



A multi-scale study of 3D printed Co-Al₂O₃ catalyst monoliths versus spheres

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ABSTRACT

This study demonstrates the characteristics of two model packing configurations: 3D printed (3DP) catalyst monoliths on the one hand, and their conventional counterparts, packed beds of spheres, on the other. Cobalt deposited on alumina is selected as a convenient model system for this work, due to its wide spread use in many catalytic reactions. 3DP constructs were produced from alumina powder impregnated with cobalt nitrate while the alumina spheres were directly impregnated with the same cobalt nitrate precursor. The form of the catalyst, the impregnation process, as well as the thermal history, were found to have a significant effect on the resulting cobalt phases. Probing the catalyst bodies *in situ* by XRD-CT indicated that the level of dispersion of identified Co phases (Co₃O₄ reduced to CoO) across the support is maintained under reduction conditions. The packed bed of spheres exhibits a non-uniform distribution of cobalt phases, including a core-shell morphology with an average crystallite size of 10–14 nm across the sphere, while the 3DP monolith exhibits a uniform distribution of cobalt phases with an average crystallite size of 5–12 nm upon reduction from Co₃O₄ to CoO. Computational Fluid Dynamics (CFD) modelling was carried out to develop digital twins and assess the effect of the geometry of both configurations on the pressure drop and velocity profiles. Finally, the activity of both Cobalt-based catalyst geometries was assessed in terms of their conversion, selectivity and turn over frequencies under model multi-phase (selective oxidation) reaction conditions, which showed that the desired 3D printed monolithic geometries can offer distinct advantages to the reactor design.

1. Introduction

The substitution of platinum group metals (PGMs) by more earth abundant 3d transition or base metals present an attractive solution from a sustainability perspective although the latter tend to suffer from lower performance. One example is cobalt catalysts, which are one of the most commonly used and known to efficiently catalyse a great number of reactions due to cobalt's redox state and the possibility to optimise the nanoparticle size, rendering its performance similar to that

of PGMs [1,2]. However, improved performance can be obtained when optimising the shape, size and structure at both the nano- and macro-scales [3–5]. One of the main advantages of monolithic catalysts is that they offer low pressure drop and higher mass and heat transfer leading to the optimisation of the reactor design [6,7].

Owing to the flexibility of the 3D printing process, there has been considerable interest in monolithic reactors for a wide range of catalytic applications [8,9]. In this work the monoliths were 3D printed with straight-line geometry and uniform cell density. In the literature, whilst

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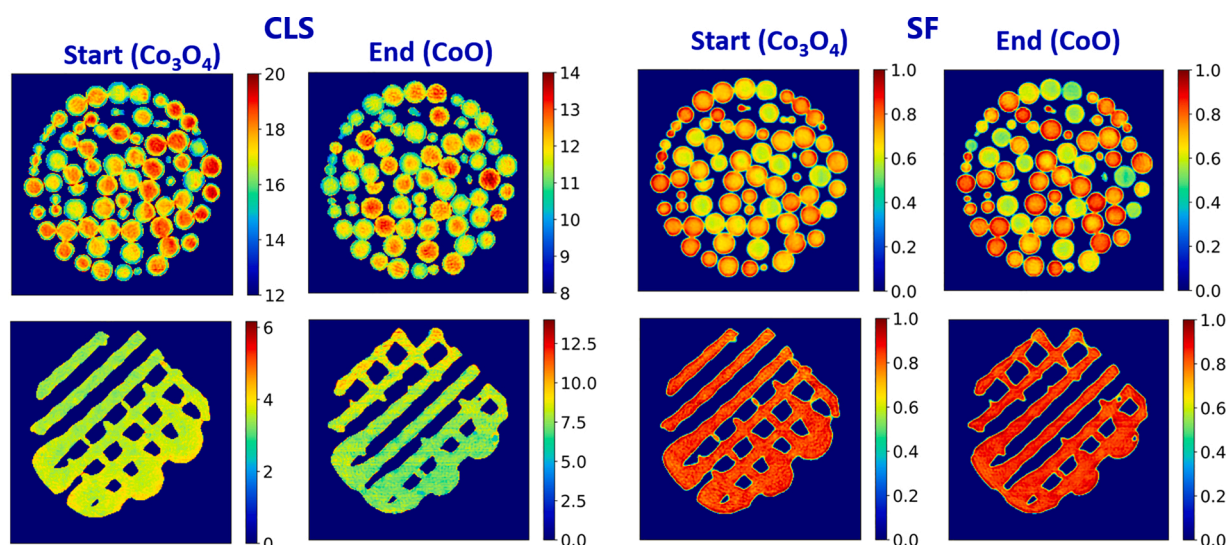


Fig. 1. Reconstructed 2D XRD-CT images of Cobalt phases across PB of spheres (top row) and 3DP monolith (bottom row) under 100 ml min^{-1} flow of 5% H_2 in He: (from left to right) average crystallite size (CLS), colour scale bar in nm, and scale factor (SF) 0–1; each set of images shows start of temperature ramp - Co_3O_4 (left) and end of temperature ramp at 400°C - CoO (right).

there is a wealth of work on CFD modelling of fixed bed reactors [10,11], little work has been done on the direct comparison of 3D printed (3DP) monoliths and their packed bed (PB) counterparts [12,13]. The straight-channel monoliths were evaluated by Computational Fluid Dynamic (CFD) comparing their flow patterns and pressure drops for varied superficial velocities with that of PB of spheres.

The vast majority of *in situ* and *operando* studies in the literature utilise the traditional 0D single-point scans of catalysts in powdered form. We present here XRD-CT analysis of the entire 2D cross sections of both configurations of the $\text{Co-Al}_2\text{O}_3$ structured catalysts, 3DP catalyst monoliths and PB of spheres, recorded before and after reduction in H_2/He . This set of analysis tools enables the catalyst bodies to be ‘chemically imaged’ in spatial and temporal dimensions in order to provide a better understanding of the relationship between catalyst structure and function under operating conditions [14,15].

Finally, the activity of both catalyst forms has been tested in a model multiphase flow reaction (the selective oxidation of benzyl alcohol to benzaldehyde) that has been widely adopted in the chemical industry for its intermediate products [16,17]. However, the majority of selective oxidation reaction processes are operated in closed batches for large volumes, or, alternatively, in microchannel reactors for continuous flows at small scale. Building on their most recent work on the effects of 3DP geometries in different bimetallic $\text{FeCo@Al}_2\text{O}_3$ and $\text{FePd@Al}_2\text{O}_3$ compositions on catalytic performance [18], the authors demonstrate here that the conventional (batch and PB) PGM-based catalytic systems can be entirely replaced by cobalt while comparing 3DP monolithic catalyst beds with their PB analogues under identical operating conditions.

2. Materials and methods

The impregnation method and 3D printing (direct write) were previously described by the authors [19,20], (for further details see Supplementary Material).

XRD-CT measurements at the ESRF ID15A beamline [21] were performed in a quartz reactor tube (see Supplementary Figure S1) using a monochromatic beam of 89 keV focused to a spot size of $30 \mu\text{m} \times 30 \mu\text{m}$. XRD-CT scans were recorded using interlaced method [22]. Each complete interlaced scan comprised 39,600 diffraction patterns, each acquired over 10 msecs. 2D powder diffraction patterns were collected using a state-of-the-art Pilatus3 X CdTe 2 M hybrid photon counting area

detector. The final XRD-CT images (i.e. reconstructed data volume) were processed using the filtered back projection algorithm and pyFAI and the nDTomo software suite [23–26].

The reactor set-up and conditions for the aerobic selective oxidation of benzyl alcohol into benzaldehyde were used as previously described by the authors [27]

CFD simulations were performed in open-source software open-FOAM. First, spheres bed and monolith were fully digitally twined. For computational mesh (CM), a procedure was applied from previous work of the authors, which proved to be effective for grid-independent velocity field [27]. The final CM consisted of ~ 1 million elements for both geometries. All CFD simulations were conducted with k-w type Reynolds averaged model.

3. Results and discussion

3.1. *In situ* XRD-CT analysis

XRD-CT was used to image the spatial (and temporal) distribution of Cobalt phases across the catalysts. Fig. 1 shows the resulting, reconstructed 2D XRD-CT images of PB of $\text{Co-Al}_2\text{O}_3$ catalyst spheres and 3DP monoliths before and after reduction. The peak fitting results obtained according to the methods previously described by the authors [28,29] are explained in more detail in Supplementary Figures S2-S5. The parallax artefact was removed from the XRD-CT data using a 3D parallax Radon we previously developed for the DLSR approach [30]. Two reconstructions were obtained per XRD-CT dataset using the odd and even projections; the data were then denoised using the Noise2Inverse approach [31] with a 3D convolutional neural network (for more details regarding the reconstruction algorithm see the Supplementary Material section, Figures S6-S16).

Clear changes in diffraction patterns can be observed in both sets of temperature ramp data (for the PB of spheres see Fig. 1, top, and for 3DP monolith Fig. 1, bottom). Both Al_2O_3 supports are crystalline, however, the spheres are more crystalline than the printed sample which can be attributed to their different preparation methods (see Supplementary Figure S15 and Table S1). Fig. 1, top shows clearly crystalline Co_3O_4 in the spheres reducing to CoO. In contrast, Fig. 1, bottom, shows the Co_3O_4 to be less crystalline in the 3DP material reducing to less crystalline CoO.

Initially Co is present in spinel form, in 18–20 nm crystallites (Fig. 1,

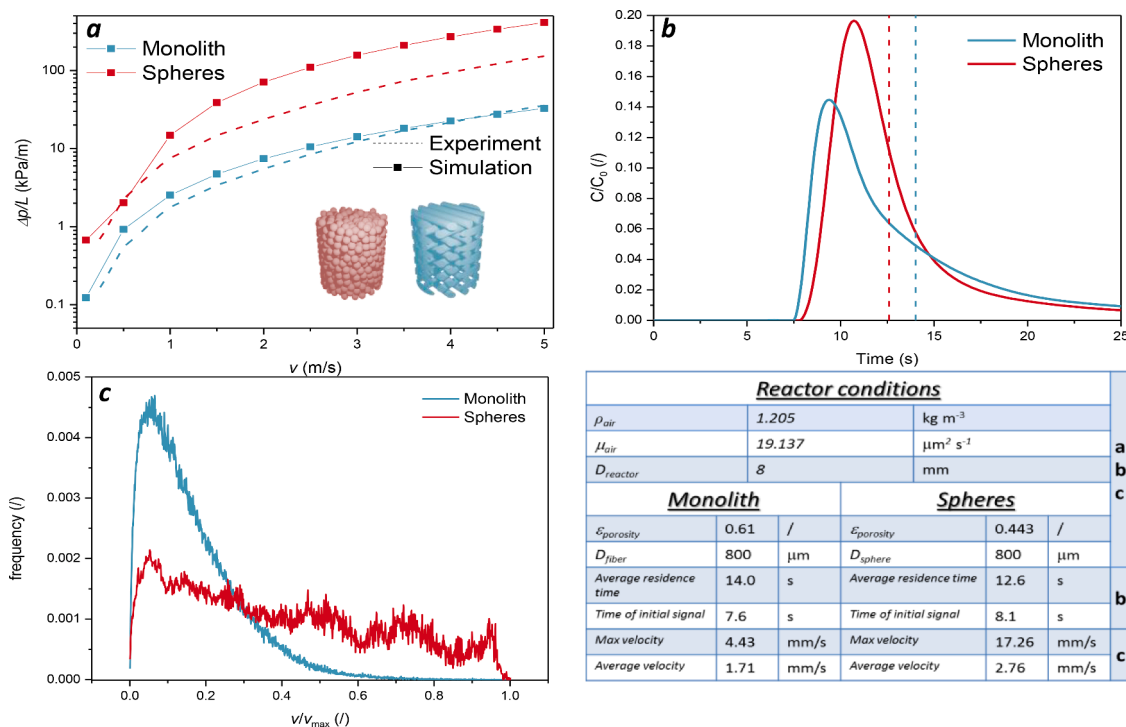


Fig. 2. CFD results for PB of spheres and 3DP monolith: (a) Pressure drops for various superficial velocities. (b) Tracer test for superficial velocity of 1 mm/s. (c) Velocity distribution for superficial velocity of 1 mm/s.

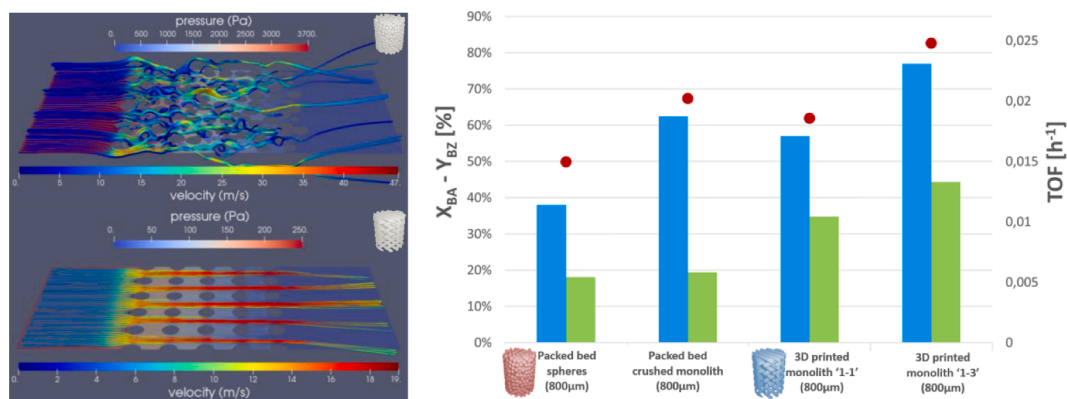


Fig. 3. Performance comparison for 3DP Co-Al₂O₃ monoliths (fibre thickness and interfibre distance of 800 μm) with their PB analogues (spheres and 3DP crushed catalysts fractions of around 800 μm) - Left panel: stream lines and pressure drops for spheres (top) and monolith (bottom) at superficial velocity of 5 m/s. Right panel: benzyl alcohol oxidation in flow reactor; Legend: X_{BA} (blue) corresponds to conversion of benzyl alcohol; S_{BZ} (green) corresponds to selectivity into benzaldehyde; TOF (red) is turn-over frequency, expressed in h^{-1} .

top left), with some variation in lattice parameters across the PB of spheres (Figure S16). The Co₃O₄ is almost fully converted to CoO by the end of the ramp. The reduction in size (from ca. 12–20 nm to 10–14 nm) is expected due to the loss of oxygen from the lattice. Cobalt crystallite sizes appear independent of support crystallite size. There may be some movement of Co in spheres as clustering of larger CoO crystallite sizes appears to have occurred. This points to tentative correlation with larger support crystallites.

Both the average crystallite size (CLS) images in Fig. 1, top left and scale factor (SF) images in Fig. 1, top right, show a non-uniform distribution of Cobalt phases across the PB of spheres, including a core-shell distribution for individual spheres which is supported by SEM/EDX images (Figure S18). In contrast, a homogeneous distribution of Cobalt phases can be observed across the 3DP monolith in both the CLS (Fig. 1, bottom left) and SF (Fig. 1, bottom right) images, which is confirmed by

the SEM/EDX results (Figure S19). This underlines the fact that 3D printing offers greater control and uniform distribution of the active catalyst material. Upon reduction from Co₃O₄ to CoO, the average CLS is expected to decrease if Co dispersion in the support is maintained. However, in this case, the Co average CLS per pixel in the 3DP monolith is shown to increase from ca. 2–6 nm to 5–12 nm (Fig. 1, bottom left). This may be indicative of some sintering, or, as previously stated, it may be due to poor crystallinity.

3.2. CFD analysis

The pressure drop of the spheres and monolith configurations is presented in Fig. 2a. As expected, the pressure drop for spheres is substantially higher than that for monoliths for the same characteristic dimension (i.e. spheres and fibre diameter of 800 μm). This is mainly

due to higher porosity of monolith, which leads to lower average velocity and consequently lower viscous losses (see Fig. 2c). In addition, the flow pattern in monolith follows creeping flow, while in case of spheres the flow is more swirly, which leads to higher inertia losses and consequently higher pressure drop (see Fig. 3 left panel). While this is a well-known fact, it is not straightforward to deduce the impact of geometry on the heat and mass transfer solely from the experiments. Therefore, a detailed CFD tracer test analysis of residence time was performed (see Fig. 2b). Even though the average velocity in case of spheres is higher than monolith, the residence time of the initial signal is shifted to later time than monolith, meaning that spheres have higher tortuosity than monolith and consequently more inertia losses. In contrast, the average residence time of monoliths is higher than that of spheres which leads to higher conversion rates.

3.3. Catalytic testing in model selective oxidation reaction

The present study exhibits a geometry dependency for Co-Al₂O₃ in the selected model reaction. When formed into monolithic catalysts by 3D printing, Co-Al₂O₃ showed higher conversion, selectivity and turn over frequency (TOF) during the benzyl alcohol oxidation to benzaldehyde over the same range of reaction conditions. Equivalent experiments were performed in the flow reactor using not only catalyst spheres but also 3DP catalyst in a crushed (and pelletised) form (see comparison results in Fig. 3, right panel). 3DP monoliths with the straight channels (designated "1-1") exhibited conversion rates and TOF comparable to their crushed counterparts while the catalyst spheres have the lowest conversion and TOF. The 3D-printed monoliths with crossed channels (designated "1-3") had a higher conversion rate (78%) and TOF (0.025 h⁻¹) than those of monoliths with straight channels. Further optimisation of 3D printed monoliths' (crossed channel) geometry and their performance in terms of conversion, selectivity and TOF, was successfully demonstrated (see Supplementary Figures S20 and S21).

4. Conclusions

A model alumina-supported cobalt catalyst was 3DP into a monolith with straight channels and examined together with its counterpart, conventional PB of model spheres. The effect of the packing geometry was studied by directly comparing CFD and experimental data for the pressure drop and velocity profiles of the respective configurations. The pressure drop in the PB of spheres was significantly higher in comparison to that in the monolithic reactor considering the characteristic dimensions are the same (i.e. monolith filament and spacing of 800 μm and sphere diameter of 800 μm). This is attributed to the higher porosity of the monolith, which leads to lower average velocity and consequently lower viscous losses.

Secondly, the application of complementary *in situ* and *ex situ* techniques (such as XRD-CT and SEM-EDX) is invaluable in assessing the fidelity of the printing process and mapping the catalyst chemical form, microstructure and behaviour. The reconstructed images of the *in situ* XRD-CT measurements under H₂ flow show (from the nano and micro to macro scale) the differences in the distribution and level of crystallinity and particle size of cobalt species between the spheres and the printed sample. The 3DP monolith exhibits a uniform distribution of cobalt phases throughout the cross section while non-uniform distribution is observed across the packed-bed of spheres including their core-shell morphology.

Lastly, the model packings were compared under model reaction conditions of the selective oxidation of benzyl alcohol to benzaldehyde. The monoliths with straight channels showed conversion rates and TOF that were equivalent to those of their crushed PB counterparts, whereas the catalyst spheres had the lowest TOF and conversion. The optimised geometry and obtained performance of 3DP Co-Al₂O₃ monoliths with crossed channels (detailed in the Supplementary Material section) reached 90% conversion of benzyl alcohol (0.029 h⁻¹ TOF),

demonstrating their potential to replace both the traditional PB configurations and PGM-based catalytic systems. This illustrates that 3D printing of catalyst monoliths allows great control over the active material distribution, flow pathways, reaction times and higher surface area to volume ratio for the reactants, facilitating easy set-up, scale-up and ultimately reaction optimisation.

CRedit authorship contribution statement

Investigation, Methodology, Validation, Visualization: C.J., L.P and V.M. carried out the synthesis/materials preparation, catalytic testing and conventional characterisation. A.P. and B.L. performed CFD modelling and interpretation. V.M., D.M. and S.D.M.J. designed and performed the XRD-CT experiments with contributions from A.V. and A.M.B. A.V. and H.D. developed the iterative GPU-based parallax reconstruction approach used in this work. Software, Visualization: A.V. developed and applied the code for the self-supervised denoising approach used in this work with contributions from H.D. XRD-CT data interpretation was performed by A.V., H.D., V.M. and S.W.T.P. with contributions from S.D.M.J. and A.M.B. A.V. performed the full profile analysis of the XRD-CT data with contributions from S.W.T.P. and V.M. Writing - review&editing: all authors made contributions to writing and editing the manuscript.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

We have published the sinogram data only on <https://zenodo.org/record/8116842> (DOI: 10.5281/zenodo.8116842).

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at [doi:10.1016/j.cej.2023.100538](https://doi.org/10.1016/j.cej.2023.100538).

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