



International Journal of ChemTech Research

CODEN (USA): IJCRGG, ISSN: 0974-4290, ISSN(Online):2455-9555 Vol.10 No.7, pp 1067-1078, **2017**

Hydrogen Production from Glycerol: A Review

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Abstract: The development of alternative sources of energy is becoming important in this era of diminishing petroleum reserves and increased environmental awareness. Hydrogen production from biomass has attracted great interest because of the potential application in fuel cells. Significant amount of glycerol is produced as a by-product during bio-diesel production by transesterification of vegetable oils, which are available at low cost in large supply from renewable raw materials. With increased production of biodiesel, a glut of glycerol (C₃H₈O₃) is expected in the world market, and therefore it is essential to find useful applications for glycerol. Finding alternative uses for glycerol is important. Using glycerol as a source of producing hydrogen is a good possibility. Steam reforming is a promising way to utilize the diluted glycerol aqueous solution to produce hydrogen. Glycerin is a potential feed stock than ethanol for hydrogen production because one mole of glycerin can produce up to four moles of hydrogen. An attempt is made to identify new catalyst with high selectivity towards hydrogen and sufficient reaction rate at mild conditions. The catalysts used in other processes are studied and their effects on temperature, contact time, and water to glycerol ratio, metal loading on hydrogen yield are investigated. Other considerations such as catalyst activity, catalyst lifetime and operating conditions are estimated for over all process economics. Thus, the paper covers the selection of process and catalyst, process parameters, catalyst properties and economics related to it.

Keywords: Hydrogen production, biomass, transesterification, steam reforming, catalyst activity, catalyst properties, economics.

1. INTRODUTION:

This section deals with the introduction to glycerol, need of converting glycerol to hydrogen, steam reforming using catalyst.

Introduction to Glycerol:

Properties of Gylcerol:

- Glycerol is a simple polyol compound.
- It is a colourless, odourless, viscous liquid that is widely used in pharmaceutical formulations.
- Glycerol is present in fats and oils in combination with fatty acids, both saturated and unsaturated.

Chemistry:

Glycerol has three hydrophilic hydroxyl groups that are responsible for its solubility in water and its hygroscopic nature. The glycerol backbone is central to all lipids known as *triglycerides*. Glycerol is sweet-tasting and of low toxicity. The term polyglycerol refers to many glycerol molecules together.

Glycerol can be produced by saponification of fats, e.g. a byproduct of soap-making. Saponification is also referred to as the hydrolysis of esters in basic solutions or environments. The mechanism of this reaction involves a very rapid equilibrium where the alkoxide functions as a base deprotonating the carboxylic acid.

Fig. 1.1 Saponification reaction that produce Glycerol as byproduct

Fig. 1.2 Transesterification that produce Glycerol as byproduct

It also is a byproduct of the production of biodiesel via transesterification. Triglycerides (1) are reacted with an alcohol such as ethanol (2) with catalytic base to give ethyl esters of fatty acids (3) and glycerol (4).

Uses of Glycerol:

- Glycerin is used in food industry, Pharmaceutical and personal care applications, antifreeze.
- It is also used in production of various chemical intermediates like citric acid, lactic acid, 1,3-propane diol, acrolein, hydrogen, ethanol etc.

Need of Converting Gylcerol to Hydrogen:

Alternative energy resources are becoming increasingly important because of dwindling petroleum reserves and mounting environmental concerns that are associated with fossil fuel utilization. Consequently, alternative biobased fuels are emerging as the long-term solution. Over the last few years, the demand and production of biodiesel has increased tremendously. With the production of biodiesel, glycerol is being produced as a byproduct and several efforts are being made to utilize excess glycerol and produce valuable added products.

At present, almost 95% of the hydrogen is being produced from fossil fuel-based feed stocks and most is used as a chemical ingredient in petrochemical, metallurgical, goods and electronic processing industries. Demand for hydrogen, the simplest and most abundant element, is growing due to the technological advancements in fuel cell industry.

If the present scenario in the production of hydrogen exists, the more carbon will be converted in to carbon dioxide, leading to global climatic changes. Renewable resource-based technologies for hydrogen production are seen in viable options for the future due to the carbon neutral nature with lesser effects on global climate.

2. METHODS OF PRODUCTION OF HYDROGEN FROM GLYCEROL

Glycerin is a potential feed stock than ethanol for hydrogen production because one mole of glycerin can produce up to four moles of hydrogen. Various methods used for production of hydrogen from glycerin are

- Steam reforming by using catalyst
- Partial oxidation
- Aqueous phase reforming
- Supercritical water reforming

Steam Reforming Process using catalyst

Introduction:

The steam reforming is the most commonly used method for producing hydrogen in the chemical industry. In this process, the substrate is reacted with steam in the presence of a catalyst to produce hydrogen, carbon dioxide, and carbon monoxide. The steam reforming of hydrocarbons has been the preferred method for many decades for industrial scale hydrogen production.

Properties of Steam Reforming:

Steam reforming is a high temperature endothermic process which uses a catalyst to react the glycerol with water to produce hydrogen.

Chemical Reaction:

The overall reaction of hydrogen production by steam reforming of glycerol is as follows:

$$C_3H_8O_3(g) + 3H_2O(g) \rightarrow 7H_2(g) + 3CO_2(g) \Delta H^\circ = 128 \text{ kJ/mol}$$

Catalysts:

Many researchers investigated the effects of catalysts on the steam reforming of glycerol for hydrogen production. The various types of catalyst which are in use are Nickel-based catalysts with MgO, CeO₂, and TiO₂ supports. Besides nickel based catalysts, other type of catalyst like Ir, Co, Ru/Y₂O₃, Ir/CeO₂ are also used.

Adhikari et al. focused on the study of nickel-based catalyst with MgO, CeO₂, and TiO₂ supports. They found that maximum hydrogen yield that is, 4 mol of hydrogen out of 7 mol of stoichiometric maximum, could be obtained at 650 °C with MgO supported catalysts.

Irion do et al. modified alumina –supported nickel catalysts with Ce, Mg, Zr and La to produce hydrogen from glycerol. Among the ceria-supported metal catalysts examined, the Ir/CeO₂ catalyst showed promising catalytic performance with more than 85% hydrogen selectivity and 100% glycerol conversion at 400 °C.

Process Parameters:

Optimal conditions for production of hydrogen from glycerin were found to be as follows:

Temperature = 925-975 K

Water/glycerin ratio = 12

Pressure = 1 atmosphere

Desired output = The maximum number of 6.2 moles of hydrogen can be produced with one mole of glycerin.

Partial Oxidation

Introduction:

Partial oxidation is the process in which a substrate is reacted with oxygen at sub-stoichiometric ratios.

Reaction:

$$C_3H_8O_3 + 1.5(O_2) \rightarrow 3(CO_2) + 4(H_2)$$

$$\Delta H^{\circ} = -603 \text{ kJ/mol}$$

Dauenhauer *et al.* performed glycerol oxidation at different carbon-to-oxygen (C/O) ratios and temperatures using noble metal catalysts. Partial oxidation of glycerol at reactant steam-to-carbon ratio (S/C) = 0 achieved equilibrium H_2 selectivity for all C/O ratios <1.3 with a maximum of 56% at C/O = 1.2.

Autothermal Reforming

Introduction:

Autothermal reforming process combines the effect of partial oxidation and steam reforming by feeding fuel, air, and water together into the reactor.

Reaction:

$$C_3H_8O_3 + O_2 + H_2O \rightarrow 3(CO_2) + 5(H_2)$$

$$\Delta H^{\circ} = -269 \text{ kJ/mol}$$

Dauenhauer *et al.* found that the addition of steam at S/C = 4.5 increased the equilibrium H_2 selectivity by 30 - 40%. However, the observed H_2 selectivity from glycerol departed from equilibrium for C/O > 1.0, producing a maximum of 79% selectivity to H_2 . High selectivities to H_2 were achieved by adjusting the fuel/air and fuel/steam feed ratios.

Swami *et al.* conducted autothermal reforming at a steam-to-carbon ratio of 3 and an oxygen-to-carbon ratio of 0.3 in the range of 550-850 °C using the Pd/Ni/Cu/K catalysts. Their results showed that the hydrogen yield during autothermal reforming was greater than that of steam reforming, though the hydrogen yield for both reactions increased with temperature.

Douette *et al.* performed a series of tests using a factorial experimental design to determine the effects of the operating parameters, including C/O and S/C ratio, and temperature. A 4.5 mol quantity of hydrogen was produced per mole of reagent-grade glycerin at oxygen to carbon ratio of 0, S/C ratio of 2.2, and temperature of 804 °C. Initially, similar hydrogen yield was obtained when using crude glycerin, the by-product of biodiesel production. However, due to the catalyst deactivation and coke and deposit formation in the reformer, the hydrogen yield

decreased quickly. This may be correlated with the possible contaminants, chloride and sodium cations, present in crude glycerin.

Aqueous-Phase Reforming

Introduction:

The aqueous-phase reforming (APR) process was established by Cortright *et al.* using platinum based catalysts and mild reaction conditions (200-250 °C, 20-25 bar) for the production of hydrogen and light alkalines from oxygenated hydrocarbons.

Lehnert *et al.* used several platinum based catalysts for catalytic conversion of glycerol to hydrogen by APR. Their study indicated that the catalytic reaction selectivity to hydrogen increased with increasing particle size from 78% to 95% while the conversion of glycerol remained almost constant at 20%.

Furthermore, it was found that the mixture of γ -, δ -, and θ -phase can increase the hydrogen production to 7.6* $10^{\text{-3}}$ mol min⁻¹/g_{cat}, compared with pure γ -alumina (1.2 * $10^{\text{-3}}$ mol min⁻¹/g_{cat}). Crude glycerol can also be used as starting material. However, the rate of hydrogen production was lower than for pure glycerol due to the impurities, such as NaCl.

Reaction:

$$C_3H_8O_{3(1)} + 3(H_2O) \rightarrow 3(CO_2) + 7(H_2)$$

 $\Delta H^{\circ} = 123 \text{ kJ/mol}$

Supercritical Water Reforming

Introduction:

Supercritical water, defined as water that is heated and compressed at its critical temperature (374 °C) and pressure (22.1 MPa), is a promising medium for reforming of glycerol to produce hydrogen.

Byrd *et al.* selected Ru/Al₂O₃ as the catalyst to generate hydrogen from glycerol by supercritical water reforming. Their experiments revealed that hydrogen yields increased directly with temperature and near-theoretical yield of **7 mol of hydrogen/mol of glycerol** could be obtained at dilute feed concentrations at 800 °C. Also, it was found that up to 40 wt% glycerol in the starting material could be completely gasified.

3. FACTORS AFFECTING FORMATION OF HYDROGEN FROM GLYCEROL

- Glycerol concentration (steam to carbon molar ratio)
- Temperature of reaction
- Pressure
- Flow rates of glycerol feed
- Catalyst loading
- Water to glycerol ratio
- Space Time and Space Velocity

4. CATALYST USED IN THE PROCESS

Preparation of Catalyst:

Following methods are used to prepare catalysts

- Deposition-precipitation method
 - This method comprises application of active component onto an existing support via chemical reaction.
 - The reaction forms an insoluble compound which involves the active element.
 - The insoluble compound can be formed by an increase of the pH of the solution, a valence change of the element in question and the like.
 - Under certain conditions, such as a suitable interaction between the compound and the carrier, the preparation has a number of unique features, e.g. a uniform distribution of the active component over the carrier surface even at high loadings

• Wet impregnation method

- Impregnation as a means of supported catalyst preparation is achieved by filling the pores of a support with a solution of the metal salt from which the solvent is subsequently evaporated.
- The catalyst is prepared either by spraying the support with a solution of the metal compound or by adding the support material to a solution of a suitable metal salt, such that the required weight of the active component is incorporated into the support without the use of excess of solution.

Table 1. Catalysts and Operating Conditions for Steam Reforming of Glycerol

Catalysts and Operating Conditions used for STEAM RFEORMING of Glycerol			
Catalyst	Operating Conditions	Remarks	
Ir/CeO ₂ , Ni/CeO ₂ , Co/CeO ₂	Temperature = 400–550 °C Mass of catalyst : 200 mg Particle Diameter : 40–60 mesh	Ir/CeO ₂ gave the hydrogen selectivity and glycerol conversion of 85% and 100%, respectively, at 400 °C ²⁴	
	C3H8O3:H2O:He = 2:18:80 vol. %		
Y ₂ O ₃ , ZrO ₂ , CeO ₂ , La ₂ O ₃ , SiO ₂ , MgO, and Al ₂ O ₃ supported Group 8–10 metals.	Temperature = 500–600 C Steam-to-carbon molar ratio (S/C) = 3.3 Pressure = Atmospheric pressure	A complete conversion of glycerol was achieved over ruthenium on Y ₂ O ₃ (Ru/Y ₂ O ₃) at 600 °C. At similar conditions, hydrogen yield was 82.8%. From the list of the tested catalysts, Ru/Y ₂ O ₃ was found to be the best ²⁵	
Ni/Al ₂ O ₃	Temperature = 850 °C Steam-to-carbon ratio (S/C) = 2.1 and 2.6	The yield of hydrogen was 23.6 g/100 g of glycerol (77% of stoichiometric yield) at S/C of 2.6 ²⁶	

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Pt/Al ₂ O ₃ , Ni/Al ₂ O ₃ , Pd/Al ₂ O ₃ , Ru/ Al ₂ O ₃ , Rh/ Al ₂ O ₃	Feed flow rate = $0.15-0.5$ mL/min Temperature = $600-900$ °C Steam/carbon molar ratio (S/C) = $1/3-3.0$	About 80% of hydrogen selectivity was obtained with Ni/Al ₂ O ₃ , whereas the selectivity was 71% with Rh/CeO ₂ /Al ₂ O ₃ at a S/C = 3, 900 °C temperature, and feed flow rate of 0.15
Ni/MgO, Ni/CeO ₂ , Ni/TiO ₂	Feed flow rate = 0. 5–0.7 mL/min Temperatures = 550–650 °C	mL/min ²⁷ Ni/CeO ₂ , best performing catalyst than Ni/MgO and Ni/TiO ₂ under the experimental conditions. Ni/CeO ₂ gave the maximum hydrogen selectivity of
	Steam/carbon molar ratio (S/C)= 2–4 Catalyst loading = 0.75–1.5 g (Ni 9.6–12.7 wt.%)	74.7% at a S/C ratio of 4, temperature of 600 C, and a feed flow rate of 0.5 mL/min compared to Ni/MgO (38.6%) and Ni/TiO2 (28.3%) under similar conditions ²⁹
Pt/Al ₂ O ₃	Temperature = 880 °C Glycerol flow per kg catalyst = 0.12 mol/min S/C = 2.5	Hydrogen selectivity of 70% and glycerol conversion to gas 100%. ³⁰
Pt supported on Al ₂ O ₃ , ZrO ₂ , CeO ₂ /ZrO ₂ , MgO/ZrO ₂ , and Carbon	Temperature = 350 °C Pressure = 1 bar Aqueous glycerol feed solution (30 wt.%) over oxide supported Pt catalysts (1.0 g) or Pt/C catalyst (0.060 g) and a feed flow rate of 0.32 cm³min⁻¹. Pt/C catalyst was tested at various feed rates and temperatures. Other catalysts tested were Pt–Ru and Pt–Re	Pt/C catalysts showed the superior performance. At 400 C and pressure = 1 bar, 100% glycerol conversion was achieved at feed rate of 0.32 cm ³ min ^{-1 31}
Pd/Ni/Cu/K supported on c-Al ₂ O ₃	Temperature = $550-850$ °C S/C = 3.0	Hydrogen yield was 42% at 850 °C. 32
Ni/c-Al ₂ O ₃ modified with Mg, Ce, La, Zr.	Temperature = 600 °C Concentration = 1 wt.%	100% conversion was achieved with all the catalysts. Catalyst promoted with Zr showed the highest H2 selectivity similar to the equilibrium values. ²⁸

- An example of this technique is the coprecipitation of metal ions with aluminium ions to produce a precipitated alumina gel containing the metal hydroxide.
- This precipitate when calcined produces a refractory support with active component dispersed throughout the bulk as well as at the surface.

 However, in the preparation of multicomponent catalysts, it is possible under improper conditions to obtain a heterogeneous product because of the different solubility products of the constituents.

Combustion

- Combustion Synthesis, the exothermicity of the redox (reduction-oxidation or electron transfer) chemical reaction is used to produce useful materials.
- Depending upon the nature of reactants: elements or compounds (solid, liquid or gas); and the exothermicity (adiabatic temperature, *T*), Combustion Synthesis is described as:
 - ✓ Self-propagating high temperature synthesis (SHS)
 - ✓ Low-temperature combustion synthesis (LCS),
 - ✓ Solution combustion synthesis (SCS),
 - ✓ Gel-combustion, Sol–gel combustion
 - ✓ Emulsion combustion, volume combustion (thermal explosion), etc.

Properties of the catalyst to be characterized

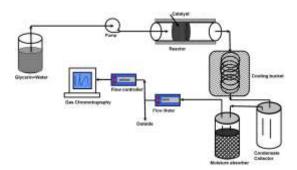
- Density
- Pore volume
- Pore size and size distribution
- Surface area
- Crushing strength
- Homogeneity
- Chemical structure
- Surface reactivity

Characterization of selected catalyst:

The following instruments are used to characterize various properties of catalyst

- Powder X-ray diffraction (XRD): X-ray diffraction (XRD), X-ray scattering techniques and computed tomography (CT) are versatile, non-destructive methods that reveal detailed information about the chemical composition, crystallographic and micro structure of all types of natural and manufactured materials.
- Micrometrics micro pore surface area analyser: surface area analysers rapidly and reliably produce accurate and repeatable surface area and porosity results. Their low cost, high speeds, simplicity of use, reliability, and ruggedness have earned them best option for surface area analyser.
- Catalyst scanning Electron micrograph: A micrograph or photomicrograph is a <u>photograph</u> or similar image taken through a <u>microscope</u> or similar device to show a magnified image of an item. A microphotograph is a very small picture, e.g. a <u>microdot</u>. Microphotography is the production of such very small photographs. Two important applications of microphotography were the creation of computer chips and the creation of microdots to hide information. An electron micrograph is a micrograph prepared using an electron microscope.

5. EXPERIMENTAL SET-UP^[23]



The experiments are carried out in a tubular furnace at a constant flow rate of 0.5 ml/min unless otherwise mentioned and at four furnace temperatures from 600 to 900 °C. The furnace temperatures could reach up to 1100 °C. Glycerin and water were mixed in a separate container and the mixture is supplied into the reactor using a HPLC pump. As depicted in fig. of experimental set up, the coated monoliths were placed in the middle of the tubular reactor. Monoliths were held at the center of the reactor with the help of alumina cloths. Molar concentration of glycerin and water is1:6, which is kept constant throughout the process.

Gas stream from the reactor was cooled using crushed ice and water. The un-reacted water and other liquids formed during the reaction were collected. The outlet gas was sent through a moisture trap before purging to the gas chromatograph. H₂ content in the outlet was analyzed by a thermal conductivity detector with Molecular Sieve column. The concentrations of carbon monoxide (CO), methane (CH₄), and CO₂ were analyzed by a flame ionization detector with HP-Plot Q column. Only four gases including H₂ were analyzed in this study.

The performance of the catalyst is presented in terms of H₂, CO, CH₄, and CO₂ selectivity, and glycerin conversion.

Selectivity and the glycerin conversion were calculated based on the following equations:

$$\%~H_2~selectivity = \frac{H_2~moles~produced}{C~atoms~produced~in~gas~phase} \times \frac{1}{RR} \times 100$$

where RR is H₂/CO₂ reforming ratio and it is 7/3 in the case of glycerin steam reforming process.

% Selectivity of
$$i = \frac{\text{C atoms in species } i}{\text{C atoms produced in gas phase}} \times 100$$

where species $i = CO, CO_2$, and CH_4 .

$$\% \text{ Conversion} = \frac{\text{C atoms in gas products}}{\text{Total C atoms in the feedstock}} \times 100$$

5. CONCLUSION

From whole review it can be concluded that hydrogen can effectively produce by partial oxidation (PO), steam reforming (SR), aqueous-phase reforming (APR), autothermal reforming (ATR), supercritical water reforming (SCWR) using glycerol as feed. Majorly steam reforming process is very efficient amongst all process because it can produce almost 4.8-6 moles of hydrogen. Although it has loop hole such as maintaining high temperature. A very few number of studies done with the help of autothermal reforming (ATR), supercritical water reforming (SCWR). Catalyst deactivation in ATR and SCWR was not discussed. Also hydrogen production from crude glycerol need to be study.

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