

Editorial

# Rational Design of Non-Precious Metal Oxide Catalysts by Means of Advanced Synthetic and Promotional Routes

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**Citation:** Konsolakis, M.; Stathopoulos, V.N. Rational Design of Non-Precious Metal Oxide Catalysts by Means of Advanced Synthetic and Promotional Routes. *Catalysts* **2021**, *11*, 895. <https://doi.org/10.3390/catal11080895>

Received: 22 July 2021

Accepted: 23 July 2021

Published: 24 July 2021

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## 1. Background

Catalysis is an indispensable part of our society, involved in numerous energy and environmental applications, such as the production of value-added chemicals/fuels, hydrocarbons processing, fuel cells applications, abatement of hazardous pollutants, among others. Although, noble metals (NMs)-based catalysts are traditionally employed in various processes, due to their peculiar characteristics and enhanced reactivity, their scarcity and consequently high cost renders them disincentive for practical applications. In this perspective, the rational design and development of earth-abundant NMs-free metal oxides of adequate activity, selectivity and durability constitutes one of the main research pillars in heterogeneous catalysis [1–6]. Towards this direction, however, one crucial question must be answered: Is it possible to fine-tune the local surface chemistry/structure of a single, binary or multicomponent metal oxide in order to be highly efficient-like NMs-in a specific process? Thanks to the huge research progress so far achieved in the fields of (nano) materials synthesis, catalyst tailoring/promotion and surface science, the answer to the aforementioned question is certainly yes.

In specific, the catalytic performance of metal oxides, such as transition metals (TMs)-based mixed metal oxides, spinels, perovskites, hexaaluminates and hydrotalcites can be considerably improved by tailoring the local surface chemistry/structure (e.g., work function, reducibility, oxygen vacancies) and interfacial interactions. The latter can be accomplished by the employment of state-of-the-art nano-synthesis routes towards engineering particle's size and shape (e.g., nanocubes, nanorods) in conjunction to the use of appropriate modifiers (e.g., alkali, graphene oxide) and special pretreatment protocols. This holistic approach can exert a profound influence not only to the surface reactivity of metal sites in its own right, but also to metal-support interfacial activity, offering highly active and stable materials for real-life energy and environmental applications, such as the CO oxidation [7–12], N<sub>2</sub>O decomposition [13–15], CO<sub>2</sub> hydrogenation to value-added products [16–20], degradation of organic contaminants [21–33], etc.

## 2. This Special Issue

In light of the above aspects, the present Special Issue is mainly focused on the fabrication and fine-tuning of NMs-free metal oxide catalysts by means of advanced synthetic and/or promotional routes. It consists of fourteen high quality papers, involving: a comprehensive review article on the recent advances on the rational design and fine-tuning of ceria-based metal oxide catalysts [5]; two articles on the ceria nanoparticles morphological effects on N<sub>2</sub>O decomposition [14] and CO oxidation [10] over ceria-based binary oxides; two articles on NO decomposition over K-promoted Co-Mn-Al mixed oxides [34,35]; one article on the effect of ceria synthesis methods on the carbon pathways in dry reforming of methane (DRM) over Ni/CeO<sub>2</sub> catalysts [36]; one article on the impact of synthesis procedure and aliovalent doping in Co-Al spinel-type oxides for lean methane combustion [37];

one article on the support effect on the direct conversion of syngas to higher alcohols over copper-based oxides [38]; one article on nanofibrous Ni/Al<sub>2</sub>O<sub>3</sub> catalysts prepared by electrospinning for methane partial oxidation [39]; one article on the influence of precursor compounds on the selective catalytic reduction (SCR) of NO<sub>x</sub> by NH<sub>3</sub> over FeMgO<sub>x</sub> oxides [40]; one article on the synthesis and active sites determination of ZrO<sub>2</sub>-supported WO<sub>x</sub> solid acid catalysts [41]; one article on the synthesis and electrocatalytic performance of RuO<sub>2</sub> nanoparticles [42]; one article on the fabrication and photocatalytic performance of mesoporous frameworks of ZnFe<sub>2</sub>O<sub>4</sub> (ZFO) and MnFe<sub>2</sub>O<sub>4</sub> (MFO) nanoparticles [43]; and one article on the synthesis of polymer supported catalysts for arylamination reaction [44].

### *Contribution Highlights*

The comprehensive review of M. Konsolakis and M. Lykaki [5] addresses the latest experimental and theoretical advances in the field of the rational design of metal oxide catalysts exemplified by CuO<sub>x</sub>/CeO<sub>2</sub> binary system. In particular, it summarizes the general optimization framework that could be followed to fine-tune metal oxide sites and their surrounding environment by means of appropriate synthetic and promotional/modification routes. It was clearly revealed that the modulation of size, shape and electronic state at nanoscale can exert a profound influence not only to the reactivity of metal sites in its own right, but also to metal-support interfacial activity, offering cost-effective and highly active materials for real-life energy and environmental applications.

In view of above aspects, the impact of ceria morphology (nanorods, nanocubes, nanopolyhedra) on the physicochemical properties and the catalytic performance of ceria-based transition metal catalysts was explored by M. Konsolakis and co-workers [10,14]. It was shown that Co<sub>3</sub>O<sub>4</sub>/CeO<sub>2</sub> of rod-like morphology exhibited the optimum N<sub>2</sub>O decomposition performance as compared to other distinct morphologies, due to its abundance in Co<sup>2+</sup> active sites and Ce<sup>3+</sup> species in conjunction to its improved reducibility, oxygen kinetics and surface area [14]. Similar conclusions were derived for Fe<sub>2</sub>O<sub>3</sub>/CeO<sub>2</sub> catalysts for CO oxidation [10]; the rod-shaped sample exhibited the optimum catalytic performance, due to its improved reducibility and abundance in Fe<sup>2+</sup> species [10]. These findings unambiguously revealed the key role of support morphology towards determining the redox properties and in turn the catalytic performance of reactions following a redox-type mechanism [5,10,14].

Lucie Obalová and co-workers [34,35] systematically explored the impact of preparation parameters and alkali doping on the direct NO decomposition of K-promoted Co-Mn-Al mixed oxides. It was shown that preparation procedure notably affects the physicochemical properties and alkali distribution/stability with great consequences in NO decomposition. Specifically, it was revealed that the presence of potassium promoter notably improves the basicity and reducibility of the catalysts, positively affecting the catalytic activity. However, the calcination time/temperature notably affects the textural characteristics and alkali metal valorization process. The best catalytic performance was achieved for a potassium loading of ca. 1.0 wt.% at a calcination temperature of 700–800 °C. These results clearly revealed the importance of pretreatment conditions in conjunction to surface promotion towards the development of highly active metal oxides.

A.M. Efstathiou and co-workers [36] elegantly designed and conducted transient and isotopic studies to gain insight into the impact of CeO<sub>2</sub> preparation method on the carbon pathways in the dry reforming of methane (DRM) of Ni/CeO<sub>2</sub> catalysts. Among the different preparation methods explored, precipitation led to the lowest amount of carbon deposition. By means of various transient and isotopic studies, it was shown that a large pool of oxygen over precipitated catalysts contributed to the gasification of carbon formed in DRM towards the formation of CO, thus offering an important path for carbon removal.

The impact of different synthetic/modification routes towards enhancing the lean methane combustion of Co<sub>3</sub>O<sub>4</sub>/Al<sub>2</sub>O<sub>3</sub> spinel-type oxides was investigated by Rubén López-Fonseca and co-workers [37]. Three different strategies for enhancing the performance of alumina-supported catalysts were examined: (i) surface protection of the alumina with

magnesia prior to the deposition of the cobalt precursor, (ii) coprecipitation of cobalt along with nickel and (iii) surface protection of alumina with ceria. The optimum performance was obtained by the addition of ceria to alumina prior to the deposition of cobalt, which was attributed to the abundance of  $\text{Co}^{3+}$  species and oxygen vacancies due to the insertion of  $\text{Ce}^{4+}$  ions into the spinel lattice.

X. Li et al. [38] reported on the impact of support nature ( $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ ) and alkali promotion (K) on the synthesis of higher alcohols from CO hydrogenation over Cu-based catalysts. Significant differences on CO conversion and product's selectivity were revealed, attributed to support- and alkali-induced effects on redox and electronic properties.

D. Dong and co-workers [39] investigated nanofibrous Ni/ $\text{Al}_2\text{O}_3$  catalysts prepared by electrospinning for methane partial oxidation. The impact of different synthesis parameters, such as metal precursor, metal content and calcination temperature were explored. It was shown that by appropriately adjusting the aforementioned parameters highly active and stable catalysts can be obtained.

In a similar manner, L. Xu et al. [40] explored the influence of precursor compounds on the selective catalytic reduction (SCR) of  $\text{NO}_x$  with  $\text{NH}_3$  over Ti-modified  $\text{FeMgO}_x$  oxides. The key role of precursors towards determining the surface acidity and redox properties and in turn the catalytic performance, was clearly demonstrated. The catalysts derived from  $\text{FeSO}_4$  and  $\text{Mg}(\text{NO}_3)_2$  precursors exhibited enhanced catalytic activity in the temperature range of ca. 200–400 °C, offering complete  $\text{NO}_x$  conversion.

R.J. Gorte and co-workers [41] explored thoroughly the reactive sites in  $\text{WO}_x/\text{ZrO}_2$  catalysts prepared by atomic layer deposition (ALD). By a comparison with a  $\text{WO}_x/\text{ZrO}_2$  catalyst prepared via conventional impregnation and by employing surface and microscopy techniques three types of catalytic sites were identified, with their concentration varied with the number of ALD cycles. Dehydrogenation sites are associated with  $\text{ZrO}_2$ , Brønsted-acid sites with monolayer  $\text{WO}_x$  clusters, while oxidation sites are associated with the  $\text{WO}_x$  coverage. Such surface chemistry differentiation with the preparation process notably affects acid catalyzed reactions, such as 2-propanol catalytic dehydration.

R. Phul et al. [42] reported on a simple wet chemical route to synthesize ultrafine  $\text{RuO}_2$  nanoparticles at controlled temperature as electrocatalysts for oxygen evolution reaction (OER) and oxygen reduction reaction (ORR). These  $\text{RuO}_2$  nanoparticles exhibited enhanced bifunctional electrocatalytic performance under different conditions (air,  $\text{N}_2$  and  $\text{O}_2$  atmosphere), showing excellent potential for electrocatalytic applications. In addition,  $\text{RuO}_2$  nanoparticles showed efficient sensing properties rendering them as active nonenzymatic electrochemical sensors for the selective detection of  $\text{H}_2\text{O}_2$ .

The group of G.S. Armatas [43] reported on the preparation of high-surface-area dual component mesoporous frameworks of spinel ferrite  $\text{ZnFe}_2\text{O}_4$  (ZFO) and  $\text{MnFe}_2\text{O}_4$  (MFO) nanoparticles with improved photochemical activity. These mesoporous nanomaterials were synthesized via a polymer-assisted method that allowed the efficient co-assembly of the spinel ferrite colloidal nanoparticles and amphiphilic block-copolymer aggregates. The MFO-ZFO composite materials exhibit excellent performance for photocatalytic reduction of Cr(VI) in aqueous solutions with coexisting organic pollutants (such as phenol, citric acid and EDTA), under UV-vis light irradiation. The enhanced photocatalytic activity of dual component MFO-ZFO mesoporous networks is originated from the combined effect of accessible pore structure, which permits facile diffusion of reactants and products and suitable electronic band structure, which efficiently separates and transports the charge carriers through the ZFO/MFO interface.

B. Chumadathil Pookunoth et al. [44] reported on the immobilization of a 1,3-bis(benzimidazolyl) benzeneCo(II) complex on divinylbenzene cross-linked chloromethylated polystyrene, as an inexpensive polymer matrix. This particular system was tested on the arylamination reaction and showed robustness in the preparation of bioactive adamantanyl-tethered-biphenylamines. Such transition metal-catalyzed cross-coupling reactions between aryl halides and primary/secondary amines to obtain aminated aryl

compounds are of particular importance due to the wide field of arylamines applications in the chemicals and pharmaceuticals.

In summary, the aforementioned special issue highlights through the fourteen novel contributions the ongoing importance of the rational design of metal oxide catalysts by means of appropriate synthesis and/or modification routes. It was clearly revealed that the fine-tuning of size, shape and electronic state through appropriate synthetic methods, special pretreatment protocols and surface/structural modification can exert a profound influence on metal's sites reactivity/stability, offering highly active and stable composites for real-life applications.

We are very pleased to serve as Guest Editors on this thematic issue involving fourteen high quality studies. In this regard, we would like to express our gratitude to editorial staff of *Catalysts*, particularly to Assistant Editor, Mrs. Adela Liao, for her efforts and continuous support. Moreover, we are most appreciative to all authors for their contributions and hard work in revising them as well as to all reviewers for their valuable recommendations that assisted authors to upgrade their work to meet high standards of *Catalysts*. We hope that this special issue will be a valuable resource for researchers, students and practitioners, to promote and advance research and applications in the field of the rational design and fabrication of cost-efficient and highly active nano-structured catalysts for energy and environmental applications.

**Funding:** This research has been co-financed by the European Union and Greek national funds through the Operational Program Competitiveness, Entrepreneurship and Innovation, under the call RESEARCH-CREATE-INNOVATE (project code: T1EDK-00094).

**Conflicts of Interest:** The authors declare no conflict of interest.

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