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Ammonia Cracking Hollow Fibre Converter for on-board Hydrogen production

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Abstract
This work studies the feasibility of a pioneer technology for on-board hydrogen production: the Ammonia Cracking Hollow Fibre Converter. The catalytic activity of a series of ruthenium-based catalysts supported on carbon xerogel, during the ammonia cracking reaction, was studied in a catalytic packed bed reactor. To improve their physical-chemical properties, carbon xerogels were activated in either carbon dioxide or ammonia atmosphere. The most active catalyst (i.e. Ru-NCX) was then deposited inside the micro-structured hollow fibre support by a combination of sol-gel and incipient wetness impregnation methods. At 450°C and 1 atm the hollow fibre reactor was 4.6 times more efficient than the packed bed reactor (i.e. \( r_{\text{NH}_3} = 6.5 \times 10^4 \text{ molNH}_3/\text{m}^3\cdot\text{h}\cdot\text{g}_{\text{cat}} \) and \( r_{\text{NH}_3} = 3.0 \times 10^5 \text{ molNH}_3/\text{m}^3\cdot\text{h}\cdot\text{g}_{\text{cat}} \), respectively), due to its narrower residence time distribution and reduced mass transfer limitations. Furthermore, the use of the hollow fibre converter entailed significantly lower pressure drop (i.e. >99% less), volume (i.e. 80% less) and catalyst loading (i.e. 80% less) compared to the packed bed reactor. Therefore, the potential of this new technology is enormous, as it will push the incorporation of green ammonia in the present-day fuel scenario.

Keywords: Multichannel asymmetric hollow fibres, Sol-gel method, Catalytic hollow fibre converter, Ammonia decomposition, On-board hydrogen production

1. Introduction
Green hydrogen has been proposed as a promising alternative to traditional carbon-based automotive fuels. However, due to safety concerns and the low energy density of hydrogen, the use of hydrogen carriers is required [1,2]. Green ammonia, which is
Liquid at room temperature and low pressure conditions (i.e. 108 kg of hydrogen per m³ at 20°C and 8.6 atm) has been proposed as a promising hydrogen carrier candidate for on-board applications [3–5]. In addition, on-board hydrogen production via ammonia decomposition presents the advantage over steam reforming that it does not need to use an additional reactant such as steam. Hence, when using ammonia as a hydrogen carrier, additional infrastructure to fit the water tank and steam generator system in the vehicle is not needed [6,7].

However, board space constraints still present challenges in the adoption of green ammonia as a future fuel. Today, the most prominent technology for hydrogen production via ammonia cracking is a traditional catalytic packed bed reactor (PBR) that used a precious metal-based catalyst [8]. As a result, this expensive technology is outsized, limiting its applicability to an ammonia-fuelled vehicles [8].

This scenario represents a unique opportunity for innovative solutions in the development of a suitable technology for on-board hydrogen production via ammonia cracking. Recently, different reactors designs for ammonia decomposition such as monolithic and micro reactors have received a lot of attention due to their compact design, reduced weight, and improved conversion compared to traditional PBRs [6,7,9,10]. Wang et al. developed a monolithic-base "miniature" ammonia cracker (i.e. overall weight of ≈195 g and volume of ≈50 cm³) for portable fuel cell power supply. This cracker was able to produce roughly 158 W equivalents of hydrogen via ammonia decomposition at high temperature [6]. Despite the lower pressure drop of the "miniature" ammonia cracker compared to a traditional PBR, its relatively large size (i.e. we have calculated volume ≈30 L to produce roughly ≈100 kW equivalents of hydrogen) and high operation temperature (i.e. conversion of >99.9% at 600°C) challenge its use for on-board applications. Furthermore, accumulation of unused catalyst in the corners of channels is an unavoidable issue in monolithic-base reactor technology, which affects their cost and limit their use [11].

Likewise, Chiuta et al. developed a super compact ammonia-fuelled microchannel reactor unit (i.e. a plate consisted of 80 microchannels) wash-coated with a commercial 8.5 wt% Ru-based catalyst for hydrogen production [10]. This type of microchannel reactors present the advantage of an easier power scale-up via integration of units [7,10]. However, despite its exceptional small size (i.e. we have calculated volume ≈1 L to produce roughly ≈100 kW equivalents of hydrogen), its high operation temperature (i.e. conversion of >99.8% at 600°C) and the large noble metal
content (i.e. 8.5 wt% Ru is far from the current low loading noble metal target) challenge its use for on-board applications.

More recently, the interest in continuous catalytic hollow fibre reactors (HFRs) has increased due, in part, to the narrower residence time distribution when compared with PBRs that suffer from recirculation and generation of preferential paths and stagnant regions. Their high thermal/mechanical stability and large area/volume ratio allow operation with a decreased catalyst metal loading and/or lower operating temperatures compared to traditional catalytic PBRs, while easy control of reactant/catalyst contact time can be used to fine tune selectivity [11–13].

Ruthenium-based catalysts supported on carbon materials have been proposed as the most efficient catalysts for the ammonia cracking reaction [14–18]. Among the different carbon materials studied, carbon nanotubes (CNTs) have demonstrated the best performance due to their outstanding electrical properties [16]. However, in addition to their high cost/performance ratio, CNTs are challenging to deposit inside the hollow fibre due to their low-density sponge structure [19]. Although, some impregnation methods allow the deposition of powder catalyst inside the hollow fibre support (i.e. dip coating and infiltration) [20], the sol-gel Pechini impregnation method is preferred as it results in the catalyst being homogeneously dispersed [12]. Among all carbonaceous catalyst supports proposed for the ammonia decomposition reaction [15,21], carbon xerogels not only have been proven to be efficient catalyst supports but they also offer a unique opportunity for addressing this challenge. Due to their synthesis method, carbon xerogels could be deposited in the hollow fibre substrate via sol-gel method and successively impregnated with the catalyst solution.

An ammonia-fuelled vehicle demands an affordable, efficient and compact technology for on-board hydrogen production. The aim of this work was to study the feasibility of a pioneering technology for on-board hydrogen production: the Ammonia Cracking Hollow Fibre Converter (HFC), which is composed of many HFR units. This work has been carried out at four different levels. At the material synthesis level, we have synthesised a series of ruthenium-based catalysts supported on carbon xerogels and studied their catalytic activity during the ammonia cracking reaction. At the coating process level, we have deposited for the first time carbon xerogels thin films in a 4-channelled hollow fibre substrate via sol-gel method, followed by wet impregnation with the catalyst solution. To the best of our knowledge this approach has not been reported before. At the reactor level, the performance of the HFR has been compared
with that of a traditional PBR during the ammonia cracking reaction. Finally, at the feasibility study level, both PBR and HFC have been designed for automotive applications, and compared in terms of volume, catalyst loading and efficiency, in order to suggest the most suitable catalytic reactor for on-board hydrogen production.

2. Experimental method

2.1. Synthesis and characterisation of the catalyst

Carbon xerogels were synthesized by the conventional sol-gel approach originally proposed by Pekala [22], using formaldehyde and resorcinol as starting materials. In order to improve their basicity, electrical conductivity, and thermal stability, two different post-synthesis activation treatments were carried out: i) 800°C, 5h, 100 mL/min (STP) of CO₂, and ii) 350°C, 3h, 100 cm³/min (STP) of NH₃:Air = 1:3. The activated supports were labelled as ACX and NCX, i) and ii) treatment, respectively, whereas the non-activated support was labelled as CX.

The supports were characterised by Scanning Electron Microscopy (SEM) (Crossbeam 550, Zeiss), Temperature Programmed Desorption (TPD) in inert atmosphere (iQ autosorb, Quantachrome and HAL-201, HIDEN ANALYTICAL). Thermogravimetric analysis in nitrogen atmosphere (N₂-TGA) (TGA/DSC 3+, METTLER TOLEDO) and N₂ adsorption/desorption isotherms at -196°C (iQ autosorb, Quantachrome).

Ruthenium-based catalysts were synthesized by incipient wetness impregnation of the carbon supports with a solution of Ru(NO)(NO₃)₃ in order to obtain 1.5 wt.% of metal loading.

The catalysts were characterised by Temperature Programmed Reduction (H₂-TPR), using the same apparatus used for TPD experiments, and Transmission Electron Microscopy (TEM) (TEM-1400 Plus, Jeol).

In order to obtain reliable kinetic data, the reaction conditions (i.e. pellet size 125 µm to 250 µm and reactant gas flowrate = 100 cm³/min (STP)) have been optimised to achieve a Thiele modulus $\phi^2 = 0.4$, avoiding mass transfer limitations.

4-channelled asymmetric $\alpha$-Al₂O₃ hollow fibre substrates were prepared using the phase inversion technique, followed by sintering at high temperatures [23]. The impregnation of a 10 cm long $\alpha$-Al₂O₃ hollow fibre substrate was carried out in two stages: deposition of the carbon xerogel in the hollow fibre substrate via sol-gel method and successive wetness impregnation with the catalyst solution. In particular,
after the impregnation with the precursor xerogel solution, the hollow fibre was subjected to pyrolysis and activation treatment in NH$_3$:Air = 1:3 atmosphere. The amount of carbon deposited in the hollow fibre substrate was calculated by measuring the difference in weight of the hollow fibre before and after impregnation with the precursor xerogel solution and its subsequent high temperature treatment, in order to determine the volume of Ru(NO)(NO$_3$)$_3$ solution required to achieve loading of 1.5 wt. % ruthenium catalyst.

The morphology and composition of the hollow fibre substrate after catalyst deposition were studied using Scanning Electron Microscopy (Crossbeam 550, Zeiss) and Energy-dispersive X-ray Spectroscopy (X-Max 150, Oxford Instruments), respectively.

2.2. Catalytic activity

Catalytic activity during the NH$_3$ decomposition reaction was measured in both PBR and HFR, using 300 mg and 30 mg of in-situ reduced catalyst (400°C, 1 h, 25 cm$^3$/min (STP) of H$_2$), respectively. Reactions were carried out between 200°C and 500°C, at atmospheric pressure, flowing 100 cm$^3$/min (STP) of 10% vol. NH$_3$ in Ar. Notice that due to health and safety in the lab, concentrated toxic gas cylinders (e.g. pure NH$_3$) are avoided when possible.

The concentration of reactants and products was measured over the duration of the experiment using an in-line mass spectrometer (QMG 250 PrismaPRo – Pfeiffer Vacuum). The ammonia conversion was defined as follows:

$$X(\%) = \frac{[NH_3]_{in} - [NH_3]_{out}}{[NH_3]_{in}} \times 100 \quad (1)$$

The reaction rate constant ($k$) for the ammonia decomposition reaction in the PBR and HFR was calculated under differential conditions (i.e. gradient-less ammonia concentration along the reactor): $280°C \leq T \leq 300°C$ and $x_{NH_3} \leq 2.5\% \pm 0.2$.

Considering the ammonia decomposition reaction as irreversible and following first order kinetics, $k$, at different temperatures was calculated as follows:

$$k = \frac{r_{NH_3}}{C_{NH_3}} \quad (2)$$

Where $r_{NH_3}$ is the rate of reaction and $C_{NH_3}$ is the ammonia concentration.
3. Results and discussion

3.1. Supports characterisation

SEM images of the carbon xerogels studied are shown in Figure 1A. It can be seen that activation treatments resulted in carbon xerogels with different textural and structural properties. For instance after the activation with carbon dioxide the surface becomes more smooth, whereas activation with ammonia resulted in a more crystalline and shiny surface. This was confirmed by nitrogen adsorption/desorption isotherm results, reported in Table 1. Both ACX and NCX presented lower surface area and a total pore volume than CX. With respect to ACX, this phenomenon could be explained due to the partial crystallisation of the support, which led to a more ordered structure. However, the higher crystallinity observed in NCX can be explained due to the formation of new nitrogen functional groups and the addition of heteroatoms into the carbon lattice. This outcome agrees with previous studies, in which it has been observed that the surface area and pore volume of carbon xerogels is affected by the order of their carbonaceous structure [24].

CO₂ and CO profiles during TPD experiments, displayed in Figure 1B, show that the amount of oxygen surface groups present in CX decreased after both activation treatments. In this respect, the CO₂–TPD profile exhibited three peaks, which can be ascribed to: i) carboxylic acid functions (i.e. 230-300°C), ii) anhydride functions (i.e. 410-420°C), and iii) lactone functions (i.e. 530-580°C). However, the CO–TPD profile exhibited only one peak (i.e. 410-420°C), which can be attributed to anhydride functions [2,16,17,25].

Particularly, TGA experiments, reported in Table 1, show that ACX and NCX presented 9 wt.% and 11 wt.% less oxygen surface groups than CX, respectively. For ACX, this phenomena can be explained due to the longer exposure to high temperature during activation treatment (i.e. 800°C, 5h) [25]. On the other hand, the absence of lactone surface groups in NCX suggests that the decrease of oxygen surface groups in this case is related to the formation and the desorption of amide surface groups during ammoxidation (i.e. $R_1$-(C=O)-O-$R_2$ + NH₃ $\rightarrow$ $R_1$-CONH$_2$ + $R_2$-OH) [25,26]. Figure 1C shows a schematic cartoon that attempts to illustrate the surface evolution of CX during carbon dioxide and ammonia post-synthesis activation treatments.

Table 1. BET surface area, pore volume and amount of oxygen surface groups (wt.%) presented by the carbon xerogels studied.
Support & SBET (m²/g) & Pore volume (cm³/g) & Oxygen surface groups (wt.%) &
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th>Total</th>
<th>Carboxylic</th>
<th>Anhydrides</th>
<th>Lactones</th>
</tr>
</thead>
<tbody>
<tr>
<td>CX</td>
<td>1050 ± 1%</td>
<td>0.4 ± 0.1%</td>
<td>14.0± 0.1</td>
<td>3.5± 0.1</td>
<td>4.3± 0.1</td>
<td>6.2± 0.1</td>
</tr>
<tr>
<td>ACX</td>
<td>960 ± 1%</td>
<td>0.3 ± 0.1%</td>
<td>2.9± 0.1</td>
<td>2.0± 0.1</td>
<td>0.7± 0.1</td>
<td>0.2± 0.1</td>
</tr>
<tr>
<td>NCX</td>
<td>325 ± 1%</td>
<td>0.2 ± 0.1%</td>
<td>2.4± 0.1</td>
<td>1.6± 0.1</td>
<td>0.7± 0.1</td>
<td>0.1± 0.1</td>
</tr>
</tbody>
</table>

3.2. Catalysts characterisation and performance study

From the TEM images of the catalysts studied are shown in Figure 1C, the average metal particle size was determined by counting 500 particles, which were assumed to be spherical. Comparing the ruthenium particle size distribution of Ru-CX with that of Ru-ACX and Ru-NCX, it can be seen that the average particle size decreases after both post-synthesis activation treatments (i.e. 2.4 nm, 2.0 nm, and 1.9 nm, respectively). The fact that both ACX and NCX possessed less acidic oxygen surface groups (i.e. carboxylic, anhydrides and lactones) than CX suggested that the electron-withdrawing effect of these groups, responsible for metal particle agglomeration, is weaker for ACX and NCX, leading to a higher ruthenium dispersion [27]. In the specific case of NCX, this result is indicative of a beneficial effect of nitrogen doping improving metal particle dispersion. It has been reported that nitrogen groups can improve both the stabilisation and dispersion of metal particles on the carbon surface, acting as an anchoring centre for metal particles [28].

Catalytic activities during the NH₃ decomposition reaction are shown in Figure 1D. Among all catalysts studied, Ru-NCX exhibited the highest catalytic activity. This behaviour can be explained by: i) the lower amount of oxygen surface groups presented by NCX compared to CX [15], ii) the promoting effect of N atoms, which improves the electron transfer between the support and ruthenium particles [29], and iii) the highest dispersion of metal particles. Likewise, the lower amount of oxygen surface groups presented by ACX compared to CX was beneficial for the ammonia decomposition reaction [27]. Furthermore, the development of the micro porosity after the CO₂ activation treatment stabilized larger ruthenium nanoparticles (i.e. ~2.0-2.1 nm for Ru-ACX), leading to higher catalytic activities compared to smaller nanoparticles (i.e. ~1 nm for Ru-CX). Figure 1F shows a schematic diagram of the electrons transfer mechanism in presence of the basic and acidic surface functionalities. In this regards, it has been reported that nitrogen atoms in the carbon lattice enhance the local basicity and electron density of the support, which improve
the catalytic activity during the ammonia decomposition reaction [30].

Figure 1. Carbon xerogel supports: A) SEM images, B) CO₂ and CO TPD profiles, C) Schematic cartoon of the evolution of CX’s surface upon carbon dioxide and ammonia post-synthesis activation treatments, Ru-based catalysts: D) TEM images and Ru particle size distribution histograms, E) Catalytic performance during the NH₃ decomposition reaction, F) Schematic diagram of the electrons transfer mechanism in presence of acidic and basic surface functionalities.

3.3. Hollow fibre characterisation and performance study

Images of the hollow fibre unit, at different stages of the experimental procedure, are shown in Figure 2A. The hollow fibre shows homogeneous colouration after the sol-gel impregnation with the precursor xerogel solution, as well as after pyrolysis and NH₃ activation treatments, proving the efficiency of this impregnation method.

SEM images showing the cross-section of the 4-channelled hollow fibre, at different magnifications, are reported in Figure 2B (i-iii). An asymmetric pore structure, consisting of two finger-like regions (orange, approximately 80% of the cross-section area) separated by a sponge-like porous region (white, remaining 20% of the cross-section area), can be identified in Figure 2B (ii). The finger-like region is comprised of hundreds of conical micro-channels (i.e. main entrance 0.1-1 µm and length 80-100 µm) perpendicularly distributed around the four lumens and the outer surface of the fibre, whereas the sponge-like region in between finger-like regions consists of a porous α-Al₂O₃ layer (i.e. pore size 0.1-1 µm).

EDX surface mapping images, in Figure 2B (iv), showed that the morphology of the hollow fibre and the use of a precursor liquid solution results in the catalyst being
uniformly dispersed. This impregnation method, in combination with the geometry of the support overcome the major limitations faced by traditional monoliths, such as the accumulation of catalyst on the walls of the substrate [13]. Figure 2C shows a schematic representation of the impregnation of the Al₂O₃ hollow fibre with the Ru-NCX catalyst. The process can be divided in three major steps: (i) two-step polymerization process through the addition reaction of resorcinol and formaldehyde, followed by the polycondensation of the hydroxymethyl derivative and its gelation and drying, (ii) carbonisation and ammoxidation of the organic xerogels and consequent formation of oxygen surface groups and nitrogen functionalities, (iii) catalyst deposition via incipient wetness impregnation.

Figure 2. A) Cross-section of the hollow fibre at different stages of the experimental procedure. B) SEM images (i-iii) of the hollow fibre substrate and EDX surface mapping (iv) after the impregnation with Ru-NCX. C) Schematic diagram of the deposition of Ru-NCX on the Al₂O₃ hollow fibre via sol-gel method and incipient wetness impregnation.
The beneficial effect of using the HFR on the catalytic activity is shown in Figure 3A and Table 2. For instance, it can be seen in Figure 3A that at 500°C, the rate of reaction in the HFR is 4 times higher than that in the PBR (i.e. $r_{NH3} = 6.1 \times 10^5$ molNH$_3$/m$^3$·h·g$_{cat}$ and $r_{NH3} = 1.5 \times 10^5$ molNH$_3$/m$^3$·h·g$_{cat}$, respectively). This result can be ascribed to the unique geometry of the hollow fibre substrate, which improve the efficiency of the catalytic process due to a combination of factors: i) uniform distribution of the catalyst on the substrate, ii) narrower residence time distribution, avoiding typical issues presented by packed bed reactors (i.e. recirculation, generation of preferential paths and stagnant regions), and iii) minimised internal and external diffusion limitations, resulting in improved reaction kinetics [11,12]. In this respect, it is worthy to highlight that the kinetic constant for the HFR at the operation conditions of a car with a power demand of 100 kW (i.e. $T = 450°C$ and $P = 1$ atm), is 4.6 times higher than that for the PBR (i.e. $7.4 \times 10^4$ 1/h·g$_{cat}$ and $1.6 \times 10^4$ 1/h·g$_{cat}$, respectively), see Table 2.

Table 2. Reaction rate constant ($k$) for the ammonia decomposition reaction in the PBR and HFR at differential conditions (*) and at the operation conditions of a car with a power demand of 100 kW calculated using the Eq. (S.1) (**).

<table>
<thead>
<tr>
<th>T [°C]</th>
<th>$r_{NH3}$ [molNH$_3$/m$^3$·h]</th>
<th>$k$ [1/h]</th>
<th>$r_{NH3}$ [molNH$_3$/m$^3$·h]</th>
<th>$k$ [1/h]</th>
</tr>
</thead>
<tbody>
<tr>
<td>280*</td>
<td>$3.9 \times 10^2$</td>
<td>$9.7 \times 10^2$</td>
<td>$3.3 \times 10^2$</td>
<td>$8.0 \times 10^2$</td>
</tr>
<tr>
<td>290*</td>
<td>$5.2 \times 10^2$</td>
<td>$1.3 \times 10^2$</td>
<td>$4.0 \times 10^2$</td>
<td>$9.8 \times 10^2$</td>
</tr>
<tr>
<td>300*</td>
<td>$7.1 \times 10^2$</td>
<td>$1.7 \times 10^2$</td>
<td>$5.4 \times 10^2$</td>
<td>$1.3 \times 10^2$</td>
</tr>
<tr>
<td>450**</td>
<td>$1.9 \times 10^4$</td>
<td>$4.8 \times 10^3$</td>
<td>$9.1 \times 10^3$</td>
<td>$2.2 \times 10^3$</td>
</tr>
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</table>

As can be seen in Figure 3B, the HFC offers the advantage of a significantly lower pressure drop compared to the PBR. The detailed procedure for the pressure drop calculation for both reactors can be found in the supplementary information. Furthermore, at comparable pressure drop, the HFC exhibits a smaller size than traditional monoliths.

In addition, as shown in Figure 3C, the HFC involves smaller volume and less catalyst compared to a traditional PBR. For example, an automobile with a power demand of 100kW, which requires 72 m$^3$/h (STP) of hydrogen supply [31], would require either a HFC of 11.5 L, or a PBR of 54 L, requiring 3.5 kg and 16.2 kg of catalyst, respectively. The detailed procedure for the design of both reactors can be found in the
supplementary information. The results of this work will underpin the design and development of novel reactors for on-board hydrogen production for automotive applications, a market that is expected to reach around USD 26 million by the end of 2025, at a compound annual growth rate of approximately 11% [32].

Figure 3. A) Ammonia cracking reaction rate vs temperature in the PBR and the HFR, B) Relationship between the size of different catalytic reactors and their pressure drop per length, C) Design of a HFC and PBR for a car with a power demand of 100 kW (i.e. 72 m³/h (STP) of hydrogen supply).

4. Conclusion

This work provides critical information to advance the uptake of green ammonia as an alternative to traditional carbon-based automotive fuels. Thus contributing towards the decarbonisation of our energy system during the transition towards 100% renewable energy. The Ammonia Cracking Hollow Fibre Converter opens the door for a new line of catalytic converters, which will be smaller (i.e. 80% less volume), cheaper (i.e. 80 wt.% less precious metal-based catalyst) and more efficient (i.e. more efficient mass transfer, improved residence time distribution and reduced pressure drop) than traditional catalytic packed bed reactors.

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