Molybdenum/Bismuth Based Mixed Metal Oxide Catalysts for Selective Propylene Oxidation and Zeolite Membrane Protected Palladium/Alumina Catalysts for Selective Carbon Monoxide Oxidation and Application in a Process Loop Using a Propane Feed

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As part of a process loop to make acrylic acid from propane, catalysts for two reactions have been investigated. Mo/Bi based mixed metal oxides for selective oxidation of propylene in a propane feed have been prepared by a sol-gel method which increased activity over the standard catalyst. Modifications of the molecular formula led to an increase in selectivity towards acrolein whilst maintaining the improved conversion. X-ray diffraction (XRD) analysis showed the sol-gel method helped incorporate the various mixed metal oxides and prevented segregation during reaction conditions. A Pd-loaded 4A zeolite membrane catalyst was developed on the surface of 1 mm beads γ-Al_2O_3 support. The membrane was employed in the selective oxidation of CO in a propane-rich mixture. The zeolite coating was developed by using a new modification of the hydrothermal synthesis (dilution method). Catalytic test results confirmed that the selective oxidation of CO in the presence of propane is possible by using a 4A zeolite membrane coated on a Pd based catalyst. Temperature and time during zeolite preparation are key parameters in the stability and reproducibility of the zeolite membrane. The seeding method improves the growth of the zeolite on the catalyst surface and does not affect the stability and reproducibility of catalyst. By using this catalyst it is possible to get a temperature window for the selective oxidation of CO (65ºC).

Introduction

CARENA (CAtalytic membrane REactors based on New mAterials for C1-C4 valorization) was a four-year Seventh Framework Programme (FP7) funded EU project carried out between 2011 and 2015 with a consortium consisting of 18 members. The partners were a mix of universities, research centres and industrial companies. The purpose of the project was to investigate several areas of industrial interest where current technology prevents commercialisation and to couple new catalysts with new membranes with the aim of improving the state of the art for these processes. Each partner would bring their own area

of expertise, i.e. catalysis, membrane development, modelling, reactor design, and characterisation.

The project was split into six main Work Packages. There were three reaction based Work Packages which aimed to develop and improve specific reactions:

- WP1 investigated methane activation. More specifically steam methane reforming (SMR), oxidative coupling of methane (OCM), and direct methane to methanol (MTM)
- WP2 developed a new process loop for the production of acrylic acid from propane. This included propane dehydrogenation (PDH), selective oxidation of propylene and selective oxidation of CO
- WP3 investigated the potential use of CO_2 as a reactant. The two reactions researched were CO_2 + H_2 to methanol, and CO_2 + methanol to dimethyl carbonate.

There were also three transversal Work Packages. These aimed to investigate more general research and methodologies that could be applied to all three of the reaction Work Packages:

- WP4 was the membrane toolbox. This investigated new high risk membranes which could be applicable in WP1–3 and also new ways to characterise membranes
- WP5 was the catalyst toolbox. The work in this WP concerned new catalyst designs, kinetic studies, mechanistic studies and characterisation
- WP6 was the modelling toolbox. The work here pertained to reactor modelling and the integration of membranes with catalysts.

One of the primary obstacles the project had to overcome was the disparity between catalyst and membrane operating conditions that often existed. As such there was a special focus within the project to develop new materials that were active at new temperatures to accommodate integrated catalytic membrane reactors for the processes investigated.

The focus of this paper is Work Package 2 and the work carried out by Johnson Matthey within it. WP2 investigated a new process loop for propane to acrylic acid (**Figure 1**) (1). Traditionally after propane dehydrogenation the resulting propane/propylene mixture has to be separated and the propylene then diluted in N_2 down to ~8%. This is then selectively oxidised to acrolein and then acrylic acid. Within CARENA the aim was to remove the separation and dilution steps and retain propane as the carrier gas for propylene during the selective oxidation steps. After removal of acrylic acid the propane can then be recycled.

The use of propane has the added benefit that the amount of propylene in the feed can be doubled to ~15%. This is because propane has a higher heat capacity and the exothermicity can be more easily controlled. A selective CO oxidation unit has been included as a buildup of CO in the loop could poison the propylene oxidation catalysts.

Johnson Matthey had two main roles within this Work Package. The first was to develop and test novel catalysts for the selective oxidation of propylene into acrolein. During this period Johnson Matthey Technology Centre, Sonning Common, UK, supplied catalysts developed on site. New synthesis methods were investigated for catalyst preparation and a new test rig was constructed so testing could be achieved in the correct gas composition. The second part of Johnson Matthey's input was working on a novel membrane catalyst for selective CO oxidation in a propane feed. Here the goal was to combine the catalyst and membrane into a single piece which could distinguish the CO from the propane and hence only combust the CO into CO_2 without converting the remaining propylene and propane. The membrane catalyst preparation was based on previous work (2–4). However the original preparation method was not reproducible and work concentrated on modifying the reaction conditions to produce a stable reproducible catalyst which gave a good separation and excellent selectivity. The material may also have other applications for gas separation and catalysis in mixed gas streams. The objective was to scale-up the membrane catalyst for testing in a demonstrator unit.

Propylene oxidation was to be carried out with pure oxygen on a pilot scale unit (at a scale of about 1–3 kg h^{-1}) where the catalyst arrangements are designed to operate at a conversion above 95% and at an acrylic acid yield above 85%. The reaction should benefit from other positive effects of the alkane rich ballast gas, which has a much higher specific heat capacity. This allows a higher heat transfer to the gas phase, a better control of the reactor temperature and thus a higher productivity, or a higher selectivity. This reduces either capital expenditure or operating cost.
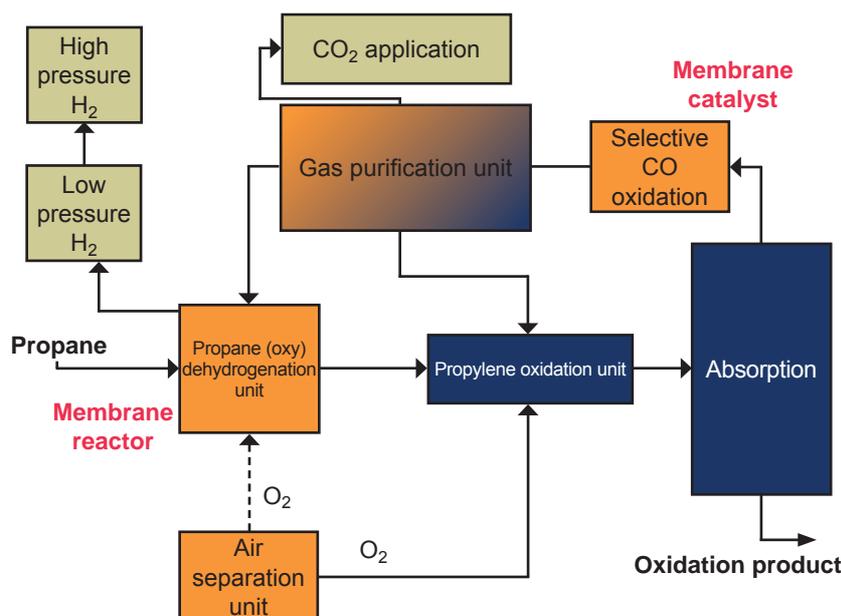
Fig. 1. Proposed process scheme for propane to acrylic acid (1)

Experimental

Propane to Acrolein: Synthesis, Characterisation and Testing

Catalysts were prepared using either slurry or a sol-gel preparation method. The slurry method reported previously was used to prepare the standard catalyst for testing (5). The sol-gel method entailed dissolving ethylene glycol and citric acid in water and heating at 60ºC until a gel was formed. The nitrates were dissolved in water. The bismuth nitrate required some extra nitric acid to dissolve fully. The ammonium molybdate and tin chloride were dissolved separately in water. The nitrates were added to the gel first followed by the chloride and then the molybdate which initiates precipitation of the product. Tungsten oxide powder was then added to the mixture. The temperature was maintained at 60ºC for several days until the majority of water had been evaporated. The resulting paste or gel was dried at 140ºC for 12 h and calcined at 500ºC for 6 h.

The catalysts were characterised by their catalytic properties as well as by XRD and temperature-programmed reduction (TPR). XRD data were collected in reflection geometry over at least the 10º < 2θ <100º range, on a Bruker AXS D8 ADVANCE fitted with a Göbel mirror using Cu Kα (λ = 1.5406 + 1.54439 Å). The diffraction patterns were fitted in TOPAS with instrumental peak profiles determined experimentally using data collected from a NIST LaB$_6$ standard. TPR was performed on a Micromeritics AutoChem II 2920 chemisorption analyser using 5% H$_2$/N$_2$ at 30 ml min^{-1} up to 900ºC with a ramp of 10ºC min^{-1}.

Catalytic testing was performed in a continuous flow fixed bed reactor working at atmospheric pressure. 200 mg of catalyst, pelleted to 255–350 μm size fraction, was held in a quartz reactor (internal diameter 4 mm) by quartz wool plugs. A gas mixture of C$_3$H$_8$/C$_3$H$_6$/O$_2$/H$_2$O/N$_2$ = 23/7/10/6/4 at a flow rate of 50 ml min^{-1} was passed over the catalyst and the temperature was raised to 320ºC. The reaction products were analysed online on a Varian gas chromatograph fitted with a 1.5 M 1/8 2 mm 13X 80/100 molecular sieve, an Agilent J&W PoraPLOT for separation and two thermal conductivity detectors and a flame ionisation detector for measurement.

Selective Carbon Monoxide Oxidation: Synthesis, Characterisation and Testing

A series of catalysts with different platinum group metal (pgm) loading was made by incipient wetness, using γ-Al$_2$O$_3$, 1 mm beads as supports. The alumina beads were coated with a zeolite 4A, as this zeolite has the correct pore size (~0.4 nm) to distinguish

CO and propane. The zeolite coating was developed by using a variation of a hydrothermal synthesis (2–4). The synthesis was modified (temperature, time and dilution) in order to improve the uniformity of the zeolite coating, thus increasing the operating window and making the zeolite synthesis more applicable in terms of scale up. The effect of seeding on the zeolite growth was also investigated. Seeding has been reported before to promote the uniformity of the zeolite layer (6). The solution was prepared using a commercial LTA nanozeolite with 100 nm particle size (4). The nanozeolite was dispersed in water in order to produce a very dilute solution. The final mixture was stirred at 40ºC for 30 min. Subsequently the $Pd/\gamma-Al_2O_3$ catalyst was dipped into the zeolite solution for different times (the seeding time was optimised by varying the time from 1 s to 5 min). Finally the seeded catalyst was dried at 140ºC overnight. For the hydrothermal synthesis, the catalyst beads were added to a zeolite gel which was prepared by rapidly adding a solution of sodium aluminate to a solution of sodium metasilicate. The catalyst containing gel was stirred at room temperature for 30 min and then hydrothermally crystallised at 60ºC. The $Pd/\gamma-Al_2O_3$ zeolite coated beads were separated from the crystallisation solution and washed with demineralised water, dried overnight, and then calcined at 500ºC.

A combination of catalytic tests, scanning electron microscopy (SEM) and XRD was used to complete understanding of the performance level and the characteristics of the coating on the catalyst. XRD of the bulk material gives a general idea of the extent of crystallisation which might be extrapolated into defining the crystallinity of the thin zeolite film. By SEM it is possible to have more information about the coating uniformity. XRD data were collected in reflection geometry over at least the 10º < 2θ < 100º range, on a Bruker AXS D8 fitted with a Göbel mirror using Cu Kα (λ = 1.5406 + 1.54439 Å). The diffraction patterns were fitted in TOPAS with instrumental peak profiles determined experimentally using data collected from a NIST LaB_6 standard. SEM analyses were conducted using a Zeiss ULTRA 55 Field Emission Scanning Electron Microscope. The sample powder was placed directly on SEM stubs. The samples were carbon coated prior to analysis to provide a conductive layer for charge dissipation.

A catalytic test was the primary method used to distinguish the best performing catalysts from the rest.

The 4 Å kinetic window of the zeolite was expected to selectively allow molecules of lesser dimension access to the active metal centres inside the macropores. Testing was conducted over uncoated and coated pgm catalysts. Catalytic evaluation was performed in a continuous flow fixed-bed catalytic reactor working at atmospheric pressure. 400 mg of catalyst were placed between quartz wool plugs in the centre of a cylindrical tube reactor (7 mm internal diameter). Internal and external temperatures of the catalyst reactor were measured by nickel-chromium thermocouples. The reaction was carried out by feeding a mixture of carbon monoxide/propane/oxygen = 0.5/25/1.0 and nitrogen as a diluent, at a total flow of 200 ml min^{-1}, gas hourly space velocity (GHSV) = 12000 h^{-1} and scanning reaction temperatures from room temperature to 300ºC. The reaction products CO and CO_2 were analysed online by a multi gas analyser from Sigma Instrumental provided with an infrared detector for CO and CO_2. Additional catalytic tests were carried out in order to evaluate the stability of the membrane catalysts at a set temperature (250ºC) for 5 hours.

Results and Discussion: Propylene to Acrolein

All the acrolein catalysts have been compared to the standard sent by Arkema (Mo/Bi/Fe/Co/Ni/Sn/W/Si/K) from Example 1 of the patent (5), labelled as AC. Initially the work focused on reproducing the Arkema standard. Several preparation methods were used, i.e. co-precipitation, evaporation and sol-gel. However, conversion was improved from 50% to 73% by the use of the sol-gel method. Selectivity was also improved, but only slightly, up from 79% to 84%.

A series of catalysts breaking down the Arkema composition to understand the relative importance and effect of various components was prepared by sol-gel and tested. **Figure 2(a)** shows the composition has a large influence on the conversion. The presence of nickel seems imperative for high conversions. Other elements as tungsten and tin also have a large positive effect. The effect of silicon is unclear and in certain cases even seems to reduce the conversion. Additionally, the effect of pgms on the catalytic performance was investigated. It was found that impregnating Pd onto the full composition had little effect on the conversion whereas rhodium actually had a negative effect.

Figure 2(b) shows the selectivities for the Arkema type catalysts. Again the Ni is necessary to achieve high selectivities and W also has a large positive effect
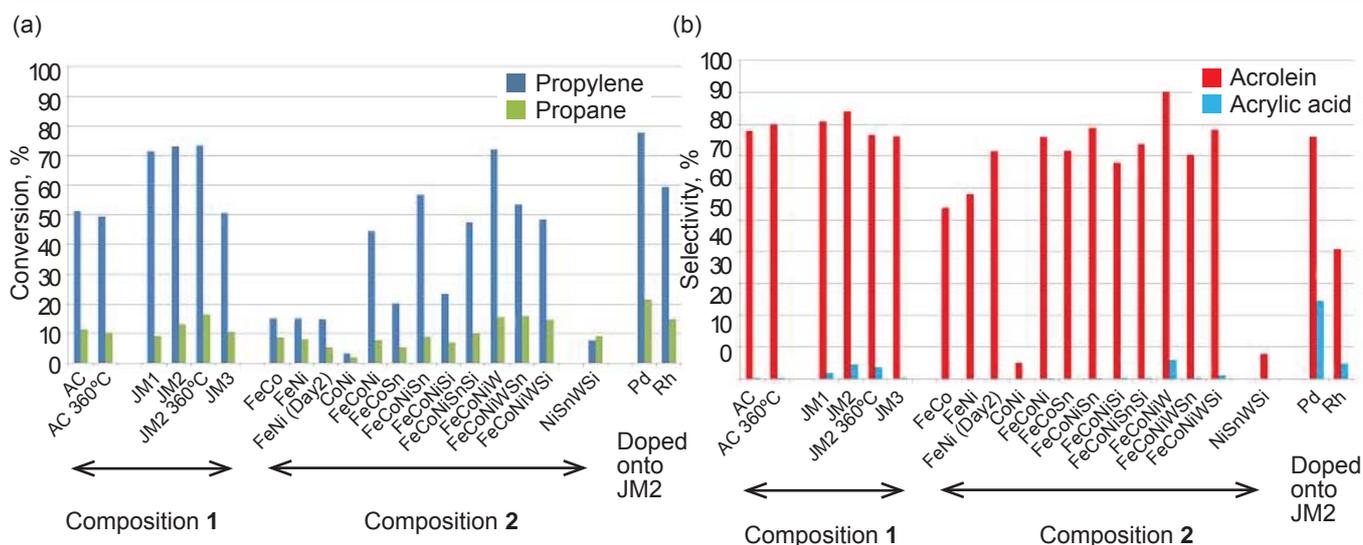
Fig. 2. Bismuth molybdate based mixed oxides for propylene to acrolein: (a) conversions of propane and propylene for Arkema type catalysts at 320ºC (unless stated); and (b) selectivities to acrolein for Arkema type catalysts at 320ºC (unless stated). Composition **1**: $BiMo_{12}Fe_{3.7}Ni_{2.6}Co_{4.7}Sn_{0.5}W_{0.5}K_{0.08}SiO_x$; Composition **2**: All contain $Mo_{12}BiK_{0.08}O_x$, other elements have molar quantities as in the Arkema composition if present

on the selectivities, whereas Sn has a small positive effect. Adding pgms (**Figure 3**) to the full composition has a negative effect on the selectivity to acrolein but in the case of Pd the amount of acrylic acid produced becomes significant (>20%). Over time the selectivity reduced at the expense of CO and CO_2 as coking of the catalyst increased.

Work was performed investigating new compositions of catalyst. This included many variations and

new active sites were investigated as well as new configurations of dopants for oxygen mobility. Of particular note were the catalysts with cerium, ceria and ceria/zirconia (**Figure 4**) added to the standard composition. The promoters were incorporated into the catalyst composition mixture by different methods. Introduction of nitrate precursors into the sol-gel mixture resulted in complete deactivation in some cases. However, incorporation of the Ce by addition
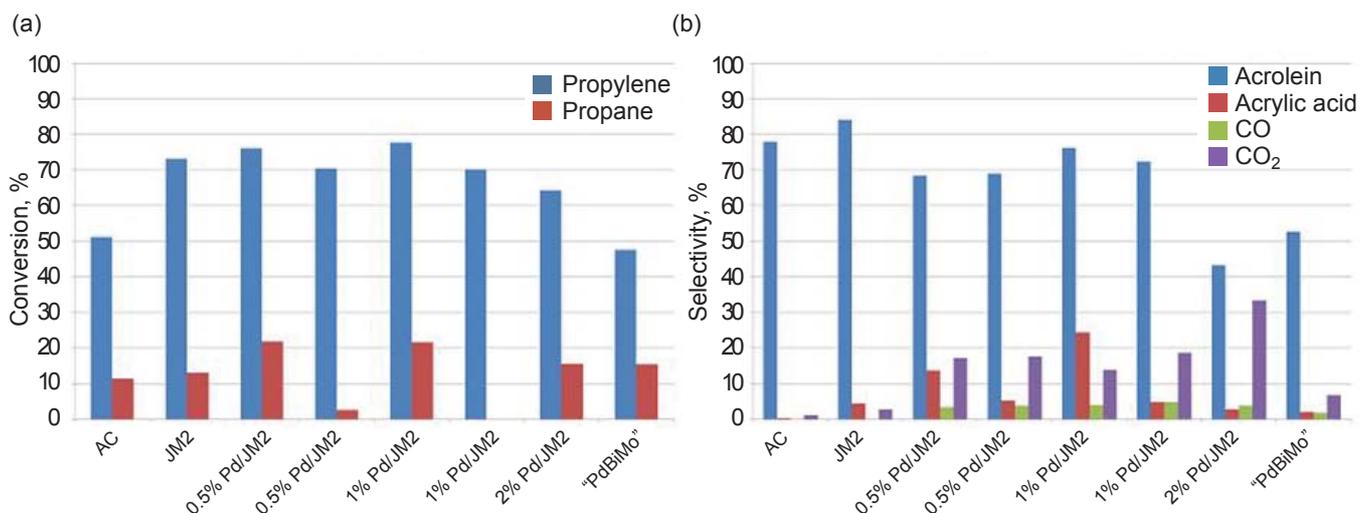

Fig. 3. (a) Conversion of propylene and propane samples based on the standard catalyst doped with Pd; (b) selectivity to acrolein and acrylic acid of samples based on the standard catalyst doped with Pd.
"PdBiMo" = $Pd_{0.205}BiMo_{12}Fe_{3.7}Ni_{2.6}Co_{4.7}Sn_{0.5}W_{0.5}K_{0.08}SiO_x$
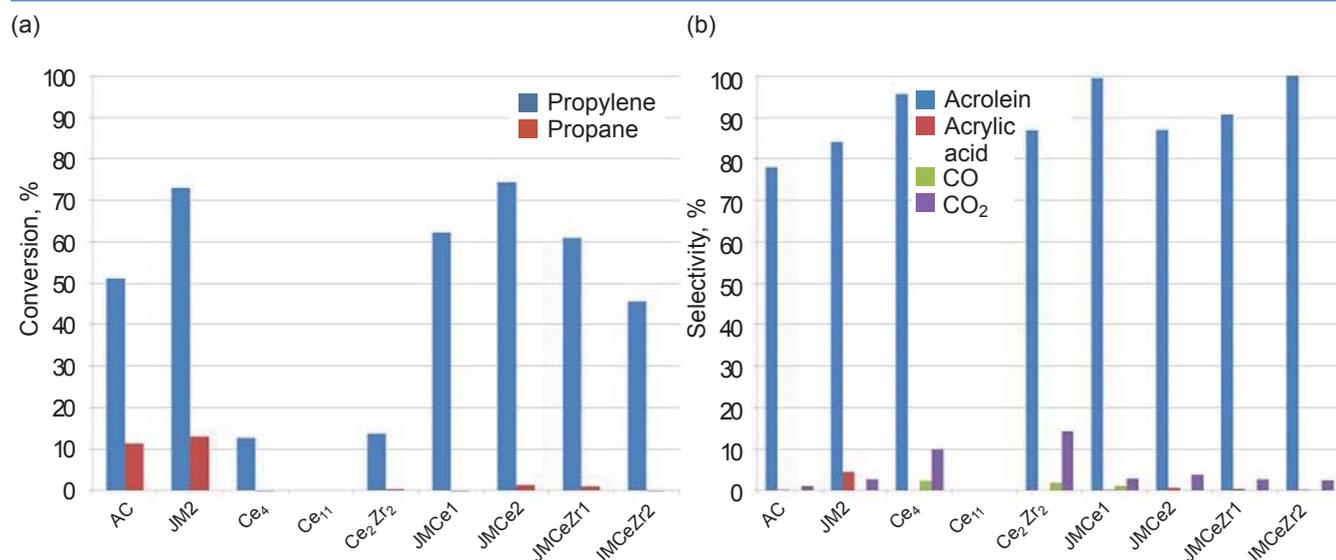
(a)

(b)

Fig. 4. Conversions and selectivities of samples incorporating Ce and CeZr: (a) conversion of propylene and propane of samples based on the standard catalyst with Ce or CeZr added; (b) selectivity to acrolein and acrylic acid of samples based on the standard catalyst with Ce or CeZr added

of preformed ceria resulted in an excellent catalyst (JMCe1) which displayed a conversion of 62% and very high selectivity >95% (**Figure 4**).

Work was also carried out on novel compositions based on molybdenum/bismuth/vanadium/cerium. A large series of catalysts was produced and tested. Mo/V ratio, Bi content and Ce content were all investigated; as was the effect of other dopants into the catalyst composition and preparation method. In general these catalysts were less active than the standard composition type catalysts and required elevated temperatures of 380ºC. The best catalyst showed a high selectivity of 90% but the conversion was only 30% even at 380ºC. Some catalysts in this series showed conversions above 40% but these had much lower selectivities.

The catalysts were then characterised to increase understanding of their behaviour. XRD characterisation of the best catalyst based on the Arkema composition (JM2) did not show the MoO_3 phase within the structure (**Figure 5**). AC on the other hand had a considerable amount of MoO_3 present. MoO_3 is known to be inactive in this reaction and its presence means not all of the Mo is being used to generate active sites. Additionally, XRD results after reaction of the AC showed a large proportion of the Bi_2Mo_x phase. This suggests bismuth molybdate is segregating out of the catalyst structure. Although bismuth molybdate is the active site, if it becomes segregated then it cannot be replenished with oxygen and the catalyst will deactivate. JM2 prepared by the sol-gel method shows a much smaller peak

which means the bismuth molybdate is remaining fully integrated in the catalyst structure. **Figure 6** shows clear CeO_2 peaks in the XRD of JMCe1 meaning the ceria does not incorporate into the bulk structure but probably aids transmission of oxygen from the gas phase into the bulk catalyst. The TPR shows an extra, large peak for the AC catalyst believed to be MO_3 which is not visible in the other samples.

Selective Oxidation of CO in Propane

The zeolite made by the modified hydrothermal synthesis was characterised with the aim of understanding the characteristics of the material. **Figure 7** shows the XRD results confirming the formation of a zeolite 4A-crystalline structure.

To begin the evaluation of the membrane catalyst, the first step was to test a Pd/γ-alumina uncoated catalyst in order to establish the CO and propane oxidation temperatures under reaction conditions. Catalytic performance shows that CO oxidation is complete by ~180ºC and propane oxidation is complete by ~130ºC (**Figure 8**) under these test conditions. This result confirms that there is an overlap in the CO and propane oxidation temperatures, and therefore the need for a membrane layer on the catalyst.

The next step was to test the catalysts coated by the zeolite prepared by the modified hydrothermal method (6), with the aim to fully investigate the separation properties of the zeolite membrane. For this, effects
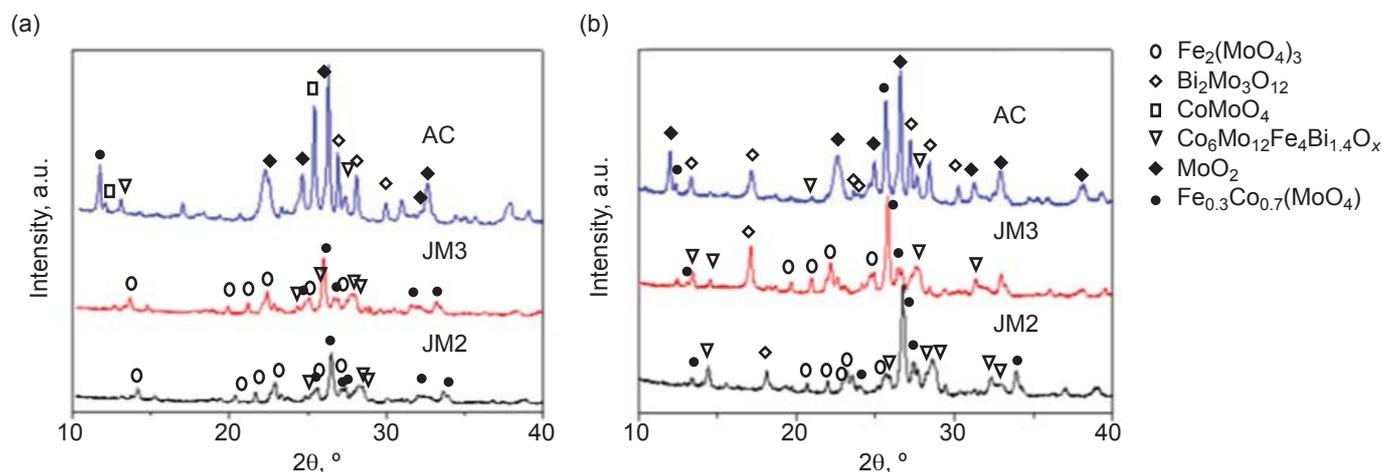
Fig. 5. XRD of standard composition catalysts prepared *via* different routes: (a) before reaction; (b) after reaction. AC is Arkema catalyst, JM3 is slurry method and JM2 is sol-gel method
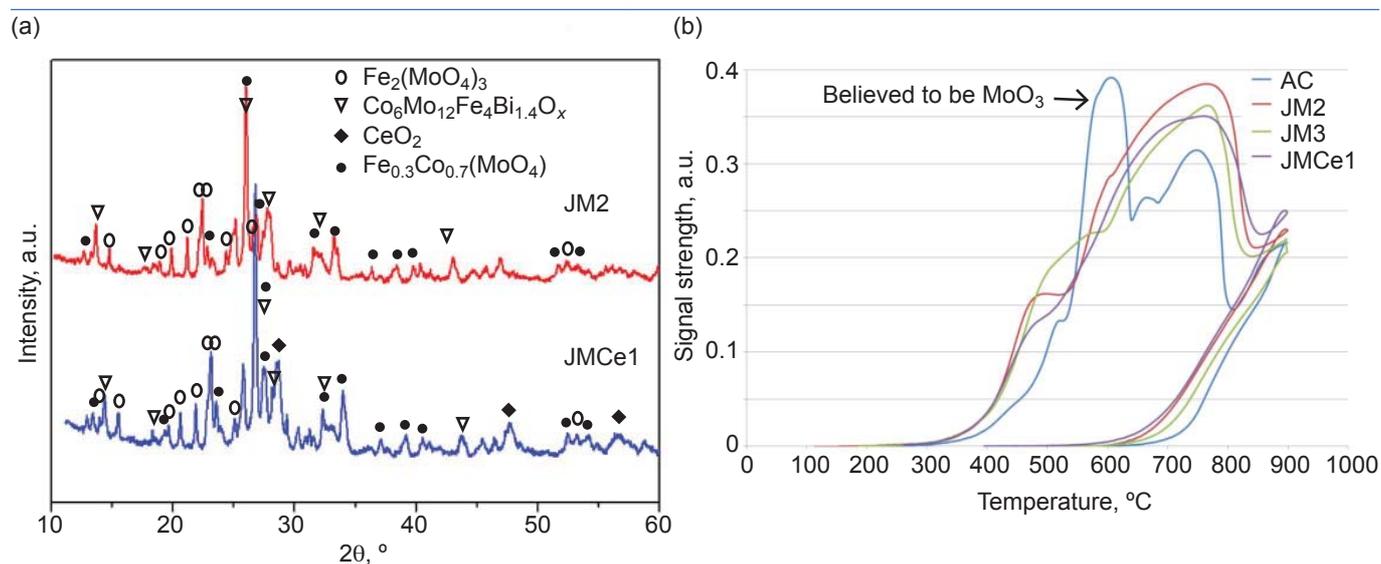

Fig. 6. (a) XRD comparison of standard composition and with ceria added; (b) TPR of different catalysts ramped to 900ºC at a rate of 10 ºC min^{-1} under 5% H$_2$ in N$_2$
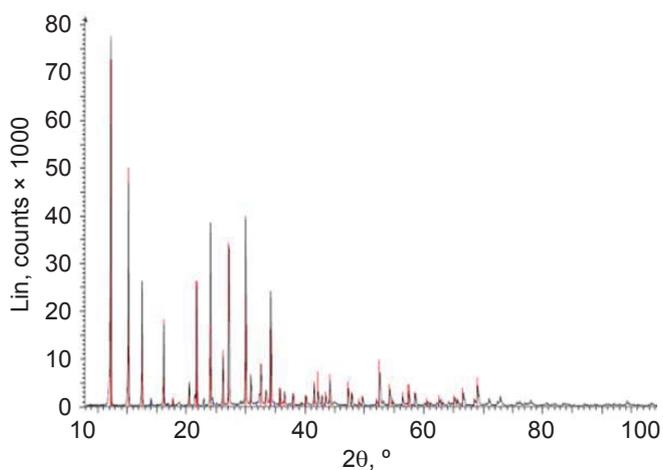

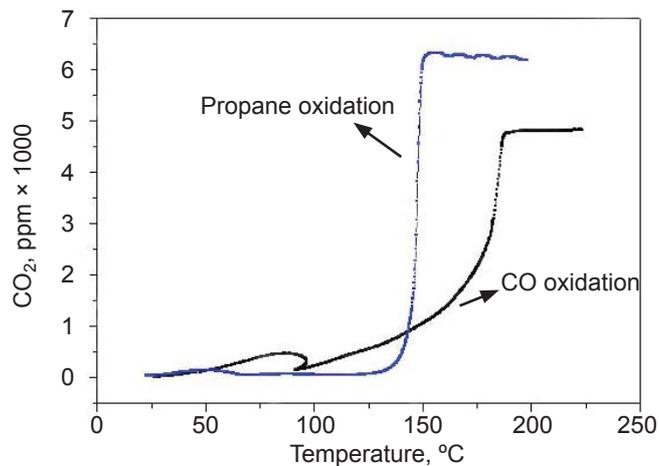Fig. 7. XRD diffractogram of the zeolite 4A synthesised hydrothermally. The result confirms the formation of 4A crystalline structure

Fig. 8. Selective CO oxidation in propane *vs.* temperature (pgm uncoated catalyst) CO/C$_3$H$_8$/O$_2$/N$_2$ = 0.5/25/1.0/balance, total flow of 200 ml min^{-1}, GHSV = 12,000 h^{-1}, T = 25–300ºC

of time and temperature on the zeolite structure were investigated. SEM results of the zeolite membrane catalyst prepared under different conditions (**Figure 9**) show a clear effect of temperature and time on the zeolite crystallinity. 3 h was not enough time for zeolite crystallisation at 60ºC. Material prepared under these conditions showed a spherical structure (**Figure 9(a)**). By increasing the synthesis time from 3 h to 6 h, a good cubic crystalline structure can be observed (**Figure 9(b)**). The degree of crystallinity of the zeolite could be improved by conducting the synthesis at 80ºC for 3 h. However, a longer synthesis time at 80ºC (6 h) led to a modification of the crystalline structure leading to a needle structure. This structure is not selective between CO and propane. Catalytic tests for selective CO oxidation in propane confirm these results (**Figure 10**). The zeolite membrane catalyst prepared at 60ºC for 6 h provided a higher operating window as shown later in the present article.

The effect of seeding on the zeolite coating was also investigated. SEM characterisation results of the membrane catalyst prepared by using the seeding step show a uniform layer of zeolite 4A formed over the catalyst (**Figure 11**). The images confirm the formation of a membrane catalyst where the active phase (PdO), γ-Al_2O_3 support, and a uniform zeolite structure can be differentiated.

Fig. 10. Effect of temperature and time on the selective CO oxidation in propane. $CO/C_3H_8/O_2/N_2$ = 0.5/25/1.0/balance, total flow of 200 ml min^{-1}, GHSV = 12,000 h^{-1}, T= 25–300ºC
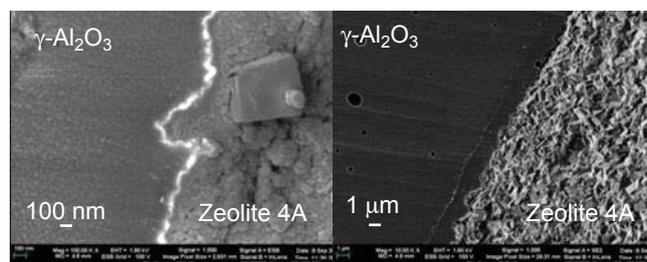
Fig. 11. Cross-section of the zeolite membrane catalyst synthesised hydrothermally

Characterisation results are in line with the catalytic behaviour where the seeded catalyst showed the best performance (7). The seeded membrane catalyst showed higher separation of CO from propane (50ºC *vs.* 60ºC). The performance was further improved after being fired at 500ºC for 2 h reaching an operation window (CO/propane separation) at 80ºC (**Figure 12**). The thermal process seems to improve the interaction between the zeolite coating and the supported catalyst, therefore improving the gas separation (8).

The stability of the zeolite membrane catalyst and the effect of propylene on the catalytic performance were also evaluated. At 230ºC for 5 h the catalyst showed only full CO conversion (no propane conversion). It is important to note that no deactivation was observed during this period of time (**Figure 13**). The results confirm that the membrane catalyst fulfils the requirements within the process loop. This process
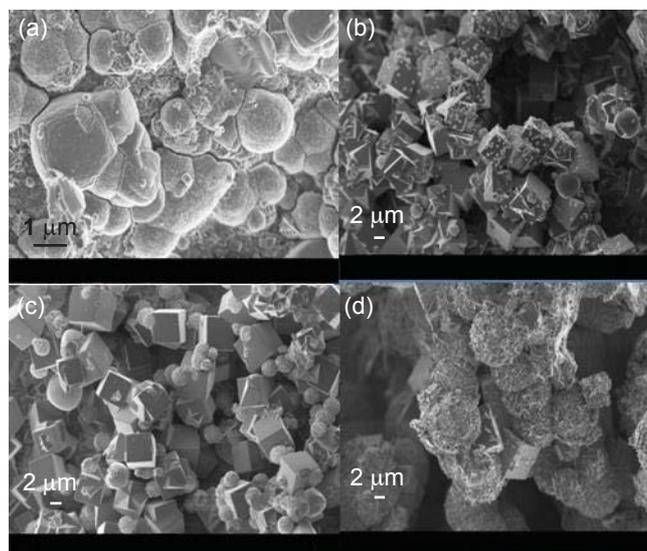

Fig. 9. SEM results of the zeolite membrane catalysts synthesised hydrothermally at different conditions (time, temperature): (a) 60ºC, 3 h; (b) 60ºC, 6 h; (c) 80ºC, 3 h; (d) 80ºC, 6 h
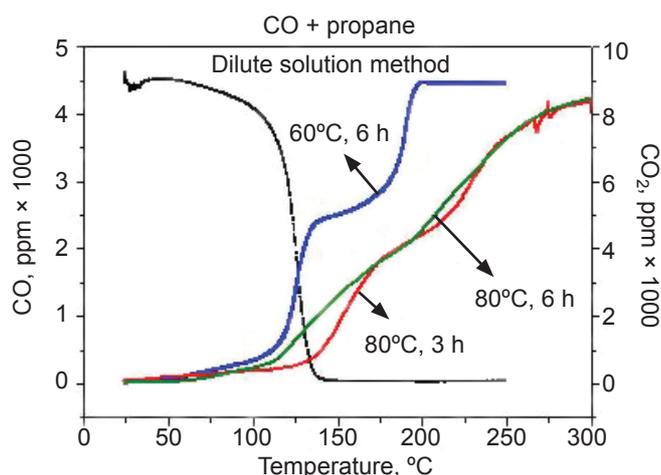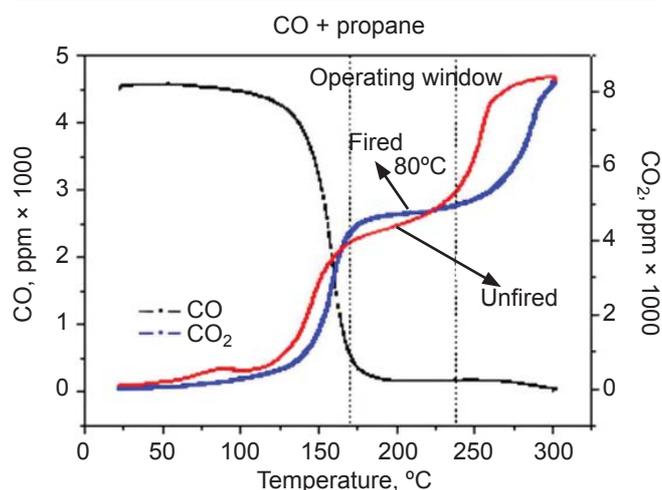
Fig. 12. Effect of seeding and calcination on the selective CO oxidation in propane. $CO/C_3H_8/O_2/N_2$ = 0.5/25/1.0/balance, total flow of 200 ml min^{-1}, GHSV = 12,000 h^{-1}, T= 25–300ºC
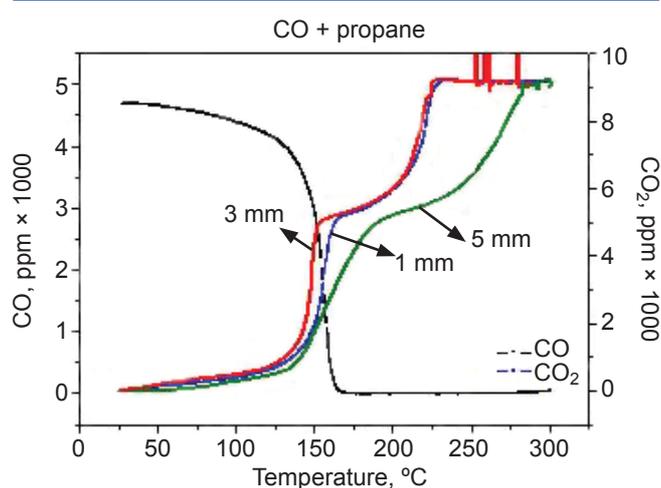
Fig. 14. Effect of the support size. $CO/C_3H_8/O_2/N_2$ = 0.5/25/1.0/balance, total flow of 200 ml min^{-1}, GHSV = 12,000 h^{-1}, T= 25–300ºC

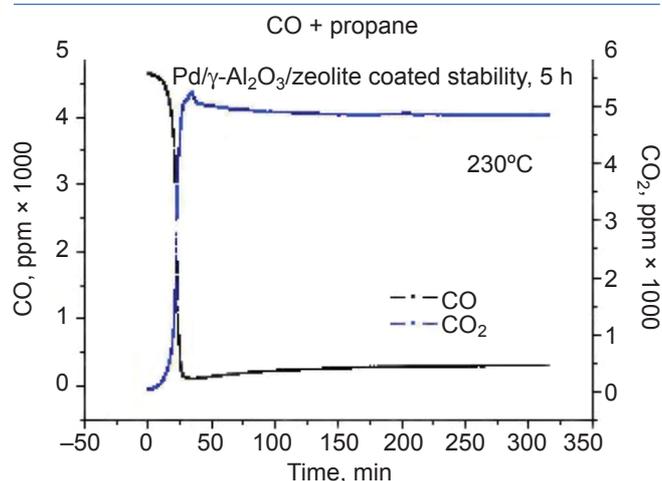Fig. 13. Stability of the membrane catalyst for selective CO oxidation in propane. $CO/C_3H_8/O_2/N_2$ = 0.5/25/1.0/balance, total flow of 200 ml min^{-1}, GHSV = 12,000 h^{-1}, T= 230ºC
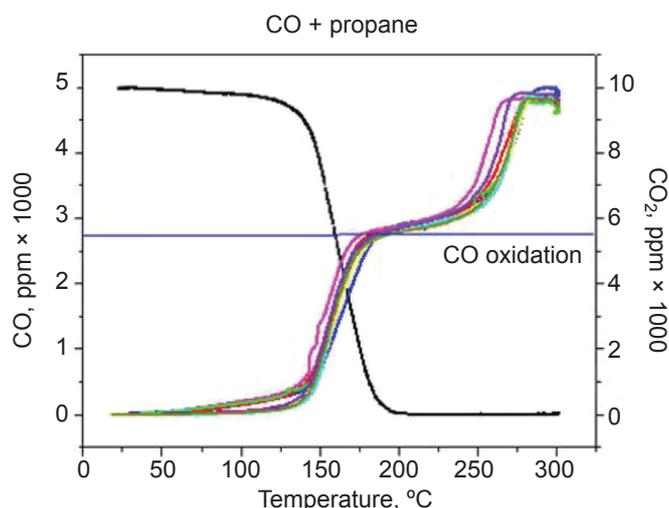

Fig. 15. Reproducibility of the zeolite coated catalyst

involves a partial oxidation of propane in propene at 250ºC.

For the scale-up process different size beads (1 mm, 3 mm and 5 mm) were coated and evaluated in the reaction. The zeolite coated catalysts showed some differences. The 1 mm and 3 mm coated beads (**Figure 14**) showed a similar catalytic behaviour, with a good separation between CO and propane. The 5 mm beads showed a more gradual oxidation over the temperature range, but still differentiated between the two gases (CO/propane). Since the catalytic performance of the zeolite membrane catalyst

synthesised by using the 5 mm beads was good in terms of separation, stability and reproducibility, the last step was to scale up the catalyst for the demonstrator unit. A batch of 0.5 kg of Pd/γ-Al$_2$O$_3$ (5 mm) beads zeolite membrane catalyst was successfully scaled up. This sample was sent to Arkema for evaluation in the pilot plant.

Conclusions

The use of new preparation methods and understanding of the properties of a complex catalyst has led to a new generation catalyst that has very high selectivity and competitive conversion over the state of the art

for the propylene to acrolein catalyst. The sol-gel preparation method gives a much improved catalyst which XRD showed is due to better incorporation of Mo in the catalyst structure creating more active sites for propylene oxidation. The incorporation of CeO_2 aided the reaction as CeO_2 is a known oxygen transport compound and oxygen availability at the active site is crucial for good catalytic activity. The manner in which the CeO_2 was introduced during the preparation method was also shown to be highly important.

Novel formulations were also investigated and although less active than the current state of the art catalysts their simple formulations would allow for greater research possibilities in the future.

Selective CO oxidation in the presence of propane is possible by using a 4A zeolite membrane coated on a Pd-based catalyst. The zeolite coating was developed by a modification of an existing hydrothermal method. Temperature and time are key parameters in the stability and reproducibility of the membrane catalyst. The best synthesis conditions are 60ºC for 6 h. It was also found that the seeding method improves the growth of the zeolite on the catalyst surface over previous methods. With these conditions an operation window of 80ºC between CO and propane was achieved. Stability and reproducibility of the membrane catalyst was not affected by the dilution method. The scale-up of the membrane catalyst to several hundred grams was successful. It was possible to achieve a good separation with a good reproducibility of the material. Further work however would be required to consider this process for full scale-up to kilo or tonnage scale. Several key points would need to be addressed including moving from hydrothermal to thermal conditions.

Acknowledgements

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