

Review On Characterization of Heterogeneous Catalysts: Influence of Particle Size and Pore Size Distribution on Environmental and Automotive Applications

Fatema Salim Merchant¹, Satish R Ingal², Ruchi Dubey³, Mohammad Sajid Mansoori⁴

^{1,2,4}Department of Chemistry,

Shri Vile Parle Kelavni Mandal's, Mithibai College of Arts, Chauhan Institute of Science & Amrutbet Jivanlal College of Commerce & Economics (Autonomous), Mumbai, Maharashtra, India.

³Thakur college of Science and Commerce, Mumbai 56, Maharashtra, India.

*Corresponding author (e-mail: mohd.mansoori@mithibai.ac.in)

Abstract: *Heterogeneous catalysts are important parts of many types of environmental technology employed today, specifically in automotive after treatment for exhaust systems including Diesel Particulate Filters (DPF), and advanced emission control devices. The catalytic efficiency, durability and long-term stability of these catalytic systems are controlled by specific physicochemical characteristics, that have a strong effect on the development of performance characteristics for catalytic systems over the last few decades have been accomplished using both empirical and theoretical studies that show how these structural characteristics contribute to the promotion catalytic activity, the amount and mobility of oxygen through a catalyst, the degree of difficulty for Mass Transport to occur in a carbon-metal-oxide hybrid structure, thermal stability in different temperatures, the resistance of soot due to high-temperature sintering, and the methods of deactivation.*

In this review I have present an integrated and detailed overview of over 31 of the most significant research studies in the area of structure performance relationship for heterogeneous catalysts that have been used for environmental and automotive applications. An essential aspect of this work will focus on the Soot Oxidation catalyst, DPF, 3-Way Catalysts (TWC), fuel cell related Catalyst Layers, and Nanostructured Porous materials. Also discussed in this review will be a critical evaluation of the currently employed characterisation techniques (Gas Physisorption, Chemisorption, Scanning Electron Microscopy, X-Ray Diffraction and Particle Size Analysis) as they relate to the catalytic performance associated with those catalysts.

Keywords: operating continuously in industrial processes; environmentally friendly; Diesel Particulate Filters; Three-Way Catalysts; Heterogeneous Catalysts; Environmental Catalysts; Nanostructured Catalysts; the effects of Pore Size; Surface Area on Particle Size can be found

I. INTRODUCTION

The introduction of the research papers outlines that Catalysis is an integral part of modern chemical processing and environmental technologies related to energy conversion processes and environmental protection. The use of Heterogeneous Catalysts, due to their greater operating efficiency, robustness, ease of separation from Reaction Media and continuous operation in industrial processes, make them an appealing choice for Catalytic Systems (Bartholomew & Farrauto, 2011) particularly in relation to Environmental/Automotive applications where the mitigation of harmful emissions (Carbon Monoxide, Nitrogen Oxides, Unburned Hydrocarbons, and Particulate Matter) presents a serious threat to human health and to the environment. In addition, early development of Catalytic Systems focused on the Chemical, Compositional Selection of Noble and Transition Metals, however subsequent landmark research has



established that the performance of Catalytic Systems cannot solely be attributed to Chemical Composition, but must also consider the Physics and Structure of Catalytic Systems (Particle Size, Surface Area, Porosity, Pore Structure/Architecture, and Morphology) which are equally important parameters in determining the Catalytic Activity, Selectivity, and Durability of Catalytic Systems (Bell, 2003; Antolini, 2015).

As emission regulations continue to tighten (Euro VI, BS-VI, China VI), catalysts need to function properly in larger temperature ranges, more frequently changing exhaust streams and longer service durations. Therefore, it is necessary to develop an understanding of catalyst structure–activity relationships so that new advanced catalysts can be designed rationally. This review provides a summary of both experimental and theoretical information as a means to better understand the combined effect upon catalytic activity created by particle size, surface area and pore structure, and how they affect the catalytic behaviours of diesel particulate filters, ceria based soot oxidation catalysts, platinum group metal (PGM) based three way catalysts (TWC) and new emerging nanostructures used in catalytic architectures.

II. BASICS OF HETEROGENEOUS CATALYSIS AND STRUCTURAL PARAMETERS

A heterogeneous reaction involves the chemical transformation of reactants to products on solid surfaces in contact with either liquid or gaseous phases. The overall rate for a heterogeneous reaction is a combination of many individual steps: external mass transfer, diffusion through the pores, adsorption of reactants onto active sites, reaction on the surface of the catalyst, desorption of products and diffusion of products from the catalytic surface(s) to the reaction medium (Fogler, 2016). All of these steps are highly dependent upon the structural parameters of the catalyst. Particle size affects the surface-to-volume ratio of the particle and therefore, affects the number of low-coordination metal atoms exposed on the surface. The surface area of the catalyst determines the number of accessible active sites and therefore, affects the number of molecules that can react with the active sites of the catalyst. The size distributions of the pores, in conjunction with their shape and connectivity, control the rate at which reactants diffuse to the active sites and the rate at which products diffuse away from the active sites. All of the above parameters are interrelated and to a large extent, changing one parameter results in changes in others. For example, changes in particle size typically produce a corresponding change in surface area but this is not the case for changing the pore size. An increase in porosity could result in a decrease in thermal stability, whereas thermal stabilization accompanied by increased porosity may result in an increased capability of mass transport and decreased mechanical integrity.

Table 1 summarizes the major structural parameters and their functional roles in heterogeneous catalysis.

Table 1. Key structural parameters and their influence on catalytic performance

Structural parameter	Primary influence	Impact on performance
Particle size	Surface atom fraction	Activity, selectivity
Surface area	Active site density	Reaction rate
Pore size distribution	Diffusion pathways	Mass transport
Pore connectivity	Accessibility	Catalyst utilization
Morphology	Facet exposure	Reaction mechanism
Metal dispersion	Active phase distribution	Stability, efficiency

III. EFFECT OF PARTICLE SIZE ON CATALYTIC PERFORMANCE

3.1 Fundamental Role of Particle Size

The particle size is considered to be one of the most important characteristics of heterogeneous catalysts because particle size affects surface area-to-volume ratio and the density of available active sites. Catalytic activity is greater for nanoscale particles than for bulk material (Bell, 2003; Lee et al., 2008) because of the larger number of surface atoms, the greater percentage of surface area compared with total volume and because of the way the electronic structure has changed from bulk materials to nanoscale.

In the case of very small particles (less than 5 nanometers), the quantum size effect and strong metal support interaction further modify adsorption energies and reaction pathways. Shaped nanoparticle synthesis has shown that both size and



shape collectively determine the selectivity of the catalyst, and that the effect of size alone is not sufficient to account for selectivity.

3.2 Particle Size and Structural Stability

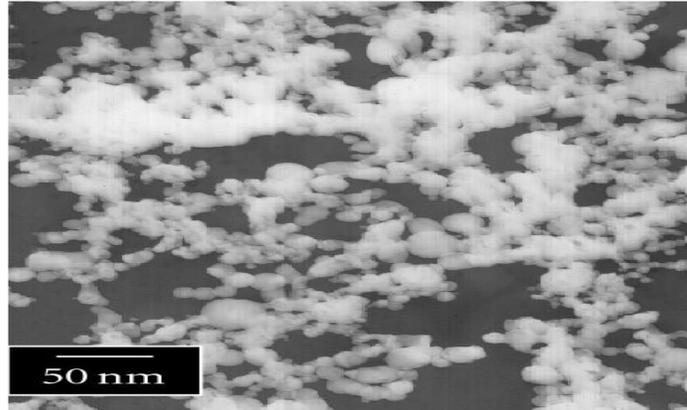


Figure 1 :- TEM micrograph

Although these smaller catalysts show greater activity, they experience lower thermodynamic stability and greater susceptibility to sintering in the environment of the average automobile catalytic converter (i.e., high-temperature working condition). Thermal deactivation (particle coalescence), loss of surface area, and ultimately loss of catalytic activity is the result of thermal aging (Miyoshi et al., 1989). Thus, Antolini (2015), and other researchers, suggest that there is a balance that must be struck between catalytic activity and structural stability.

3.3 Particle Size Effects in Soot Oxidation Catalysts

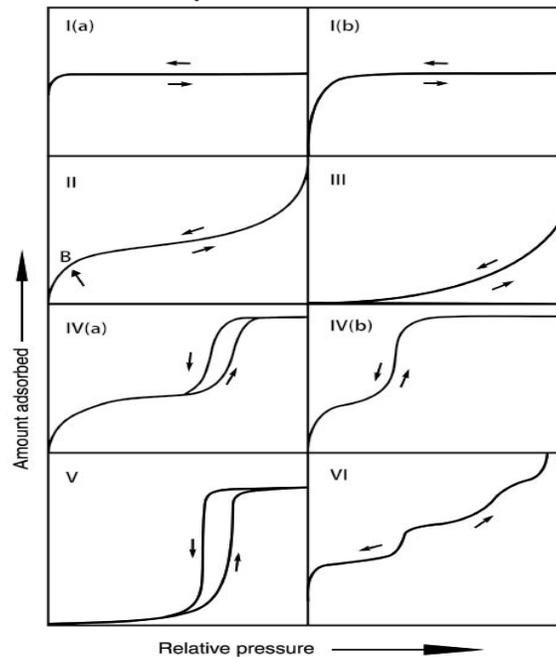


Figure 2. Nitrogen adsorption-desorption isotherms as the method of analysis of surface area and pore size (Thommes et al., 2015).



Figure 2 presents an example of a Type IV adsorption-desorption diagram (isotherm) which typically occurs with mesoporous material. The area in which the two lines cross each other indicates the point at which capillary condensation occurs and demonstrates a structured pore network that provides ample surface area for effective transport of mass to facilitate the catalytic reaction.

Also, The size of the soot oxidation catalyst particles also has a significant effect on the efficiency of the catalyst/soot interaction for diesel particulate filters (DPF). Specifically, smaller catalyst particles create more contact area for an interaction with the soot and enhance the transfer of oxygen between the catalyst and the soot; therefore, lowering the ignition temperature of the soot (Konstandopoulos et al., 2007). Alternatively, high levels of agglomeration can result in poor contact between the catalyst and soot, thus increasing the temperature required for regeneration of the DPF. Thus, uniform particle size distribution and controlled dispersion of the catalyst are important for long-term operation of diesel particulate filters.

IV. SURFACE AREA AND ITS INFLUENCE ON CATALYTIC ACTIVITY

The amount of surface area of a catalyst directly reflects the available number of accessible active sites for catalytic reactions to occur. Catalysts with a high surface area tend to have a greater capacity to adsorb materials onto their surfaces and greater reaction speed for gas–solid reactions that are important for environmental catalysts (Sing et al., 1985).

A high surface area does not guarantee superior performance. The surface area must be accessible, thermally stable, and supported by a suitable pore structure. Strong metal–support interactions in supported catalysts stabilize the small metal particles and preserve the surface area during long-term use (Antolini, 2015). Thermal degradation of high surface area supports, such as γ -Al₂O₃, still presents a significant challenge for exhaust applications that operate at elevated temperatures.

V. PORE SIZE DISTRIBUTION AND MASS TRANSPORT EFFECTS

5.1 Classification of Pores



According to the IUPAC classification, pores can be defined as micropores (<2 nm), mesopores (2–50 nm), and macropores (>50 nm) (Thommes et al., 2015), and each of the three classes of pores provides distinct benefits to a catalyst.

5.2 Role of Mesoporosity in Environmental Catalysis

For automotive and environmental catalysis applications, mesoporous materials provide an excellent combination of both high surface area and effective mass transfer due to their pore geometry. Hydrocarbon molecules and soot, for example, require large pores to easily access the active sites of the catalyst. Hierarchical pore structures that contain varying sizes of micropores, mesopores, and macropores enhance the rate of diffusion while maintaining a high catalytic activity (Seliem et al., 2020).



Table 2. Role of pore size regimes in catalytic systems

Pore Types	Size Range and its Functional Role
Micropores	<2nm and High surface area
Mesopores	2-5nm and Diffusion and reaction
Macropores	>50nm and Transport channels

VI. STRUCTURES PARAMETER CHARACTERIZATIONS TECHNIQUES.

Characterisation of a catalyst requires multiple techniques to obtain a complete understanding of the structure of the catalyst. For example, measurements of surface area and pore size distribution can be obtained by the gas physisorption technique (Brunauer-Emmett-Teller (BET) and Barrett-Joyner-Halenda (BJH)). Morphology, dispersion, and sintering of catalyst particle can be determined using electron microscopy techniques, such as scanning (SEM) and transmission electron microscopy (TEM). Information about the crystallite size, phase composition, and structural changes can be determined from X-ray diffraction (XRD) measurements. The H₂, CO, and O₂ measurement in chemisorption provide information about the dispersion and active site density of the metals in a catalyst. Increasingly, researchers use Particle Size Analyzers (PSAs) and laser diffraction techniques (LDTs) to quantify and measure the particle size distribution of catalyst particles as well as coating particles. To associate the structural measurement with the catalytically active sites, it is necessary to correlate all of these measurements.

VII. PARTICLE SIZE DISTRIBUTION IN DIESEL PARTICULATE FILTERS

The diesel particulate filter (DPF) is an important part of any aftertreatment system for the reduction of diesel engine particulate emissions. The particle size distribution of the soot and the catalyst material both influence filtration efficiency, regeneration behaviour and pressure drop through the filter. Gasoline engine soot particles will range from ultrafine nanoparticles to large agglomerates and will have different oxidation kinetics based on their size and morphology. In our research, we have shown that on DPFs the DPF's supported catalyst configuration provides the most efficient and effective means for soot-to-catalyst interactions, and has been shown to provide the lowest soot ignition and conversion temperatures compared to using loose soot particles. Therefore, on DPFs, the soot-to-catalyst interactions have the greatest effect on increasing the rate of soot carbon combustion. (Konstandopoulos et al., 2007).

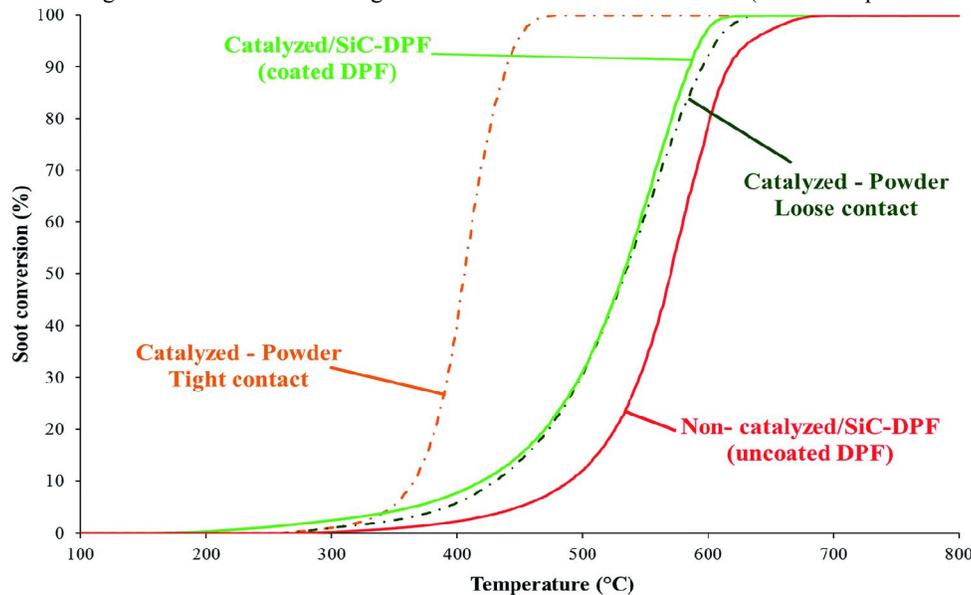


Figure 4: Soot combustion profiles (T vs conversion) for $\text{CuO/Ce}_{0.8}\text{Zr}_{0.2}\text{O}_2$ catalysts on SiC-DPF vs powder catalyst showing lower oxidation onset temperatures and enhanced activity (Quiles-Díaz et al., 2015).



Explanation:

In addition, the use of various engine transient operating conditions influences and changes the way exhaust particle size distributions and soot load behavior exist in the DPF. Many researchers have examined how changes in engine operation create changes to PSDs (Liu et al., 2003) and, as a result, affect the regeneration efficiency and longevity of the DPFs and how these PSD differences result in uneven thermal load on the filters (i.e., greater thermal stress on some filter areas than others). Thus, new devices and analytical techniques for advanced aerosols have been created to allow precise measurements of both soot PSD and the soot's interaction with the catalyst surface (Xiao et al., 2013). All of these studies on the research papers illustrate a clear need for precise control over the PSD of soot and catalyst, which is necessary to achieve optimal DPF operation while meeting stringent emissions regulations while also providing a high degree of durability.

VIII. THREE-WAY CATALYSTS FOR AUTOMOTIVE EXHAUST TREATMENT

TWCs, when in operation, are subjected to extreme thermal conditions which can cause sintering of the metal particles and the destruction of the pore structure. Sintering causes growth of metal particles therefore the loss of surface area and reduced efficiency of NO_x conversion over time, as demonstrated by Miyoshi et al. (1989). In addition to thermal aging, more recent experimental simulation research has shown that the morphology of the catalyst, the pore architecture, and the metal/support interactions may also significantly affect the long-term exhaust conversion efficiencies of the catalysts (Chen et al., 2019). These are some of the main reasons that the loss of structural integrity due to high-temperature exposure is a major challenge in the design of TWCs.

IX. POROSITY–PERFORMANCE RELATIONSHIPS IN CATALYST LAYERS

Porosity is also one of the structural characteristics of a catalyst layer that controls the transport of mass through the catalyst layer and how much of the catalyst is used in a reaction, as well as how much of the catalyst remains intact during use. Therefore, in an automotive catalyst, or in an electrolyzer or fuel cell system, an interconnected network of pores allows for efficient movement of reactants and removal of products from the catalyst layer during operation. Sabharwal and Secanell (2022) demonstrated that catalyst layers that are moderately porous and have well-distributed pores could significantly outperform those that are either very porous or very dense structurally in electrochemical and catalytic performance. A major benefit of an increase in porosity is that it improves the transport of gases to and from the catalysts. A downside of an increase in porosity is that when porosity becomes excessive, mechanical integrity can be compromised, as well as the ability for electrical current to flow through the catalyst layers between catalyst particles. On the other hand, a catalyst layer that has an insufficient level of porosity will develop limitations on the diffusion of reactants and as a result, will not fully utilize the active catalytic site for converting the reactants into products. Therefore, the use of a hierarchical pore structure in a catalyst layer has demonstrated an optimal combination of surface area for the catalyst and the ability to efficiently transfer mass across the catalyst layer (Sing et al., 1985; Thommes et al., 2015). Therefore, in summary, the findings detailed above show that porosity must be optimized to produce durable and high-performance catalytic layers.

X. NANOSTRUCTURED AND POROUS MATERIALS IN ENVIRONMENTAL APPLICATIONS

For a long time now, porous materials have been of large interest for several environmental and catalytic remediation applications. Due to their very high surface area, controllable pore structures and unique properties, porous nanostructured porcelain and porous microspheres are being sold with high amounts of accessible and linked active sites. The porous materials mentioned above allow for enhanced adsorption rates of contaminants and higher reaction rate (Seliem et al., 2020). The advantage of having a well-defined microporous and nanoporous structure, which gives very good selectivity during catalytic reactions, is a major benefit of the colorful structure–function properties of these materials.



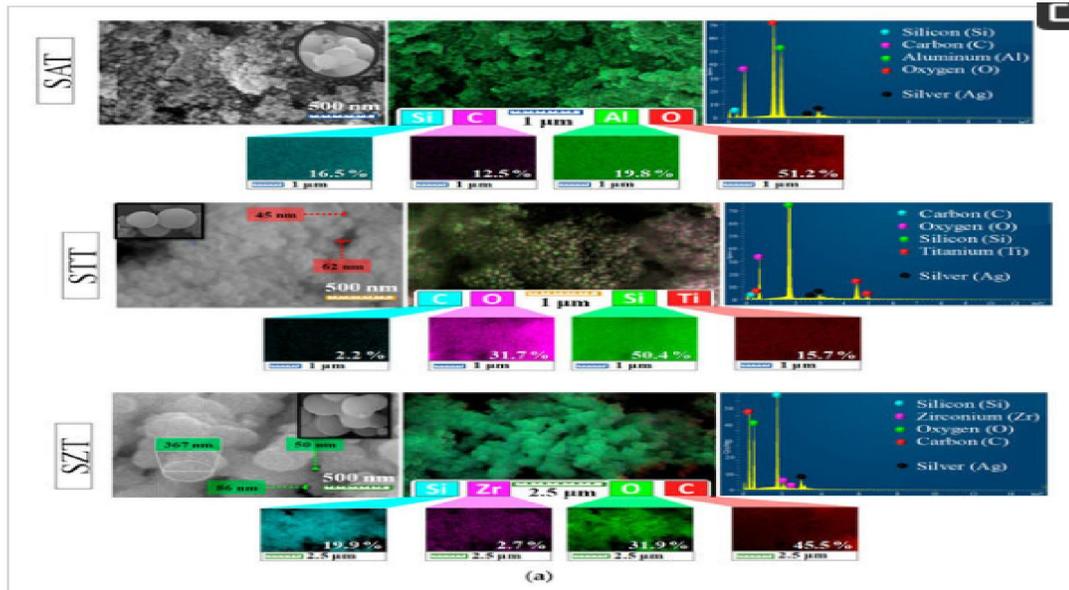


Figure 5: SEM pictures of nanocomposite adsorbent (Seliem et al., 2020).

As shown in Figure 6, SEM images show the distinct characteristics of the porous catalytic materials as "nanospheres" and "nanopore" networks. The amount of active sites available increases with the very high surface area and accessibility of the porous structures, which provide enhanced catalysis. Together with their magnetic recovery properties, these materials improve environmental remediation efficiency.

A wide variety of applications exist for porous microspheres, particularly in catalysis, adsorption and support of active compounds has been observed because they are able to store large amounts of material and provide a high degree of structural stability. Ughreja et al. (2025) demonstrated that these materials are among the most efficient materials available for catalyst applications, including environmental remediation and in the biomedical area, due to the mass transport advantage of their interconnected pore structure and the availability of active catalytic sites. The findings by Ughreja et al. (2025) illustrate the fundamental relationship between structure and performance established in catalytic automotive applications across a number of different environmental technology applications.

XI. CONTEMPORARY TRENDS IN CATALYST DESIGN

Current trends in catalyst design are leading to an increase in the use of nanoscale engineering and precise control of the structure. The development of synthesized nanoparticles with defined shapes allows for selective exposure of specific crystal faces that directly affect both the adsorption and reaction pathways (Lee et al., 2008). Another emerging area of design strategy is the use of hierarchical porosity in order to combine multiple porosity sizes to simultaneously increase both the surface area and mass transport.

Another recent area of interest is the use of atomically dispersed catalysts, which use the maximum potential of metals through the means of highly controlled placement of metal atoms and create catalytic properties unlike that of nanoparticle systems (Ye et al., 2016). Furthermore, the development of in-operando and in-situ techniques will greatly enhance our understanding of catalyst behaviour under realistic working conditions, giving insight into the how the structure may change throughout a reaction. These trends ultimately signal a change to a more rational and durable catalyst design approach.

XII. RESEARCH GAPS AND FUTURE PERSPECTIVES

Heterogeneous catalysts have come a long way with the development of catalytic converters for the automotive and environmental industries. Still, there are significant challenges that need to be overcome before heterogeneous catalysts can be used commercially. Long-term catalyst durability is currently limited due to sintering of the catalyst at high



temperatures, particularly where the catalyst will see rapid fluctuations in the exhaust stream. In addition, there is limited research data available on the long-term stability of catalysts under real-world operating conditions. Laboratory test data does not provide a good representation of how a catalyst will perform in an industrial setting (Thommes et al., 2015).

Research in the future must develop the methods to run operando characterizations to be able to trace dynamic changes in structure with increasing resolution during operation of An example is a Heterogeneous Catalyst. The goal is to create a new generation of Multi-functional Catalytic Systems (MCS) that incorporate multiple catalytic characteristics and provide enhanced stability and prevention of deactivation. For a sustainable catalyst development strategy to comply with future tighter regulations for reducing emissions, it will include minimization of the use of precious metals and use of abundant earth materials (Konstandopoulos et al., 2016)

XIII. IMPLICATIONS FOR SUSTAINABILITY AND ENVIRONMENTAL PROTECTION

The optimization of the particle size, surface area, and pore structure of catalysts has important implications for environmental protection and sustainability. Higher catalytic efficiency will reduce harmful emissions caused by combustion and improve the fuel usage efficiency of vehicles while allowing for greater lifetime of catalysts due to improved structural stability, which reduces the amount of waste generated from material consumption. By allowing for a greater dispersion of the active metals (catalytic elements), catalyst designs can reduce the amount of noble metals used (and thus the costs and environmental impact of those noble metals); Antolini (2015) supports this assertion. In addition to the catalytic benefits of optimizing the catalytic structure, the use of optimally designed catalysts will require lower regeneration temperatures, thus requiring less energy input to operate. Collectively, these considerations will also facilitate the development of cleaner combustion technology and environmentally friendly methods of producing goods.

XIV. CONCLUSION

All of the reviews in this series shows that particle size, surface area, and pore structure are the primary factors affecting the performance of heterogeneous catalysts used in the automotive industry and the environmental sector. The correlation of the catalytically active structure and performance is shown through numerous examples, including the oxidation of soot, diesel particulate filters, and automotive exhaust after-treatment systems. The current advancements in nanoscale engineering, hierarchical pore structure design, and "on-the-fly" characterisation will enable the development of more effective, longer-lasting, and environmentally friendly catalyst systems. As the structural optimization of catalysts and realistic performance measurements become increasingly integrated, the ability to address future environmental challenges and to comply with current regulations will be greater than ever before.

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