

CAT4BIO Conference: Advances in Catalysis for Biomass Valorization

Highlights of platinum group metal catalysts development for conversion of biomass to energy, fuels and other useful materials

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Introduction

The transformation of biomass into fuels and chemicals is becoming increasingly popular as a way to mitigate global warming and diversify energy sources. Catalysis will serve as key technological driver to achieve efficient and practical biomass conversion routes to useful products. As part of the satellite conferences complementing the 15th International Congress on Catalysis 2012 (held 1st–6th July 2012, in Munich, Germany), the Greek Catalysis Society organised CAT4BIO, an international conference on “Advances in Catalysis for Biomass Valorization”, that was successfully held in Thessaloniki, Greece, on 8th–11th July 2012 (1). The conference was held under the auspices of the Aristotle University of Thessaloniki (AUTH) and the Centre for Research and Technology Hellas (CERTH), with financial support from the Faculty of Engineering and the Department of Chemical Engineering at AUTH and the School of Engineering of the University of Patras. Industrial sponsors included the companies Arkema (France), BIO^eCON (The Netherlands) and Hellenic Petroleum (Greece).

The conference’s scientific programme covered the most recent progress in fundamental and applied catalysis research for the conversion of biomass. It consisted of eight keynote lectures from internationally renowned experts in the field, 36 high quality oral presentations and 95 posters from research groups worldwide. The programme was organised in nine sessions, structured around the following main topics:

- Conversion of cellulose/hemicellulose into platform molecules;
- Conversion of oils extracted from seeds and algae;
- Conversion of biomass into fuels and chemicals *via* thermochemical processes;
- Catalytic routes for lignin valorisation; and
- Upgrading of biomass-derived products to high added value fuels and chemicals.

Overall, there was excellent attendance with around 135 participants from both industry and academia from 28 countries worldwide. The conference succeeded in serving as a platform for the presentation of the most recent progress in fundamental and applied catalysis research for the conversion of biomass. Presenters shared their most up to date results on catalyst design, synthesis and characterisation, surface and catalytic reaction mechanisms and catalytic reaction processes in the area of biomass valorisation. The conference also provided ground for fruitful discussions among catalysis experts from industry and academia. Selected oral and poster contributions will be published as full papers in a special issue of *Applied Catalysis B: Environmental* (2).

This review focuses on the progress presented at the conference on platinum group metal (pgm) catalysts for the conversion of biomass to fuels and chemicals. The main bulk of the pgm work reported at CAT4BIO involved platinum catalysts, followed by papers on ruthenium, palladium and rhodium. The highlights of the pgm work presented in this review are categorised based on the type of reaction employed for the conversion of biomass and biomass-derived products to high added value fuels and chemicals.

Hydrolysis of Cellulose/Hemicellulose to Platform Chemicals

The catalytic conversion of cellulose to platform chemicals has gained increasing research attention in

the past decade. Noble metal catalysts can be used to achieve the one-pot synthesis of sorbitol from cellulose, however commercial application is hindered by the cost of the catalyst. The high stability of cellulose presents another problem, as the reaction requires harsh process conditions which degrade the final products and reduce selectivity. In a paper presented by Jorge Beltramini and coworkers (University of Queensland and Monash University, Australia), it was shown that small amounts of Pt promote nickel catalysts and significantly improve their catalytic activity. This synergistic effect was attributed to Pt and Ni particles in close vicinity. **Figure 1** shows sorbitol and mannitol yields, as well as cellulose conversion, from the aqueous phase hydrolysis and hydrogenation of cellulose using supported alumina and alumina nanofibre (Alnf) catalysts.

Hirokazu Kobayashi and colleagues (Hokkaido University, Japan) showed that cellulose can also be hydrolysed effectively to glucose by carbon-supported Ru catalysts. 2 wt% Ru supported on ordered mesoporous CMK-3 carbon gave a yield of 24% glucose and 16% cello-oligosaccharides at 503 K. The conversion of cellulose was 56%, and thereby the selectivity for glucose was 43%. The conversion of cellulose was slightly improved by increasing the content of Ru. This showed that the Ru species hydrolyse both cellulose and oligosaccharides, and show especially high activity for the latter substrate. Results from X-ray absorption fine structure (XAFS)

Keynote Lectures

Professor Enrique Iglesia (University of California at Berkeley, USA), **François Gault Lecture:** 'Monofunctional and Bifunctional C–C and C–O Bond Formation Pathways from Oxygenates'

Professor Johannes Lercher (Technical University of Munich, Germany), 'From Biomass to Kerosene – Tailored Fuels *via* Selective Catalysis'

Professor Daniel Resasco (University of Oklahoma, USA), 'Deoxygenation of Phenolics, Acids and Furfurals Derived from Biomass'

Professor Atsushi Fukuoka (Hokkaido University, Japan), 'Conversion of Cellulose into Sugar Compounds by Carbon-Based Catalysts'

Professor George Huber (University of Massachusetts, USA), 'Aqueous Phase Hydrodeoxygenation of Carbohydrates'

Jean Luc Dubois (ARKEMA, France), 'Added Value of Homogeneous, Heterogeneous and Enzymatic Catalysts in Biorefineries'

Paul O'Connor (BIO^eCON, The Netherlands), 'Catalytic Pathways towards Sustainable Biofuels'

Claire Courson (University of Strasbourg, France), 'Strategy to Improve Catalytic Efficiency for Both Thermal Conversion of Biomass, Tar Reduction and H₂S Absorption in a Fluidized Bed'

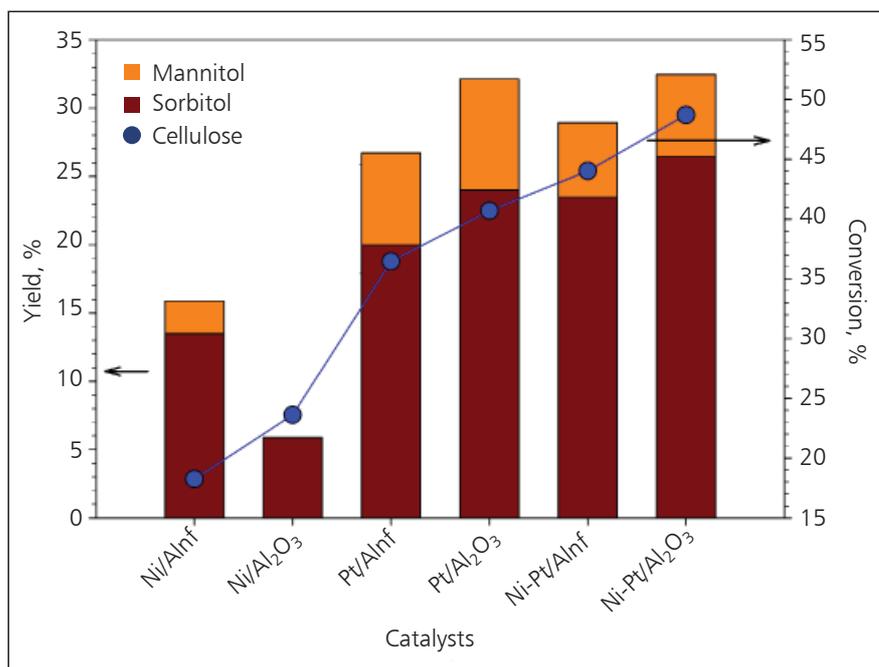


Fig. 1. Sorbitol and mannitol yield and cellulose conversion from the aqueous phase hydrolysis and hydrogenation of cellulose using supported platinum and nickel on alumina and alumina nanofibre (Almf) catalysts (Courtesy of Jorge Beltramini, University of Queensland, Australia)

analysis for the Ru catalyst were also presented. Characterisation showed that the Ru species on CMK-3 is not metal but $\text{RuO}_2 \cdot 2\text{H}_2\text{O}$ regardless of the hydrogen reduction in its preparation. Accordingly, one possible origin for the catalytic activity is that the Ru species works as Brønsted acid by the heterolysis of water molecules on Ru.

Hydrogen Production via Reforming of Biomass-Derived Products

A good number of contributions dealt with the use of pgms, mainly Pt, for the reforming of alcohols and other oxygenates from biomass to hydrogen. Leon Lefferts (University of Twente, The Netherlands) presented interesting results on the aqueous phase reforming of ethylene glycol in supercritical water over Pt-based catalysts. Ethylene glycol was investigated as a representative model compound for the aqueous phase of bio-oil, derived from biomass pyrolysis. Pt/Al₂O₃ and Pt-Ni/Al₂O₃ catalysts, although active in the reaction, were shown to deactivate rapidly with time on stream. Acetic acid, an intermediate of the reaction, was shown to be responsible for the deactivation of Pt and Pt-Ni catalysts. The presenter explained that acetic acid behaves as a strong acid in sub- and supercritical water resulting in hydroxylation of the Al₂O₃ surface. Redeposition of the dissolved Al₂O₃ on

the catalyst leads to blocking of catalytic Pt sites and hence deactivation of the catalyst, as observed with transmission electron microscopy (TEM) (Figure 2).

Taking their work one step further, the authors reported the development of stable Pt catalysts for ethylene glycol supercritical aqueous phase reforming supported on carbon nanotubes (CNTs). CNTs were found to be stable in hot compressed water. Moreover, the Pt/CNT catalysts exhibited stable activity for the reforming of both ethylene glycol and acetic acid, confirming that deactivation of Pt/Al₂O₃ is caused by the support and demonstrating the great importance of the type of support for reactions under supercritical conditions. The aqueous phase reforming of ethylene glycol and other polyols (glycerol and sorbitol) over Pt supported on hollow-type ordered mesoporous carbon (OMC) with three-dimensional (3D) pore structure was also reported in a poster contribution by Chul-Ung Kim and colleagues (Korean Research Institute of Chemical Technology, Korea). Better catalytic performance, including carbon conversion, hydrogen selectivity, yield and production rate was observed over these materials, implying that 3D interconnected mesopore systems allow faster pore diffusion of reactive molecules.

The steam reforming of bioethanol over Pt catalysts was discussed in two oral presentations at the

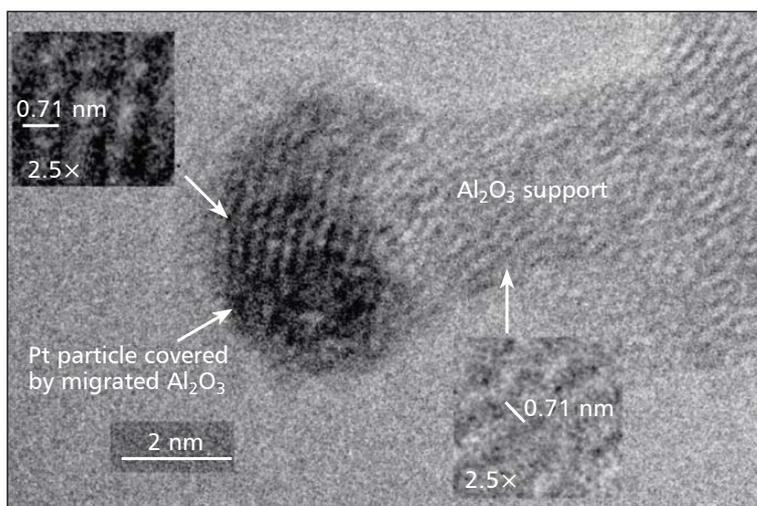


Fig. 2. TEM image of a deactivated platinum catalyst for the aqueous phase reforming of ethylene glycol in supercritical water (Reproduced from (3), Copyright 2012, with permission from Elsevier)

conference. An especially interesting contribution, reporting mechanistic aspects of the reaction over Pt, came from the group of Professor Xenophon Verykios (University of Patras, Greece). Paraskevi Panagiotopoulou (University of Patras, Greece) first presented a very systematic work on ethanol reforming over catalysts with different pgms (Pt, Pd, Rh and Ru) and different supports (zirconia, Al_2O_3 and ceria). Catalytic performance was found to depend strongly on the nature of the dispersed metallic phase employed, with Pt and Pd exhibiting good activity and selectivity towards hydrogen. However, the presenter showed that specific activity is defined primarily by metal crystallites and secondarily by metal/support interface. The normalised reaction rates were found to increase with decreasing perimeter of the metal/support interface and with increasing Pt crystallite size, implying that active sites are terrace sites and that ethanol adsorbs flat on the Pt surface. *In situ* diffuse reflectance infrared Fourier transform spectroscopy (DRIFTS) experiments also showed that the oxidation state of Pt seems to affect catalytic activity, which decreases with increasing population of adsorbed carbon monoxide (CO) species on partially oxidised ($\text{Pt}^{\delta+}$) sites. Moreover, the combined results of temperature programmed surface reaction (TPSR) and *in situ* DRIFTS experiments provided evidence that the key step for ethanol reforming at low temperatures is the ethanol dehydrogenation reaction, producing surface ethoxy species and subsequently acetaldehyde, which is further decomposed toward methane, hydrogen and carbon oxides.

Similar conclusions were also reported in the presentation of Filomena Castaldo *et al.* (University of Salerno, Italy) who investigated the ethanol reforming reaction over a 3 wt% Pt/10 wt% Ni/ CeO_2 catalyst. Investigation of the reaction pathway by kinetic experiments showed that ethanol steam reforming is probably not the reaction that actually occurs at 370°C. Instead, the involved reactions are most likely to be the following: ethanol dehydrogenation; ethanol and acetaldehyde decomposition and reforming; water gas shift reaction; and methanation. The same seems to apply for feedstocks other than ethanol, based on the work that was presented by Ricardo Reis Soares (Universidade Federal de Uberlândia, Brazil). This contribution reported results on glycerol reforming over Pt/C catalysts and also showed that dehydrogenation is the key limiting step of the reaction. Moreover, the reaction is sensitive to the structure of the Pt/C catalysts, with the activity decreasing and the selectivity shifting towards acetol and glycolaldehyde as particles decrease in size. In other words, C–O cleavage seems to occur preferentially on smaller particles.

In a poster contribution by Weijie Cai and Pilar Ramírez de la Piscina (University of Barcelona, Spain) and Narcís Homs (University of Barcelona and Catalonia Institute for Energy Research, Spain), the importance of pgms for the effective oxidative reforming of bio-butanol was reported. Doping of cobalt/zinc oxide catalysts with Rh, Ru and Pd significantly improved the catalytic performance and stability of the materials, with CoRh/ZnO exhibiting the

best catalytic performance in bio-butanol oxidative reforming.

Hydrodeoxygenation of Biomass and Biomass-Derived Products to Fuels and Chemicals

Currently, there is considerable interest in investigating the hydrodeoxygenation process, due to the high oxygen content of the feedstocks used for the production of renewable fuels. One of the main advantages of the hydrodeoxygenation route relative to other methods for making biomass-derived fuels is that the corresponding renewable fuel product is a high quality, oxygen free, hydrocarbon fuel which can be readily blended with conventional petroleum-based refinery fuel blendstocks and components.

Johannes Lercher (Technical University of Munich, Germany) reported exciting aspects of the deoxygenation of components from both proteinaceous biomass (grown in an aqueous environment) and lignocellulose (grown terrestrially) during his keynote lecture. Professor Lercher showed how detailed knowledge of the elementary reaction steps and of

the surface chemistry of the catalyst components in water allow suitable stable catalysts to be designed for the aqueous phase hydrodeoxygenation of biomass and biomass-derived components to alkanes. Results relevant to pgms were shown on the kinetics of the catalytic conversion of palmitic acid and intermediate products, 1-hexadecanol and palmityl palmitate. The impacts of the catalytically active metal (Pt, Pd or Ni) and the support (C, ZrO₂, Al₂O₃, silica, or the zeolites HBEA or HZSM-5), as well as the role of H₂, were explored in order to elucidate the reaction pathway. The speaker shared results on the conversion of palmitic acid at 260°C in the presence of H₂ for three monofunctional metal catalysts: Pt/C, Pd/C and Raney Ni (Table I). High selectivity to *n*-pentadecane was obtained on all three metals (70% on Ni; 98% on Pt and Pd), but relatively low conversions were attained on Pt and Pd at 31% and 20%, respectively. A high selectivity to lighter hydrocarbons (16%) through C–C bond hydrogenolysis together with a low selectivity to palmityl palmitate (4.6%) was observed over the Raney Ni catalyst. In a H₂ atmosphere, the direct decarboxylation or decarbonylation routes

Table I

Comparison of Palmitic Acid Conversion on Carbon- or Zirconia-Supported Metal Catalysts^a

Catalyst	Conversion, %	Selectivity, % ^b					Initial rate, mmol g ⁻¹ h ⁻¹
		C ₁₅	C ₁₆	A	B	C	
Raney Ni ^c	100	71	3.7	16	4.6	4.6	2.0
5% Pt/C	31	98	1.6	0.2	–	–	0.4
5% Pd/C	20	98	1.9	0.3	–	–	0.3
5% Ni/ZrO ₂	100	90	0.8	9.0	–	–	1.3
5% Pt/ZrO ₂	99	61	6.5	0.5	22	7.3	1.0
5% Pd/ZrO ₂	98	98	0.7	1.0	–	0.1	1.2

^a Reaction conditions: 1 g palmitic acid, 100 ml dodecane, 0.5 g catalyst, 260°C, 12 bar H₂ with a flow rate of 20 ml min⁻¹, 6 h

^b A: Lighter alkanes, B: 1-Hexadecanol, C: Palmityl palmitate

^c 0.25 g catalyst

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proceeded in parallel to the hydrogenation pathway. Direct decarboxylation and/or decarbonylation of fatty acids were the major pathways on carbon supported Pt or Pd, much faster than the hydrogenation of the fatty acid. However, the hydrogenation route took precedence over decarbonylation on the pure metallic Ni, as the decarbonylation on Ni was much slower than on Pt or Pd. When the support was changed from carbon to ZrO₂, the conversion increased from 20–30% to 100% for supported 5 wt% Pt and Pd catalysts under identical conditions, indicating that the hydrogenation of fatty acids was promoted by ZrO₂. High selectivity for *n*-pentadecane (98%) was observed on Pd/ZrO₂, while Pt/ZrO₂ led to a relatively low selectivity towards C15 alkanes (61%) due to the high concentrations of 1-hexadecanol (22%). Ni/ZrO₂ also led to 90% selectivity towards *n*-pentadecane at 100% conversion. These results imply that by using ZrO₂ as support, the three metals (Pt, Pd and Ni) varied the primary route from direct decarboxylation/decarbonylation to hydrogenation-decarbonylation, as large concentrations of alcohol intermediates were observed during the reactions. Thus, support aided hydrogenation became the primary route for reaction on these ZrO₂-based catalysts in H₂. The three metals, however, also led to different hydrogenolysis activities; for example, Pt/ZrO₂ or Pd/ZrO₂ produced less than 1% lighter alkanes, while Ni/ZrO₂ led to 9%, in line with the marked hydrogenolysis activity of Ni.

George Huber (University of Massachusetts, USA) gave a comprehensive keynote lecture on the aqueous phase hydrodeoxygenation of carbohydrates to produce a wide range of products including C1–C6 alkanes, C1–C6 primary and secondary alcohols, cyclic ether and polyols. The lecture focused on the hydrodeoxygenation of sorbitol, xylose and glucose, as well as pyrolysis oils, and discussed several aspects of the hydrodeoxygenation process, such as catalytic challenges, chemistry, kinetic modelling and reaction engineering. The reaction takes place over Pt bifunctional catalysts that involve both metal and acid sites. The presenter showed that three classes of reactions occur during the aqueous phase hydrodeoxygenation of carbohydrates: (a) C–C bond cleavage on metal sites; (b) C–O cleavage reaction on acid sites; and (c) hydrogenation on metal sites. **Figure 3** shows the rich reaction chemistry involved in aqueous phase hydrodeoxygenation of biomass derived oxygenates that according to the speaker can be further tuned by adjusting the relative reaction pathways through further catalyst design

and optimisation of reaction conditions. In terms of catalyst design, Professor Huber showed that the Pt metal sites and the acid sites can be atomically mixed (as in the case of a Pt-ReO₃/C catalyst) or atomically separate (as in the case of a platinum/zirconium phosphate catalyst). These differences in the catalyst properties result in the formation of different products. The product selectivity can be further adjusted by tuning the metal to acid site ratio. The type of acid sites is also important in this reaction to avoid undesired coking reactions.

Another excellent keynote lecture was delivered by Daniel Resasco (University of Oklahoma, USA) who focused on the deoxygenation of phenolics, acids and furfural derived from biomass to monofunctional compounds or hydrocarbons. Of interest to the progress of pgms in the area of biomass valorisation were the developed ruthenium/titania/carbon catalyst for the liquid phase conversion of acetic acid to acetone and the palladium-iron catalysts for the hydrogenation of furfural. The novel Ru/TiO₂/C catalyst proved to be very effective at temperatures much lower than typically needed for the reaction using existing catalysts. After detailed characterisation of the material, Professor Resasco proposed that the origins of this high activity are the oxygen vacancies and the Ti³⁺ sites which are promoted by the presence of Ru. Moreover, the hydrophobicity of the carbon support is believed to decelerate the inhibiting effect that water typically has on catalysts with hydrophilic surfaces. Concerning furfural conversion, the presenter showed that whereas Pd is active for the decarbonylation of furfural to furan and methylfuran, by alloying Fe with Pd a dramatic change in selectivity occurs. It seems that hydrogenolysis of the C–O bond is favoured on Pd-Fe alloys, whereas on Pd the preferred reaction is C–C bond breakage. Selectivity to methylfuran was found to be a strong function of the degree of Pd-Fe alloying. The extent of Pd-Fe interaction also strongly depended on the type of support (SiO₂ > α-Al₂O₃ > γ-Al₂O₃).

The pgm catalysts were also reported to be active for the hydrodeoxygenation of fatty acids to renewable diesel fuel. In an oral contribution from Bartosz Rozmysłowicz (Åbo Akademi University, Finland), Pd/C was shown to be an effective catalyst for the deoxygenation of algae oil, tall oil fatty acids and macauba oil, which were chosen as representative renewable oils of different origins (algae, wood and fruits). The presented work also included a set of kinetic experiments which revealed the reaction pathway over

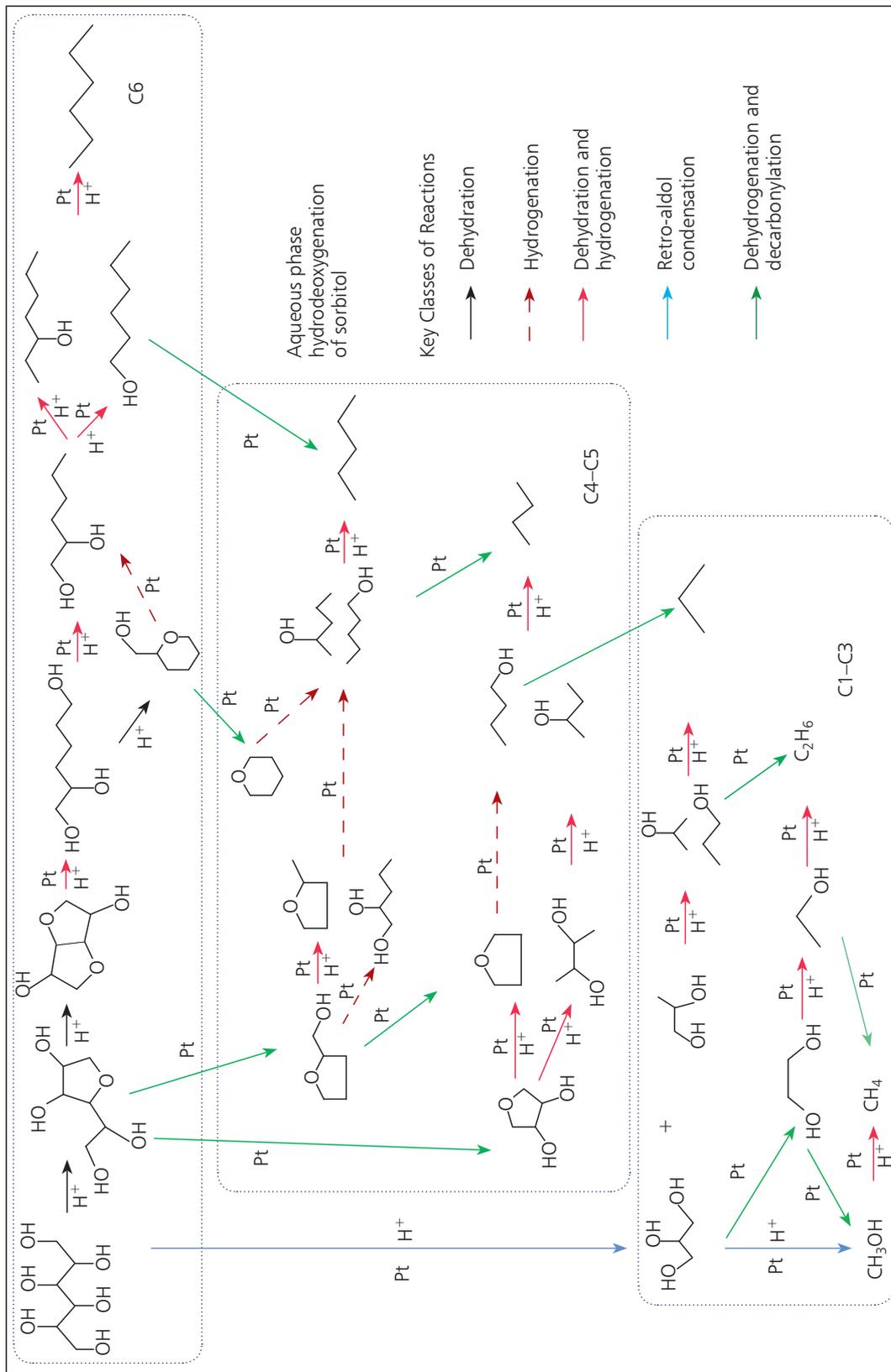


Fig. 3. Reaction pathways for the hydrodeoxygenation of sorbitol over a platinum bifunctional catalyst (Courtesy of George Huber, University of Massachusetts, USA)

Pd/C. The results showed that the catalyst deactivates in a low hydrogen atmosphere due to unsaturation of the feedstock. Moreover, deactivation is related to feedstock purity and its production technology.

Worth mentioning is an interesting poster contribution by the group of Regina Palkovits (RWTH Aachen University, Germany) which demonstrated the feasibility of using a heterogeneous Ru catalyst to convert levulinic acid (LA), a versatile intermediate that can be obtained directly from cellulose, to γ -valerolactone (γ -VL), a compound that can be utilised directly as a fuel additive. The contribution investigated the effect of different supports (carbon, TiO₂, SiO₂ and Al₂O₃) on 5 wt% Ru. Catalyst screening demonstrated that variation of the catalyst support can have a profound influence on the reaction outcome. The Ru/C catalyst exhibited the highest γ -VL yield (89.1%) when reacted with LA at 130°C in an ethanol/water solvent mixture.

Hydrogenolysis of Glycerol to Chemicals

Glycerol, a byproduct of biodiesel production, can be converted by hydrogenolysis to different high value added chemicals, such as 1,2-propanediol (1,2-PDO) and 1,3-propanediol (1,3-PDO), which are promising targets because of the high production cost using conventional processes and the reasonably large production scale. The production of propanediols from glycerol, however, normally requires the use of organic solvents and high hydrogen pressures. Two contributions presented novel results on the hydrogenolysis of glycerol to 1,2-PDO and 1,3-PDO over Pd- and Pt-containing catalysts without the need for externally added hydrogen. Gustavo Fuentes (Universidad A. Metropolitana Iztapalapa, Mexico) showed that it is possible to obtain 1,3-PDO and 1-hydroxyacetone with significant selectivity (30% and 46%, respectively at 220°C) without the addition of external hydrogen and without the production of appreciable amounts of ethylene glycol, the main degradation product in basic medium, over a copper-palladium/titania-5% sodium catalyst. It is important to note that the highest selectivity reported so far for 1,3-PDO using hydrogen pressure is 34%, a value comparable to the authors' results.

Another approach to alleviate the need for an external hydrogen supply is the *in situ* formation and consecutive consumption of H₂, either by using a part of the glycerol *via* a reforming reaction or by adding a hydrogen donor molecule *via* dehydrogenation.

Efterpi Vasiliadou and Angeliki Lemonidou (Aristotle University of Thessaloniki, Greece) presented a novel one-pot catalytic route for efficient 1,2-PDO production using a crude glycerol stream as a feedstock under inert conditions in the presence of Pt-based catalysts (Pt/SiO₂ and Pt/Al₂O₃). A European patent application has been filed (5). The H₂ needed for glycerol conversion was formed *via* a methanol reforming-glycerol hydrogenolysis cycle taking advantage of the unreacted methanol after biodiesel production through transesterification. The use of a Pt/SiO₂ catalyst results in satisfactory 1,2-PDO yields (~22%) at 250°C, 3.5 MPa nitrogen and 4 h reaction time.

Concluding Remarks

The conversion of biomass and biomass-derived compounds to platform molecules and high added value fuels and chemicals is a dynamic area of research, which holds the attention of numerous research groups worldwide. It is clear that catalysis plays a key role in achieving efficient and practical biomass conversion routes to useful products. The CAT4BIO conference on "Advances in Catalysis for Biomass Valorization" was a successful event, where groups from all over the world presented the most recent progress in fundamental and applied catalysis research for the conversion of biomass. As demonstrated in the conference, pgm constitute essential components of catalysis research for biomass conversion reactions. Either as main active components or as promoters, pgms find use in a wide range of chemical reactions. Their impact will render them an essential component of future catalytic processes for biomass valorisation.

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