

# New Advances into Nanostructured Oxides, 2nd Edition

Silvia Mostoni  and Roberto Nisticò \* 

Department of Materials Science, INSTM, University of Milano-Bicocca, U5, Via R. Cozzi 55, 20125 Milano, Italy; silvia.mostoni@unimib.it

\* Correspondence: roberto.nistico@unimib.it; Tel.: +39-02-6448-5111

## 1. Introduction

The interest in inorganic nanostructured oxides is growing extensively, thanks to their remarkable features and their wide range of applications, which include (photo)catalysis [1–4], controlled drug-delivery and chemical release [5–7], environmental remediation [8–10], energy and batteries [11], smart materials [12], and so on. One interesting aspect of inorganic nanostructured oxides is the high level of control that can be achieved over particle morphology, size, shape, and porosity [13], as well as their surface properties [14,15], which further broaden the possible application of these materials in a wide variety of fields. In fact, the use of suitable synthetic pathways and ad hoc surface functionalization procedures provides a powerful tool to obtain pure inorganic or hybrid inorganic–organic composite materials that represent strategic materials in the most recent research literature [16,17].

In this context, after the success of the first edition published in *Inorganics* in 2022, the second edition of the Special Issue, entitled “New Advances into Nanostructured Oxides, 2nd Edition” was launched to bring together the most recent developments on the class of inorganic materials. This Special Issue is inserted in the “Inorganic Materials” section that, since its birth, has rapidly grown thanks to its attention to advanced inorganic materials, as well as their high technological demand. The aim was to collect research papers and reviews focused on the synthesis and characterization of inorganic oxide nanomaterials through soft chemistry approaches, favoring a high control on the particle morphology, size, porosity and surface functionalities. In addition, in view of their application, the materials’ performances are described with the ambitious goal of identifying the structure–property relations to connect the surface, morphological and structural features to the material activity. This represents a shared interest in the development of innovative materials, as the modulation of the key structural parameters may be used to tune and improve the materials’ resulting performances.

Prior to proceeding with the overview of the contributions, the Guest Editors would like to thank all of the reviewers who spent their valuable time thoroughly reviewing and improving the articles published in this volume. We also sincerely thank all the authors for choosing *Inorganics*, and, in particular, the “Inorganic Materials” section, as the recipient of their excellent science.

## 2. An Overview of the Published Articles

Overall, this Special Issue collected 10 original papers (i.e., seven research articles and three reviews) and received more than 16,000 views, which paved the way for the further proposal of a third edition of the same Special Issue. The published papers can be categorized into three main subsections as reported in Table 1, including: (i) environmental



Received: 6 February 2025

Revised: 7 February 2025

Accepted: 13 February 2025

Published: 16 February 2025

**Citation:** Mostoni, S.; Nisticò, R. New Advances into Nanostructured Oxides, 2nd Edition. *Inorganics* **2025**, *13*, 60. <https://doi.org/10.3390/inorganics13020060>

**Copyright:** © 2025 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (<https://creativecommons.org/licenses/by/4.0/>).

remediation, (ii) development and optimization of new synthetic routes for metal oxides, and (iii) drug-delivery and biomedicine. This classification was made considering both the materials applications and the applied synthetic routes for the preparation of the materials.

**Table 1.** Correlation between subsections and contributions collected in the present Special Issue.

Subsections	Title	References
Environmental remediation	“Efficient catalytic reduction of organic pollutants using nanostructured CuO/TiO <sub>2</sub> catalysts: synthesis, characterization, and reusability”	[18]
	“Core/shell ZnO/TiO <sub>2</sub> , SiO <sub>2</sub> /TiO <sub>2</sub> , Al <sub>2</sub> O <sub>3</sub> /TiO <sub>2</sub> , and Al <sub>1.9</sub> Co <sub>0.1</sub> O <sub>3</sub> /TiO <sub>2</sub> nanoparticles for the photodecomposition of Brilliant Blue E-4BA”	[19]
	“Significantly enhanced self-cleaning capability in anatase TiO <sub>2</sub> for the bleaching of organic dyes and glazes”	[20]
	“Research progress of TiO <sub>2</sub> modification and photodegradation of organic pollutants”	[21]
	“Morphological dependence of metal oxide photocatalysts for dye degradation”	[22]
Development and optimization of new synthetic routes for metal oxides	“The influence of annealing temperature on the microstructure and electrical properties of sputtered ZnO thin films”	[23]
	“Synthesis and redox properties of iron and iron oxide nanoparticles obtained by exsolution from perovskite ferrites promoted by auxiliary reactions”	[24]
	“Mesoporous titania nanoparticles for a high-end valorization of <i>Vitis vinifera</i> grape marc extracts”	[25]
Drug-delivery and biomedicine	“Precipitative coating of calcium phosphate on microporous silica–titania hybrid particles in simulated body fluid”	[26]
	“The story, properties and applications of bioactive glass “1d”: from concept to early clinical trials”	[27]

### 2.1. Environmental Remediation

A large portion of the contributions reported in this Special Issue is focused on the exploitation of inorganic nanomaterials (mainly TiO<sub>2</sub>) for environmental remediation. In fact, the urgency of solving environmental pollution caused by the anthropogenic activities both in gaseous and liquid phases has driven research interest from more than a decade [28–30]. In this field, the use of photocatalysis is one of the most studied techniques to reduce the concentration of organic pollutants in wastewater, by using inorganic nanomaterials as efficient photo-catalysts. At the same time, organic pollutants can be degraded by using other catalytic reactions, such as catalytic reduction reactions in the presence of suitable nanostructured catalysts. In this first section, three research papers and two reviews were published [18–22].

Abouri et al. [18] reported the synthesis of nanostructured CuO/TiO<sub>2</sub> catalysts via a combustion technique, followed by calcination at 700 °C to achieve a rutile-phase TiO<sub>2</sub> structure with varying copper loadings (in the 5–40 wt.% range). Tests were performed aiming to reduce 4-Nitrophenol and Methyl Orange as target molecules with sodium borohydride (NaBH<sub>4</sub>) in the presence of the CuO/TiO<sub>2</sub> catalysts. Results indicated a 98% reduction of 4-Nitrophenol in 480 s and 98% reduction of Methyl Orange in 420 s. The catalysts exhibited high stability over 10 reuse cycles, maintaining over 96% efficiency for Methyl Orange and 94% efficiency for 4-Nitrophenol.

Dolatyari et al. [19] reported the synthesis of core/shell ZnO/TiO<sub>2</sub>, SiO<sub>2</sub>/TiO<sub>2</sub>, and Al<sub>2</sub>O<sub>3</sub>/TiO<sub>2</sub> nanoparticles using ethylene glycol for governing the nanoparticle size, fol-

lowed by calcination at 400 °C to decompose the organic residues at the nanoparticles' surface. Furthermore, Cobalt ( $\text{Co}^{3+}$ ) was added during the  $\text{Al}_2\text{O}_3$  nanoparticle synthesis to form Co-doped nanoparticles with formula  $\text{Al}_{1.9}\text{Co}_{0.1}\text{O}_3/\text{TiO}_2$ . All of the synthesized nanoparticles were tested for the photocatalytic degradation of Brilliant Blue E-4BA under UV and visible light irradiation. Photocatalytic tests revealed that both  $\text{Al}_2\text{O}_3/\text{TiO}_2$  and  $\text{Al}_{1.9}\text{Co}_{0.1}\text{O}_3/\text{TiO}_2$  showed superior degradation under UV and visible light compared to  $\text{ZnO}/\text{TiO}_2$  and  $\text{SiO}_2/\text{TiO}_2$  with complete photodecomposition of the target dye (20 ppm) in only 20 min using a 10 mg of photocatalyst. Furthermore, the "Co-doped"  $\text{Al}_{1.9}\text{Co}_{0.1}\text{O}_3/\text{TiO}_2$  nanoparticles showed the best performance under visible light irradiation, due to the increased absorption in the visible range as Co-doping introduces additional energy levels into  $\text{Al}_2\text{O}_3$ , resulting in improved electron–hole pair generation.

Zhao et al. [20] reported the hydro/solvothermal synthesis of Mg-doped  $\text{TiO}_2$  anatase samples in water/ethanol environment at 180 °C for 36 h without using any surfactants or templates. Subsequently, glaze samples were prepared by sintering the raw powders (95% Kaolin clay, 5% Mg-doped  $\text{TiO}_2$ ) at ca. 1200 °C. Photocatalytic experiments revealed that Mg-doped  $\text{TiO}_2$  samples have higher photocatalytic activities (99.5% in 80 min under visible light) against Rhodamine B compared with undoped ones, due to pure anatase phase formation. Moreover, ceramic glaze materials present self-cleaning properties, achieving a water contact angle of ca. 6° at room temperature.

Mao et al. [21] analyzed the scientific literature describing the latest advances in the synthesis procedures and strategies to produce and/or properly modify  $\text{TiO}_2$ -based nanomaterials, aiming at maximizing/improving the photocatalytic performances in environmental remediation processes for the abatement of organic pollutants.

Lastly, Naggar et al. [22] reviewed the latest scientific literature describing the recent progress regarding the use of metal oxides in photocatalysis, with a particular focus on the critical role played by their morphology in the overall degradation process. The state-of-the-art analysis revealed that non-spherical morphologies exhibit enhanced photocatalytic performance due to their unique crystal facets and surface areas, which can promote charge transfer and improve catalytic efficiency. Furthermore, porous design and substantial specific surface area are responsible for an increased photocatalytic activity, whereas flake-like structures exhibit comparatively lower performance.

## 2.2. Development and Optimization of New Synthetic Routes for Metal Oxides

Careful consideration has been dedicated in this Special Issue to the development of new synthetic routes for metal oxides, and to the optimization of the synthetic approaches for the preparation of metal oxide nanostructures. In fact, the use of specific experimental conditions can drive the structural and morphological properties of the materials, and the fine control of these parameters play a key role in the determination of the material performances. Moreover, the use of innovative and more recent methodologies is reported, as well as the preparation of hybrid materials [23–25].

Alshoaibi [23] reported a study involving the deposition of a hetero-structured ( $\text{ZnO}/\text{Zn}/\text{ZnO}$ ) thin film on a glass substrate using the DC magnetron sputtering technique. Subsequently, samples were annealed at different temperatures in the 100–500 °C range. Characterization results indicated the formation of both metallic zinc and the hexagonal  $\text{ZnO}$  crystal structure for samples annealed below 200 °C, whereas pure hexagonal  $\text{ZnO}$  formed for samples annealed at 300 and 500 °C, with a slight crystallinity decrease for the sample annealed at the highest temperature. Since both roughness and particle size are inversely proportional to the annealing temperature (with the exception of the sample annealed at 500 °C), the optimal annealing temperature was determined to be 400 °C.

Filimonov et al. [24] reported an original approach to synthesize hollow and layered oxide magnetic nanoparticles (either  $\text{Fe}_3\text{O}_4$ ,  $\gamma\text{-Fe}_2\text{O}_3$ , or  $\text{Fe}_3\text{O}_4/\text{La}_{1-x}\text{Ca}_x\text{FeO}_{3-\gamma}$ ), by a solid-state exsolution process carried out in a reducing environment at elevated temperatures from Ca- and La-based unsubstituted (and substituted) perovskite-related ferrites, and using h-BN as a reducing agent.

Lastly, Abduraman et al. [25] reported a synthetic sol-gel protocol assisted by solvothermal treatment (100 °C, 24 h) using either a triblock copolymer (Pluronic P123) or a nonionic surfactant (Pluronic F127) as soft-templating agents, followed by purification through either Soxhlet extraction or calcination at 400 °C for the production of mesoporous titania nanoparticles. The results indicated that samples prepared using Pluronic F127 presented a higher surface area and less agglomeration than the sample synthesized with Pluronic P123. Furthermore, an extract from *Vitis vinifera* grape marc (*Feteasca neagra* cultivar) with high radical scavenging activity was encapsulated in mesoporous titania and compared with reference SBA-15 silica support. Both resulting materials showed biocompatibility and even better radical scavenging potential than the free extract. Furthermore, the titania encapsulated sample showed better cytocompatibility than the silica one, thus making it suitable for skin-care products.

### 2.3. Drug Delivery and Biomedicine

In this last part, inorganic nanomaterials for drug delivery systems and for biomedicine purposes are discussed [26,27].

Kimura et al. [26] reported the development of a calcium phosphate-coating method to homogeneously cover silica-titania porous nanoparticles (with a well-defined spherical shape, uniform size, and tunable nanoporous structure) in simulated body fluids. The results indicated that the pore size distribution is a fundamental parameter significantly affecting the coating formation, with surfaces with bimodal pore sizes becoming rough after the calcium phosphate precipitation, whereas those with a unimodal pore size remaining smooth, thus indicating that pore sizes serve as different nucleation sites leading to different surface morphologies.

Lastly, Tulyaganov et al. [27] reviewed and critically discussed the genesis, development, properties, and applications of the bioactive glass “1d” (i.e., from the primary crystallization field of pseudo-wollastonite in the  $\text{CaO-MgO-SiO}_2$  ternary system, after addition of  $\text{P}_2\text{O}_5$ ,  $\text{Na}_2\text{O}$  and  $\text{CaF}_2$ ) and its relevant glass-ceramic derivative products (i.e., diopside, fluorapatite, and wollastonite crystalline phases, formed by performing a thermal treatment), which are extremely appealing inorganic biomaterials alternatives to the reference 45S5 Bioglass<sup>®</sup> exploitable in a variety of bone-regenerative clinical applications, such as the repair of periodontal defects, ridge preservation and sinus augmentation.

## 3. Conclusions

With this Special Issue “New Advances into Nanostructured Oxides, 2nd Edition” published in the “Inorganics Materials” section, and also published as a book, the Editors hope that the high quality of the contributions collected here will receive the visibility and attention they deserve. These would help readers to increase their knowledge in the field of inorganic materials, and be a new source of inspiration for novel, focused investigations.

**Acknowledgments:** The Editors would like to thank all authors, reviewers, and the entire editorial staff of *Inorganics* who provided their new science, constructive recommendations, and assisted in the realization of the present Special Issue.

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

## References

1. Innocenzi, P.; Malfatti, L. Mesoporous ordered titania films: An advanced platform for photocatalysis. *J. Photochem. Photobiol. C* **2024**, *58*, 100646. [[CrossRef](#)]
2. Wang, L.; Zhang, J.; Zhang, Y.; Yu, H.; Qu, Y.; Yu, J. Inorganic metal-oxide photocatalyst for H<sub>2</sub>O<sub>2</sub> production. *Small* **2022**, *18*, 2104561. [[CrossRef](#)] [[PubMed](#)]
3. Kumar, A.; Priyanka, C.; Kumar, A.; Camargo, P.H.C.; Venkata, K. Recent advances in plasmonic photocatalysis based on TiO<sub>2</sub> and noble metal nanoparticles for energy conversion, environmental remediation, and organic synthesis. *Small* **2022**, *18*, 2101638. [[CrossRef](#)] [[PubMed](#)]
4. Zhang, Q.; Gao, S.; Yu, J. Metal sites in zeolites: Synthesis, characterization, and catalysis. *Chem. Rev.* **2023**, *123*, 6039–6106. [[CrossRef](#)] [[PubMed](#)]
5. Qiao, R.; Fu, C.; Forgham, H.; Javed, I.; Huang, X.; Zhu, J.; Whittaker, A.K.; Davis, T.P. Magnetic iron oxide nanoparticles for brain imaging and drug delivery. *Adv. Drug Deliv. Rev.* **2023**, *197*, 114822. [[CrossRef](#)]
6. Wagner, J.; Goßl, D.; Ustyanovska, N.; Xiong, M.; Hauser, D.; Zhuzhgova, O.; Hočevár, S.; Taskoparan, B.; Poller, L.; Datz, S.; et al. Mesoporous silica nanoparticles as pH-responsive carrier for the immune-activating drug resiquimod enhance the local immune response in mice. *ACS Nano* **2021**, *15*, 4450–4466. [[CrossRef](#)] [[PubMed](#)]
7. Caldera, F.; Nisticò, R.; Magnacca, G.; Matencio, A.; Khazaei Monfared, Y.; Trotta, F. Magnetic composites of dextrin-based carbonate nanosponges and iron oxide nanoparticles with potential application in targeted drug delivery. *Nanomaterials* **2022**, *12*, 754. [[CrossRef](#)] [[PubMed](#)]
8. Chairungsri, W.; Subkomkaew, A.; Kijjanapanich, P.; Chimupala, Y. Direct dye wastewater photocatalysis using immobilized titanium dioxide on fixed substrate. *Chemosphere* **2022**, *286*, 131762. [[CrossRef](#)] [[PubMed](#)]
9. Danish, M.S.S.; Estrella, L.L.; Alemaida, I.M.A.; Lisin, A.; Moiseev, N.; Ahmadi, M.; Nazari, M.; Wali, M.; Zaheb, H.; Senjyu, T. Photocatalytic applications of metal oxides for sustainable environmental remediation. *Metals* **2021**, *11*, 80. [[CrossRef](#)]
10. Abdullah, F.H.; Abu Bakar, N.H.H.; Abu Bakar, M. Current advancements on the fabrication, modification, and industrial application of zinc oxide as photocatalyst in the removal of organic and inorganic contaminants in aquatic systems. *J. Hazard. Mater.* **2022**, *424*, 127416. [[CrossRef](#)]
11. Mezzomo, L.; Bonato, S.; Mostoni, S.; Di Credico, B.; Scotti, R.; D'Arienzo, M.; Mustarelli, P.; Ruffo, R. Composite solid-state electrolyte based on hybrid poly (ethylene glycol)-silica fillers enabling long-life lithium metal batteries. *Electrochim. Acta* **2022**, *411*, 140060. [[CrossRef](#)]
12. Sun, L.; Liu, H.; Ye, Y.; Lei, Y.; Islam, R.; Tan, S.; Tong, R.; Miao, Y.-B.; Cai, L. Smart nanoparticles for cancer therapy. *Sig. Transduct. Target. Ther.* **2023**, *8*, 418. [[CrossRef](#)] [[PubMed](#)]
13. Nisticò, R.; Scalarone, D.; Magnacca, G. Sol-gel chemistry, templating and spin-coating deposition: A combined approach to control in a simple way the porosity of inorganic thin films/coatings. *Microporous Mesoporous Mater.* **2017**, *248*, 18–29. [[CrossRef](#)]
14. Zhou, Y.; Liu, L.; Li, G.; Hu, G. Insights into the influence of ZrO<sub>2</sub> crystal structures on methyl laurate hydrogenation over Co/ZrO<sub>2</sub> catalysts. *ACS Catal.* **2021**, *11*, 7099–7113. [[CrossRef](#)]
15. Liccardo, L.; Bordin, M.; Sheverdyeva, P.M.; Belli, M.; Moras, P.; Vomiero, A.; Moretti, E. Surface defect engineering in colored TiO<sub>2</sub> hollow spheres toward efficient photocatalysis. *Adv. Funct. Mater.* **2023**, *33*, 2212486. [[CrossRef](#)]
16. Masoud, M.; Khodamorady, M.; Tahmasbi, B.; Bahrami, K.; Ghorbani-Choghamarani, A. Boehmite nanoparticles as versatile support for organic–inorganic hybrid materials: Synthesis, functionalization, and applications in eco-friendly catalysis. *J. Ind. Eng. Chem.* **2021**, *97*, 1–78. [[CrossRef](#)]
17. Bona, G.; Viganò, L.; Cantoni, M.; Mantovan, R.; Di Credico, B.; Mostoni, S.; Scotti, R.; Nisticò, R. An experimental demonstration on the recyclability of hybrid magnetite-humic acid nanoparticles. *Sustain. Mater. Technol.* **2025**, *43*, e01275. [[CrossRef](#)]
18. Abouri, M.; Benzaouak, A.; Zaaboul, F.; Sifou, A.; Dahhou, M.; El Belghiti, M.A.; Azzaoui, K.; Hammouti, B.; Rhazi, L.; Sabbahi, R.; et al. Efficient Catalytic Reduction of Organic Pollutants Using Nanostructured CuO/TiO<sub>2</sub> Catalysts: Synthesis, Characterization, and Reusability. *Inorganics* **2024**, *12*, 297. [[CrossRef](#)]
19. Dolatyari, M.; Tahmasebi, M.; Dolatyari, S.; Rostami, A.; Zarghami, A.; Yadav, A.; Klein, A. Core/Shell ZnO/TiO<sub>2</sub>, SiO<sub>2</sub>/TiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>/TiO<sub>2</sub>, and Al<sub>1.9</sub>Co<sub>0.1</sub>O<sub>3</sub>/TiO<sub>2</sub> Nanoparticles for the Photodecomposition of Brilliant Blue E-4BA. *Inorganics* **2024**, *12*, 281. [[CrossRef](#)]
20. Zhao, T.; Cao, T.; Bao, Q.; Dong, W.; Li, P.; Gu, X.; Liang, Y.; Zhou, J. Significantly Enhanced Self-Cleaning Capability in Anatase TiO<sub>2</sub> for the Bleaching of Organic Dyes and Glazes. *Inorganics* **2023**, *11*, 341. [[CrossRef](#)]
21. Mao, T.; Zha, J.; Hu, Y.; Chen, Q.; Zhang, J.; Luo, X. Research Progress of TiO<sub>2</sub> Modification and Photodegradation of Organic Pollutants. *Inorganics* **2024**, *12*, 178. [[CrossRef](#)]
22. Naggar, A.H.; Ahmed, A.S.A.; El-Nasr, T.A.S.; Alotaibi, N.F.; Chong, K.F.; Ali, G.A.M. Morphological Dependence of Metal Oxide Photocatalysts for Dye Degradation. *Inorganics* **2023**, *11*, 484. [[CrossRef](#)]
23. Alshoaibi, A. The Influence of Annealing Temperature on the Microstructure and Electrical Properties of Sputtered ZnO Thin Films. *Inorganics* **2024**, *12*, 236. [[CrossRef](#)]

24. Filimonov, D.; Rozova, M.; Maksimov, S.; Pankratov, D. Synthesis and Redox Properties of Iron and Iron Oxide Nanoparticles Obtained by Exsolution from Perovskite Ferrites Promoted by Auxiliary Reactions. *Inorganics* **2024**, *12*, 223. [[CrossRef](#)]
25. Abduraman, A.; Brezoiu, A.-M.; Tatia, R.; Iorgu, A.-I.; Deaconu, M.; Mitran, R.-A.; Matei, C.; Berger, D. Mesoporous Titania Nanoparticles for a High-End Valorization of *Vitis vinifera* Grape Marc Extracts. *Inorganics* **2024**, *12*, 263. [[CrossRef](#)]
26. Kimura, R.; Shiba, K.; Fujiwara, K.; Zhou, Y.; Yamada, I.; Tagaya, M. Precipitative Coating of Calcium Phosphate on Microporous Silica–Titania Hybrid Particles in Simulated Body Fluid. *Inorganics* **2023**, *11*, 235. [[CrossRef](#)]
27. Tulyaganov, D.U.; Agathopoulos, S.; Dimitriadis, K.; Fernandes, H.R.; Gabrieli, R.; Bairo, F. The Story, Properties and Applications of Bioactive Glass “1d”: From Concept to Early Clinical Trials. *Inorganics* **2024**, *12*, 224. [[CrossRef](#)]
28. Zamora-Ledezma, C.; Negrete-Bolagay, D.; Figueroa, F.; Zamora-Ledezma, E.; Ni, M.; Alexis, F.; Guerrero, V.H. Heavy metal water pollution: A fresh look about hazards, novel and conventional remediation methods. *Environ. Technol. Innov.* **2021**, *21*, 101504. [[CrossRef](#)]
29. Rahman, A.; Sarkar, A.; Yadav, O.P.; Achari, G.; Slobodnik, J. Potential human health risks due to environmental exposure to nano- and microplastics and knowledge gaps: A scoping review. *Sci. Total Environ.* **2021**, *757*, 143872. [[CrossRef](#)] [[PubMed](#)]
30. Siddiqua, A.; Hahladakis, J.N.; Al-Attiya, W.A.K.A. An overview of the environmental pollution and health effects associated with waste landfilling and open dumping. *Environ. Sci. Pollut. Res.* **2022**, *29*, 58514–58536. [[CrossRef](#)] [[PubMed](#)]

**Disclaimer/Publisher’s Note:** The statements, opinions and data contained in all publications are solely those of the individual author(s) and contributor(s) and not of MDPI and/or the editor(s). MDPI and/or the editor(s) disclaim responsibility for any injury to people or property resulting from any ideas, methods, instructions or products referred to in the content.