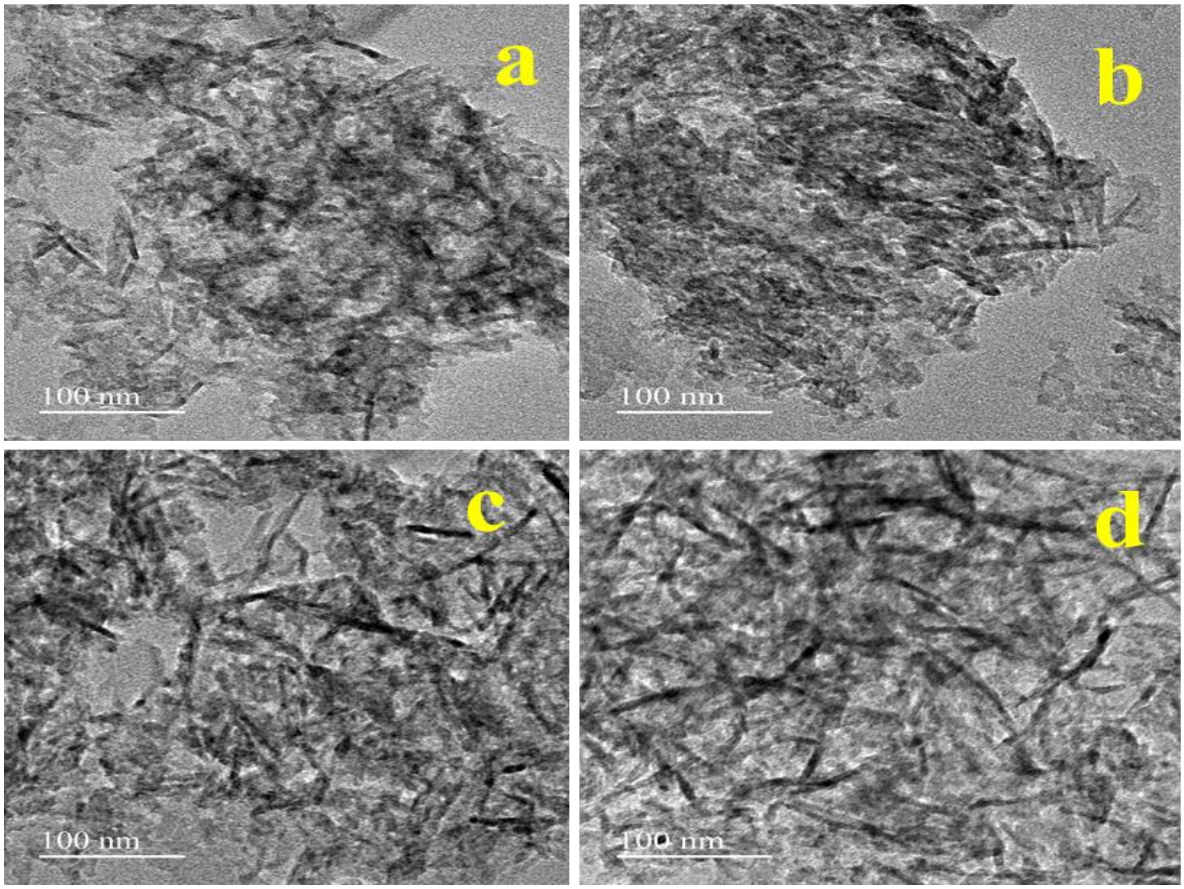

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Figure.7.

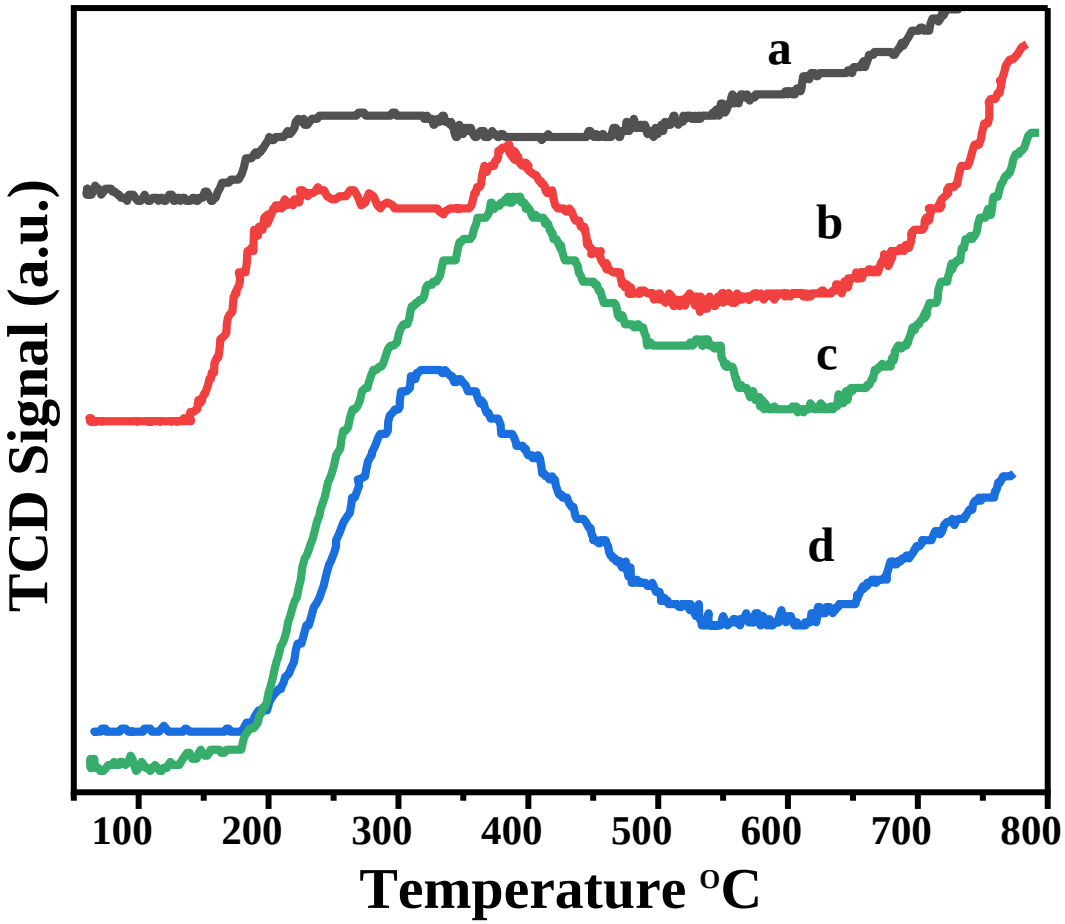

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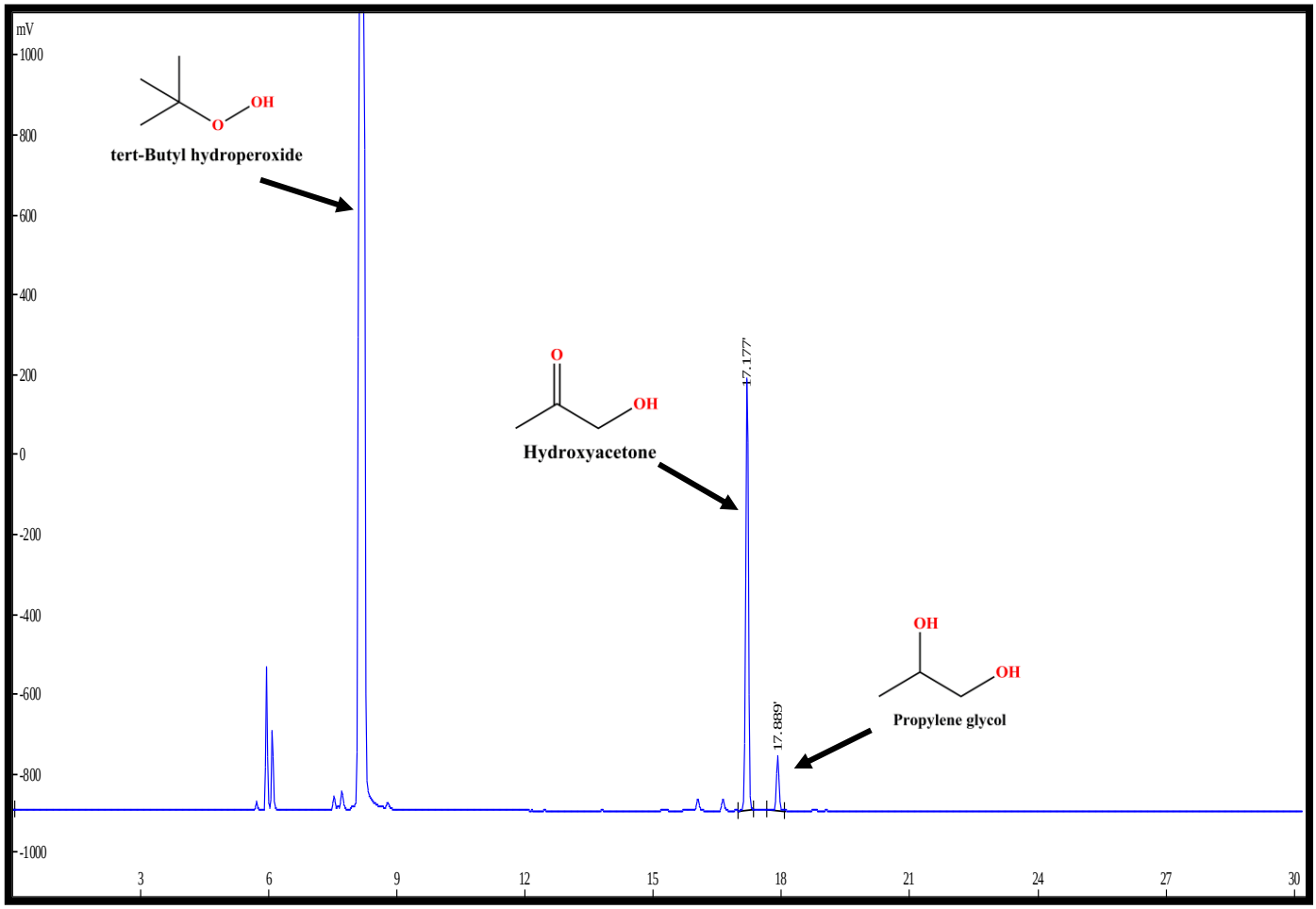
Figure 8.



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FORM2

THE PATENTS ACT, 1970 (39 of 1970)

&

The Patents Rules, 2003

COMPLETE SPECIFICATION (see section 10)

Title

ZrO_2/SO_4/Cu nano particles supported on reduced graphene oxide for selective oxidation of propylene glycol in continuous reactor

Applicants

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4. Dr. K. SIVAGAMI, Associate Professor, School Of Chemical Engineering, Vellore institute Of Technology, Vellore -632 014, Tamil Nadu, India.

The following specification particularly describes the invention and the manner in which it is to be performed.

$ZrO_2/SO_4/Cu$ nano particles supported on reduced graphene oxide for selective oxidation of propylene glycol in continuous reactor

PREAMBLE

The specification describes that it utilizes a bimetallic heterogeneous catalyst to convert propylene glycol to hydroxyacetone. The production of hydroxyacetone (Acetol) results from selective propylene glycol oxidation at the secondary hydroxyl group. However, due to the near reactivity of polyols, selective oxidation of only one hydroxyl group is a difficult operation. In this invention$ZrO_2/SO_4/Cu$ nanoparticles supported on reduced graphene oxide for selective oxidation of propylene glycol in a continuous fixed-bed reactor with propylene glycol conversion and hydroxyacetone selectivity results in 90 to 100%.

BACKGROUND AND PRIOR-ART

[0001] In propylene glycol (hereinafter called PG), which contains both primary and secondary alcoholic groups, the secondary alcoholic group undergoes preferential oxidation over the primary alcoholic group is quite difficult. Selective oxidation of propylene glycol using a bimetallic heterogeneous catalyst was graphically illustrated in Figure.1

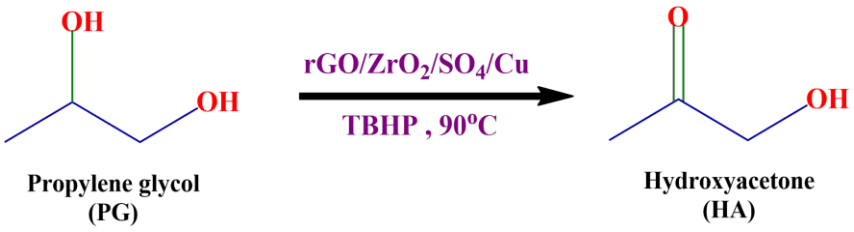

Figure.1 Selective Oxidation of Propylene glycol (PG)

[0002] Glycerol hydrogenolysis involves dehydration to yield hydroxyacetone(hereinafter called HA).The following US patents provide information on the numerous techniques and catalytic methods used to produce hydroxyacetone (U.S.Pat.No.10,889,840 B2; U.S.Pat.No.8,809,593 B2;U.S.Pat.No.8,686,192 B2;U.S.Pat.No.8,835,694 B2; U.S.Pat.No. 8,017,816 B2). The selectivity

of hydroxyacetone is only up to 65% when it is made from glycerol using metallic catalysts such as copper, nickel, alumina, and others in the presence of water.

[0003] The U.S.Pat.No. 8,017,816 B2 teaches us the “Method ofProducing LowerAlcohols from Glycerol” and U.S.Pat.No.8,809,593 B2 “Process for preparation of Hydroxyacetone or Propylene glycol.” Teaches us the Cu, Cr, Al, Mg, Zr, Zn, Si, or mixtures of these elements in the presence of a solvent, are the catalysts used for the conversion of glycerol to hydroxyacetone and other compounds. CN102070422B patent teaches us under the action of a copper-based catalyst, the reactions are carried out continuously in a fixed bed reactor under normal pressure. Glycerol is dehydrated to produce HA (acetol).

[0004] U.S.Pat.No.10,889,840 B2 teaches us about using methylglyoxal reductase enzymes which convert methylglyoxal into hydroxyacetone. CN101284234A patent reported to us natural glycerin is selectively dehydrated to produce hydroxyacetone in a fixed bed flowing-type reactor with a metal catalyst. Glycerin conversion was reported to be 99%, while hydroxyacetone selectivity was reported to be 89%.U.S.Pat.No.8,686,192 B2 teaches us the conversion methods for cellulose or other bio-renewable carbohydrate sources into high-value chemical compounds (glycerin, propylene glycol, ethylene glycol, hydroxyacetone).

[0005]Hydroxy acetone and lactic acid synthesis from aqueous propylene glycol/hydrogen peroxide catalysis on Pd-black have been reported, with authors claiming the conversion PG to HA was achieved by 80 to 85% conversion and selectivity towards HA was 40 to 45% (R.S. Disselkamp et al. /Catalysis Communications. 2008 Jul 20;9(13):2250-2). Propylene glycol oxidation with tert-butyl hydroperoxide over Cr-containing metal-organic frameworks MIL-101 and MIL-100 reported that the predominant oxidation product was hydroxyacetone (HA), whereas acetaldehyde and acetic acid were minor oxidation products, with 88% selectivity towards HA and 10% PG conversion (V.V. Torbina et al. / *Catalysis Today* 278 (2016): 97-103). Propylene glycol oxidation with hydrogen peroxide over Zr-containing metal-organic framework UiO-66 has been reported, with authors claiming the PG oxidation over UiO-66 proceeds 80 to 85% selectivity towards HA (V.V. Torbina et al./*Catalysis Today* 333 (2019): 47-53).

[0006] According to prior art search results, 100% selectivity towards hydroxyacetone has not yet been achieved by the liquid-phase selective oxidation of propylene glycol. Furthermore, the selective oxidation of propylene glycol to hydroxyacetone in the present inventionof a bimetallic heterogeneous catalyst to achieve 90 to 100% selectivity to hydroxyacetone by selective oxidation of propylene glycol.

OBJECTIVES OF THE INVENTION

[0007] The primary objective of the invention is to provide selective oxidation of propylene glycol to hydroxyacetone, through liquid phase oxidation using ZrO_2/SO_4/Cu nanoparticles supported on reduced graphene oxide catalyst.

[0008] Another objective is to develop multifunctional, enduring, reusable, and heterogeneous efficient nanocatalysts.

[0009] Yet another objective of this invention is to carry out the reaction in a continuous fixed bed reactor by loading the feed PG through a fixed bed of the catalyst.

[0010] Another objective of the present invention is to develop a method of preparation that shows 90 to 100% selectivity towards the selective oxidation of propylene glycol to hydroxyacetone by generating directly upgradable products with the use of the catalyst.

[0011] The objective of achieving hydroxyacetone (HA) is confirmed through GC analysis.

[0012]Yet another objective of this invention is examining the impact of the variation in reaction factors, such as reaction temperature and reaction pressure, the influence of WHSV, solvent and time parameters were optimized to maximize Propylene glycol (PG) conversion and hydroxyacetone (HA) selectivity.

SUMMARY OF THE INVENTION

[0013] The present disclosure relates to a process for the liquid-phase selective oxidation of propylene glycol using ZrO_2/SO_4/Cu nanoparticles supported on reduced graphene oxide catalyst. The method for creating copper-infused sulfated zirconium oxide heterogeneous bimetallic nanoparticles supported on reduced graphene oxide as the catalyst is described in this invention. The synthesized rGO/ZrO_2(10%)/SO_4^{2-}(4%)/Cu (5–20%) nano-catalyst, possessing various active Brønsted and Lewis acid sites were employed for the selective oxidation of the propylene glycol reaction.

[0014] In a preferred aspect of the invention, the various catalyst composition ofrGO/ZrO_2(10%)/SO_4^{2-}(4%)/Cu (5–20%) nano-catalyst is used for selective oxidation of PG to HA.Utilizing rGO/ZrO_2/SO_4/Cu (15) as the catalyst in the Continuous reactor with optimum conditions to achieve high conversion, the liquid-phase oxidation of propylene glycol using tert-Butyl hydroperoxide (TBHP) as solvent was studied. Under these studies, rGO-supported sulfated zirconium oxide incorporating Cu (15%) exhibited excellent catalytic activity and stability, with a PG conversion rate of 90 to 100% and HA selectivity towards 95 to 100%.

[0015] Another aspect of the invention, in the presence of the rGO/ZrO_2/SO_4/Cu (15%), excellent reusability was attained even after seven consecutive cycles without any metal leaching or any reduction in catalytic activity in the Continuous reactor.

BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1 is a schematic flow of the Selective oxidation of Propylene glycol(PG).

Figure 2 is a schematic flow diagram of high pressured fixed bed Continuous reactor.

Figure 3 illustrates the X-ray diffraction patterns of the catalysts.

Figure 4 illustrates the FT-IR spectra of the catalysts.

Figure5 illustrates the N_2-adsorption isotherms of the catalysts.

Figure 6 illustrates the High-resolution transmission electron microscopy (HR-TEM)studies of the catalysts.

Figure 7 illustrates the Temperature-programmed desorption (NH_3-TPD)analysis of the catalysts.

Figure 8 illustrates the gas chromatography analysis of the oxidation of propylene glycol.

DESCRIPTION OF THE INVENTION

[0016] The present invention relates to a process for the selective oxidation of propylene glycol into hydroxyacetone. By interacting in a continuous reactor with a catalyst composite made by depositing ZrO_2/SO_4/Cu nanoparticles supported on reduced graphene oxide.

[0017] This invention's embodiments are not confined to specific techniques of PG conversion, which can vary and are understood by the prior art. It is also noted that any terminology used herein is for the sole purpose of describing specific embodiments and is not meant to be restricting in any way or scope.

[0018] In an embodiment of this invention, propylene glycol (PG)was selectively oxidized using a variety of rGO/ZrO_2(10%)/SO_4(4%)/Cu (Cu molar ratio = 5, 10, 15, and 20%) samples under continuous reaction conditions. By examining the impact of the variation in reaction factors, such as reaction temperature (60-100 °C), and reaction pressure, the influence of WHSV, solvent and

time parameters were optimized to maximize Propylene glycol (PG) conversion and hydroxyacetone (HA) selectivity**.**

[0019] In another embodiment of this invention, the reactions were carried out in a 6 mm inner diameter down-flow mode fixed bed stainless steel reactor (high pressure). The catalyst (size 1-2 mm) was taken and dried in CO_2-free air for 6 hours at 250°C. The reactor was then cooled for 1 hour at standard pressure and temperatures ranging from 60 to 110°C. When the airflow, temperature and pressure were tuned to the required values, the feed was then injected. The reactions occurred at temperatures between 60 and 100°C, under pressures of 10 bar, 3.2 g of catalyst loaded, and WHSV (h^{-1}).

[0020] In an important embodiment of the invention, the propylene glycol example, the free air (CO_2 free)/feed (mole ratio) was 1:1 (solvent comprising 25 mmol aqueous tertbutyl hydroperoxide (TBHP), propylene glycol (20 mmol)). The output components were evaluated in a gas chromatograph machine equipped with a flame ionization detector using N_2 carrier gas in SGE BPX70 capillary column.

[0021] Yet another embodiment of the invention is carried out in a continuous process. The invention process involves recycling experiments that take place in a continuous reactor.

EXAMPLES

The following examples are given to illustrate the process of the present invention and should not be construed to limit the scope of the present invention.

EXAMPLE 1

This example illustrates the preparation of reduced graphene oxide (rGO). A mixture of concentrated H_2SO_4 and $H_3PO_4$with a 9:1(v/v) volume ratio (720 ml: 80 ml) is stirred for 15min. The mixture was then added to a 1000 ml glass beaker containing graphite (6g, 1wt Equiv) and $KMnO_4$ (36 g, 6wt Equiv) and cooled under an ice bath to keep the reaction at a low temperature

and avoid any gas evolved during the oxidation step (15-20min). After that, the reaction was stirred for 15 hours at (50–55°C). After the reaction had cooled to room temperature and 10mL of 30% H_2O_2 was added to convert the manganese ion to soluble manganese sulphate and manganese oxides. The solution was centrifuged at 4000 rpm by washing with DI water, followed by 30% HCl aqueous solution and by ethanol, several times and graphene oxide (GO) was generated.

The resulting GO was diluted in deionized water and then sonicated for an hour to create a homogeneous solution, keeping the pH value between 10 to 11 (by using 0.1 M of NaOH solution). Hydrazine hydrate was added, and it was stirred for one hour at 90 °C. The solid product was filtered out using Whatman filter paper and washed with ethanol and distilled water. Then it was dried in an oven at 110 °C.

EXAMPLE 2

The preparation of sulphated zirconia support on rGO is described in this example. The synthesized rGO was dried at 383 k and calcined for 5 hours at 550°C. Using a hydrolysis technique, ZrO_2 (10 wt%) was deposited on reduced graphene oxide. Initially, 4g of rGO was dissolved in 60 mL of water containing 1.422g of $ZrOCl_28H_2O$ (ZrO_2 source) to create ZrO_2 (10 wt.%)-rGO. The mixture was stirred for two hours. Sulphated ZrO_2 possesses extremely active Brønsted and Lewis acid sites, which increases their surface acidity and would favour oxidation processes. By adding 60ml of 2M H_2SO_4 solution to the ZrO_2-rGO solution at room temperature and stirring for two hours, 4% sulfation was achieved. The sulphate sample was dried at 110°C and then calcined for 5 hours at 550°C to obtain the final catalysts known as rGO/ZrO_2 /SO_4^{2-}.

EXAMPLE 3

The preparation of impregnation of Cu sulphated zirconia support on rGO is described in this example. ThesynthesizedrGO/ZrO_2/$SO_4$was impregnated with metal Cu through the wet impregnation method utilizing copper sulphate as a metal precursor. By employing this method, the percentage of copper varied from 5-20%. At 110°C, the Cu-containing rGO/ZrO_2 /SO_4 was dried

for 12 hours. The dried sample was then calcined for 5 hours at 550°C. The rGO/ZrO_2 /SO_4/Cu(x) catalyst was obtained, where x is the copper ratio. Various ratio of rGO/ZrO_2(10%)/SO_4(4%)/Cu (Cu molar ratio = 5, 10, 15, and 20%) catalyst was used to examine the catalytic activity and utilized for the selective oxidation of propylene glycol to hydroxyacetone.

EXAMPLE 4

This example illustrates the physicochemical properties. The textural properties of the solid catalyst, particularly its specific surface area and average pore diameter, must be measured in heterogeneous catalysis. The surface areas and average pore diameters of oxide catalysts, such as those shown in Example 3, may be easily calculated by knowing the quantity of nitrogen gas adsorbed by the samples at different pressures at liquid nitrogen temperature using the BET and BJH techniques. The surface areas and average pore diameter obtained for the catalysts and total acidity of the catalysts are shown in the table appended below as Table 1.

Table 1. Textural properties of catalysts

S.No.	**Catalyst**	**BET-surface area (m^2/g)**	**Pore volume (cm^3/g)**	**Average pore diameter (nm)**	**Total Acidity (mmol/g)^2**	**Low-Temperature Peak**	**High-Temperature Peak**
1	**rGO/ZrO_2/SO_4/Cu(5%)**	410.02	0.923	7.14	1.01	0.68	0.33
2	**rGO/ZrO_2/SO_4/Cu(10%)**	398.77	0.892	7.05	1.25	0.82	0.43
3	**rGO/ZrO_2/SO_4/Cu(15%)**	375.60	0.867	7.0	1.54	0.77	0.77
4	**rGO/ZrO_2/SO_4/Cu(20%)**	340.10	0.821	6.67	1.50	0.74	0.76

EXAMPLE 5

This example illustrates the experiments were carried out in a high-pressure stainless-steel reactor (fixed bed) with an inner diameter of 6 mm in a down-flow mode. 3.2 g of the catalyst (size 1-2

mm) was typically used, undiluted. The high-pressure fixed-bed microreactor was used in this investigation for selective oxidation of PG. The catalyst (size 1-2 mm) was taken and dried in CO_2-free air for 6 hours at 250°C. The reactor was then cooled for 1 hour at standard pressure and temperatures ranging from 60 to 110°C. When the airflow, temperature and pressure were tuned to the required values, the feed was then injected. The reactions occurred at temperatures between 60 and 100°C, under pressures of 10 bar, 3.2 g of catalyst loaded, and WHSV (h^{-1}).

EXAMPLE 6

This example states Catalytic activity and reaction parameter Optimization. Propylene glycol (PG)was selectively oxidized using a variety of rGO/ZrO_2(10%)/SO_4(4%)/Cu (Cu molar ratio = 5, 10, 15, and 20%) samples under continuous reaction conditions. By examining the impact of the variation in reaction factors, such as reaction temperature (60-100 °C), and reaction pressure, the influence of WHSV, solvent and time parameters were optimized to maximize Propylene glycol (PG) conversion and hydroxyacetone (HA) selectivity.

EXAMPLE 7

This example illustrates the influence of temperature on propylene glycol (PG) conversion and selectivity by (a) rGO/ZrO_2/SO_4/Cu (5%), (b) rGO/ZrO_2/SO_4/Cu (10%), (c) rGO/ZrO_2/SO_4/Cu (15%), and (d) rGO/ZrO_2/SO_4/Cu (20%) catalysts. In the temperature range of 60-100°C, liquid phase aerobic oxidation of propylene glycol was carried out over Cu supported on rGO/ZrO_2/SO_4 catalysts (Cu = 5,10,15, and 20%) in the continuous reactor. At temperatures between 60 to 100 °C the rGO/ZrO_2/SO_4/Cu(15) supported catalysts exhibit the highest selectivity when compared to the other catalysts. As a result, 90°C is the optimized reaction temperature for maximum oxidation of propylene glycol conversion and selectivity yields 90 to 100% in the continuous reactor

EXAMPLE 8

This example shows the selective oxidation conversions of propylene glycol across the various catalysts over a pressure range of 0 to 20 bar. As the pressure range of 0 to 20 bar was studied, the reactivity was observed in the continuous reactor. As a result, 10 bar CO_2 free air pressure would be sufficient to carry out the selective oxidation of propylene glycol given the higher conversion of 90 to 100% and selectivity of HA towards 95 to 100%.

EXAMPLE 9

For the selective oxidation of propylene glycol, the influence of WHSV was studied for conversion and selectivity in the various catalysts. The constant pressure of 10 bar and temperature of 90°Cas the change in the WHSV was investigated in the 0.5 to 2.0 h^{-1} range in the continuous reactor.Among the supported catalysts, the rGO/ZrO_2/SO_4/Cu (15%) catalyst is the most active when we used 10 bar pressure, 90°C temperature, and 1.0 h^{-1} WHSV. As a result, the WHSV = 1.0 h^{-1} is suggested for use in the propylene glycol conversion reaction.

EXAMPLE 10

The influence of process time on propylene glycol conversion and selectivity of hydroxyacetone is investigated for the catalysts (a) rGO/ZrO_2/SO_4/Cu (5%), (b) rGO/ZrO_2/SO_4/Cu (10%), (c) rGO/ZrO_2/SO_4/Cu (15%), and (d) rGO/ZrO_2/SO_4/Cu (20%) at the specified temperature of 90°C, WHSV of 1.0 h^{-1}, the pressure of 10 Bar, and O2 flow rate of 1.0 mol h^{-1} carried in a continuous reactor. The product's selectivity and conversion (%) changed very slowly during the period of 2 to 10 hours of reaction. Achieving the optimum reaction time to get the highest conversion of 90 to 100% and selectivity rate of HA was observed to be 4 hours.

EXAMPLE 11

This example shows the most effective rGO/ZrO_2(10%)/SO_4(4%)/Cu(15) catalyst were evaluated for recycling performance under optimum conditions. The reusability of the rGO/ZrO_2/SO_4/Cu (15) catalyst was examined. The propylene glycol conversion was in the range of 90–100% for the

seven consecutive runs tested. The hydroxyacetone selectivity reduces from 99.4% in the first run to 96.6% in the seventh run. Despite minor losses in conversion and selectivity during the seven consecutive runs, the rGO-supported $ZrO_2/SO_4/Cu$ (15%) catalyst appears to be an effective catalyst for the selective oxidation of propylene glycol, with a high hydroxyacetone yield (HA).

We claim:

1. A process for the single-step preparation of selective oxidation of propylene glycol into hydroxyacetone with high conversion and selectivity.
2. The process as claimed in claim 1 is used as a multifunctional bimetallic heterogeneous catalyst for the selective oxidation of propylene glycol.
3. The process as claimed in claims 1 and 2, wherein copper-infused sulfated zirconium oxide heterogeneous bimetallic nanoparticles supported on reduced graphene oxide as the catalyst.
4. The process as claimed in claim 3, wherein a variety of rGO/ZrO_2(10%)/SO_4(4%)/Cu (Cu molar ratio = 5, 10, 15, and 20%) is used for selective oxidation of PG.
5. The process as claimed in claims 3 and 4, wherein (a) rGO/ZrO_2/SO_4/Cu (5%), (b) rGO/ZrO_2/SO_4/Cu (10%), (c) rGO/ZrO_2/SO_4/Cu (15%), and (d) rGO/ZrO_2/SO_4/Cu (20%) various catalyst was synthesized.
6. The process as claimed in claim 1, where the catalytic process is carried out in the liquid phase selective oxidation.
7. The process as claimed in claims1, 5 and 6, wherein said the process is conducted in a high-pressured fixed bed Continuous reactor.
8. The process as claimed in claims6 and 7 are conducted in the presence of an oxygen flow rate of 1.0 mol h^{-1}.
9. The process as claimed in claims1 and7 are carried out at high pressures in the range of 0 to 20 bar.
10. The process as claimed in claims1 and 7 are conducted at temperatures between 60°C and 100°C.
11. The process as claimed in claims1 and 7, wherein the contacting of the feed is done in the presence of a solvent.

12. The process as claimed in claims1, 7 and 11, wherein the solvent is tertbutyl hydroperoxide (TBHP).
13. The process as claimed in claims 1 and 5, wherein the rGO/ZrO_2/SO_4/Cu (15%) catalysts exhibit the highest selectivity and conversion when compared to the other catalysts.
14. The process as claimed in claims 1 and 13, where the highest PG conversion was 90 to 100% and HA selectivity was 95 to100%.
15. The process as claimed in claims 1 and 13, utilizing rGO/ZrO_2/SO_4/Cu (15) as the catalyst in the Continuous reactor with optimum conditions of the high conversion rate of 90 to 100%.
16. The process as claimed in claims 1, 7 and 13, whereinthe liquid-phase oxidation of propylene glycol using TBHP as solvent.
17. The process as claimed in claims 1 and 15, wherein the presence of the rGO/ZrO_2/SO_4/Cu (15%), excellent reusability was attained even after seven consecutive cycles without any metal leaching or any reduction in catalytic activity in the Continuous reactor.

Date: 19-Apr-23

Signature:

Agent for Applicant

Ms. DIVYA SRIDHARAN,
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ABSTRACT

ZrO_2/SO_4/Cu nano particles supported on reduced graphene oxide for selective oxidation of propylene glycol in continuous reactor

The present invention relates to a process for the selective oxidation of propylene glycol into hydroxyacetone. By interacting in a continuous reactor with a catalyst composite made by depositing ZrO_2/SO_4/Cu nanoparticles supported on reduced graphene oxide with high surface area mesoporous material, it is feasible to generate directly upgradable products with the use of the catalyst. The use of this bimetallic heterogeneous catalyst results in 90 to 100%conversion and selectivity for the conversion of propylene glycol to hydroxyacetone (Acetol).

Fig. 1

Date: 19-Apr-23

Signature:

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