

Research article

Studies into the reduction of domestic fuel burning emissions by means of facile catalytic abatement technology

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Abstract

The negative health and socio-economic impacts of emissions associated with domestic fuel burning are widely recognized. Although there has been much progress in the provision of electricity to households in South Africa, many still rely on solid fuel sources such as wood and coal. While various investigations have been done on reducing household emissions by reducing the use of polluting fuels and improvements in combustion efficiency, comparatively fewer studies have been conducted on the reduction of emissions through use of abatement technology. Catalytic oxidation could be utilized to oxidize particulate matter precursors such as volatile organic compounds and soot particles to reduce secondary particulate formation. Although catalytic methods have not been effectively utilized in practical domestic applications, studies have shown effective soot reduction during laboratory testing. This study investigated the synthesis and use of a manganese oxide based catalyst to reduce particulate matter from domestic fuel burning stoves. The catalyst was synthesized onto a mesh substrate and inserted into the flue of the stove. During field testing, the presence of the catalyst increased the mass of particulate matter collected onto PTFE filters used for gravimetric analysis, with Scanning Electron Microscopy (SEM) analysis showing spherical particles in the pores of the filters used during the catalytic runs. The baseline runs had very few of these particle clusters. Energy Dispersive X-Ray (EDX) analysis of the catalyst run filters did not detect manganese, revealing that increased particulate concentrations were not as a result of macroscopic particles of the catalyst being dislodged from the support. Dislodgement of very small metal particles from the catalyst could, however serve as nucleation nodes for particle growth which would have a non-metal coating leading to the non-detection of manganese. The increase in particulate matter could also be caused by the impingement of particulate matter precursors on the catalyst followed by particle growth and dislodgement into the flue gas. The testwork showed that an active catalyst can be synthesized onto a mesh catalyst support in a relatively simple and cost-effective manner, which can be utilized in domestic fuel burning devices. It is recommended that a range of optimized, potentially active catalysts be tested to improve the oxidation of particulate matter precursors to carbon dioxide.

Keywords

household air pollution; particulate matter; domestic fuel burning; catalytic reduction

Introduction

Domestic fuel burning

The negative health and socio-economic impacts of emissions associated with domestic fuel burning are widely recognized. The Global Burden of Diseases Study estimated that household air pollution was responsible for 2.8 million deaths and 85.6 million disability adjusted life years (DALYs) globally in 2015 (Cohen et al 2017). The study concluded that household fuel

burning contributed significantly to mortality in low- and middle-income countries. The 1996 and 2011 census data indicate that the proportion of households making use of solid fuels decreased in South Africa during this period, however there remain pockets of households that are still reliant on solid fuel (Pauw et al 2020). Areas that experience the highest impact of domestic fuel burning would be densely populated with a high density of emission sources. The negative impacts of household emissions are exacerbated by the fact that they are released

in the breathing zone of individuals (DEA 2019). Globally, the greatest negative health impact of domestic energy use is from incomplete combustion of fuels in low efficiency stoves and lighting devices (WHO 2014).

Although there has been much progress in the provision of electricity to households in South Africa, many still rely on solid fuel sources such as wood and coal (Israel-Akinbo et al 2018). A study by Israel-Akinbo et al. (2018) examined how poor households transition from traditional energy carriers such as wood to modern energy carriers such as electricity, biofuels and liquefied petroleum gas. In the case of energy usage for heating purposes, households would not necessarily switch to modern fuels as income rises, in line with the energy stacking model, where households use a combination of energy carriers on the upper and lower stages of the energy ladder depending on their needs. An earlier study conducted by the then Department of Energy (presently the Department of Minerals and Energy) came to a similar conclusion as their results indicated that poorer households relied on multiple sources of energy regardless of electrification status, which further points to an energy stacking model rather than the energy ladder theory (DOE 2012).

A South African study analyzing the economic impact of various air quality initiatives found that technology interventions in the domestic sector would be the most efficient way to reduce healthcare costs associated with urban air pollution (Leiman et al 2007). The interventions that have been investigated in South Africa include the electrification of households, the use of alternative fuels, improved household energy efficiency and encouraging the use of top down fire ignition methods, amongst others (Matimolane 2017, Msibi and Kornelius 2017, Mdluli et al 2010, Wagner et al 2005, Scorgie 2012, Makonese 2015).

While various investigations have been done on reducing household emissions by reducing the use of polluting fuels and improvements in combustion efficiency, comparatively fewer studies have been conducted on the reduction of emissions using abatement technology.

The aim of this study was to investigate the potential of particulate matter emission reduction in the domestic sector using an appropriate abatement technology. The study thus investigated the emissions that are likely to arise from domestic fuel burning and the available catalytic and non-catalytic abatement technologies for the reduction of the particulate emissions reported in the literature. The study sought to identify, synthesise and test a suitable abatement technique to decrease particulate matter emissions from domestic fuel burning devices.

Implementation of abatement technologies

Previous studies

Investigations and research into the reduction in emissions from household appliances appears to have been largely focused on improving combustion efficiency and little information seems to

be available on secondary or end of pipe type measures to reduce emissions. The use of secondary measures to reduce emissions from small combustion sources in Europe was found to be limited due to the high cost and maintenance requirements of such installations which were limited to the use of electrostatic precipitators (ESPs) (Amann et al 2018).

A review by Lim et al (2015) investigated the use of emission reduction technologies for small scale installations. The study evaluated several technologies that had been tested such as the use of additives, catalytic filters, electrostatic precipitators as well as technologies from the automotive industry. It was concluded that most technologies were still under development and had experienced challenges on implementation.

Cheng et al (2018) investigated the use of high gradient magnetic separation in capturing airborne particles but it achieved low capture efficiency for particles smaller than 50 μm .

In another study, Yamamoto et al (2013) investigated the use of a heated carbon fiber filter to remove soot particles from diesel combustion. Without heating the filter, particles would accumulate on the filter, reducing the porosity significantly. By increasing the filter wall temperature using an electric heater, the study found that most of the particulates could be burned off, resulting in continuous filter regeneration. The study indicated that, while most of the particulates were burned off in the filter, new ultra-fine particles of less than 30 nm were formed, which may have an impact on human health.

Hukkanen et al (2012) investigated the use of a catalytic combustor in reducing emissions from a wood fired boiler. The catalyst utilized consisted of metal wire mesh covered with a platinum and palladium catalyst. For the entire combustion cycle, reductions of 21% for CO, 14% for organic gaseous carbon and 30% of PM_{10} was achieved. In this case, the flue gas temperatures were high enough to activate the catalyst, but the study notes that some residential heaters may not have sufficiently high flue gas temperatures to achieve this. A major disadvantage of this technology is the high cost of the precious metals used as catalysts.

Ozil et al (2009) investigated the use of two catalysts, a cordierite honeycomb monolith support and a metallic corrugated structure impregnated with an alumina washout. The study showed that the temperature of the flue gas of a wood fired boiler was too low during start up and shut down for the catalyst to be effective and the use of a heating system was found to dramatically reduce the emissions of CO and VOCs, with a CO reduction of between 80 and 90% being achieved.

Emissions from domestic fuel burning

Flue gas from domestic solid fuel combustion typically has a low particle loading and the particles are small in size (Xu 2014); specifically, the particulates are expected to be 95% $\text{PM}_{2.5}$ (Zhang et al 2018).

Previous studies have indicated that the particulate matter emissions from solid fuel burning comprised mainly of organic particles, soot and inorganic fly ash (Zhang et al 2018, Torvela et al 2014, Nystrom 2016, Makonese et al 2019). These particulates are also expected to react with each other and with gaseous emissions in the combustion chamber and flue. The temperature of the flue gas, which is likely to depend on the positioning of the abatement device, will influence the catalyst efficiency as well as both the aging of the particulates and their properties. Closer to the combustion chamber, sticky, tarry carbonaceous particles may form (Makonese et al 2019). Various other chain-like or cluster-like particulates are also likely to be present (Forbes 2012). In addition, organic particles with inorganic inclusions may form. A smaller fraction of inorganic fly ash emissions is expected, which may be larger in size than the organic particles (Zhang et al 2018, Makonese et al 2019).

Catalytic abatement

The collection efficiency for particles smaller than 5 μm , as is expected in flue gas streams, is low and non-catalytic methods are unlikely to achieve a meaningful reduction in particulate matter concentration (Perry et al 1997). Catalytic oxidation could be utilized to oxidize particulate matter precursors such as volatile organic compounds and soot particles to reduce secondary particulate formation. Catalytic abatement allows users to utilise the available fuel, with reduced negative health outcomes. Although catalytic methods have not been effectively utilized in practical domestic applications, studies have shown effective soot reduction during laboratory testing (Gao et al 2020).

Volatile organic matter and soot can be oxidized using supported noble metal catalysts and metal oxide catalysts. Noble metal catalysts generally are more active than metal oxide catalysts, but metal oxide catalysts are more resistant to certain catalyst poisons such as halogens, As and Pb. Metal oxide catalysts importantly have a substantially lower cost and can be sufficiently reactive for some applications (Huang et al 2015) and were therefore selected for this study. The most effective single metal catalysts have been found to be oxides of V, Cr, Mn, Fe, Co, Ni and Cu (Spivey 1987). Manganese was selected for this study due to the promising results achieved with laboratory testing (Gao et al 2020). The results indicated that the soot conversion percentage was temperature dependent and increased at temperatures above 250°C. Furthermore, manganese oxide catalysts have low toxicity, low raw material cost and a diversity of crystalline structures which determine the catalytic activity (Huang et al 2015). In this study, a manganese oxide based catalyst was prepared and characterized. It was then tested for potential local application, utilizing a commercial domestic stove.

Methods

Catalyst Requirements

In order to be considered effective, the designed catalyst

should conform to various safety and performance criteria such as acceptable emission reduction, limited clogging, ease of installation, heat resistance, cost effectiveness and the catalyst should be non-toxic. Installation should be possible on non-standardised units, should be self-cleaning or easy to clean with little or no electricity required for operation. The catalyst must further be acceptable to the end user.

Emissions are also expected to vary during the different stages of the combustion process. As the abatement will be in operation during the entire combustion cycle, it should accommodate the emissions from the combined combustion cycle.

Catalyst Synthesis

Three catalyst preparation techniques were tested namely precipitation, wet deposition and dry deposition. The choice of the catalyst support was based on the risk of clogging, ease of installation, minimizing pressure drop, high air flow through the catalyst, and cost, amongst other parameters. A coarse 0.5 mm thick steel mesh was thus used as the catalyst support for direct use in domestic flues as shown in Figure 1.

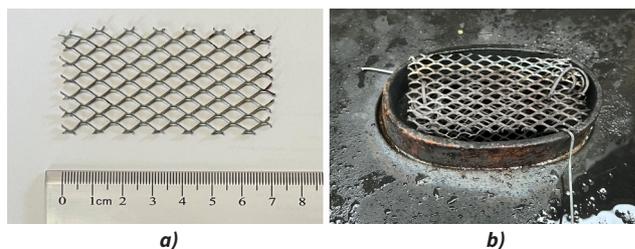


Figure 1: 0.5 mm thick steel mesh used as catalyst support (a) for installation at the base of the flue (b)

The synthesis utilizing precipitation was based on the polyol method, which is well-known for producing metallic nanoparticles (Augustin et al 2015, Li et al 2011, Liu et al 2010, Sukhdev et al 2020). A glycol solvent such as ethylene glycol functions as a solvent for the metal precursors as well as functioning as the reducing agent (Benseeba 2013, Rao et al 2017). The glycol further controls particle formation by controlling nucleation, growth and agglomeration of the particles. The method described by Augustin et al (2015) utilizes mild reaction conditions to produce manganese oxide particles with varying structures and compositions and was therefore used as a basis for the synthesis of the manganese oxide catalyst in this project.

Manganese (II) acetate tetrahydrate (>99%), tetraethylene glycol (99%), iron (III) nitrate nonahydrate (>98%) and ethylene glycol (>99%) were obtained from Sigma-Aldrich (South Africa). All reagents were used as received.

Typical test conditions were as follows: The reaction solution was prepared using 0.5 g of magnesium acetate dissolved in 60 mL of ethylene glycol and 6 mL of tetraethylene glycol (TEG). The catalyst support mesh (commercially available 0.5mm thick steel mesh obtained from Leroy Merlin Hardware Store) was washed using acetone prior to synthesis. The mesh was then

suspended in the reaction mixture using wire hooks secured to the beaker using rubber-based putty adhesive. The mixture was then gradually heated on a magnetic stirrer hot plate (Heidolph, Germany) and stirred using a magnetic stirrer. The temperature of the reagent mixture was measured manually using a thermometer. The reagent mixture was gradually heated to a temperature of 170°C. Samples were removed from the reaction mixture after precipitation occurred at 170°C and were washed using ethanol (>98%, Sigma-Aldrich). The samples were dried under argon (99.9%, Afrox) and were then transferred to a muffle furnace (Lenton, South Africa) at 550°C for five hours to calcine under stagnant air.

The wet deposition catalyst was synthesized by dipping the catalyst support into a concentrated manganese acetate tetrahydrate solution prior to calcining. A mesh sample was prepared using 8 g of manganese acetate tetrahydrate in 30 mL of water. The sample was dipped in the solution, removed and calcined without drying at 550°C for two hours.

The dry deposition catalyst was covered in dry manganese acetate tetrahydrate powder prior to calcining at 550°C for two hours.

Catalyst Testing Methodology

The experimental setup consisted of a commercial one plate combustion device with a flue as shown in Figure 2 (Ndebele Appliances and Coal Stoves, Bronkhorstspuit, South Africa), which is representative of cooking stoves available in South Africa.

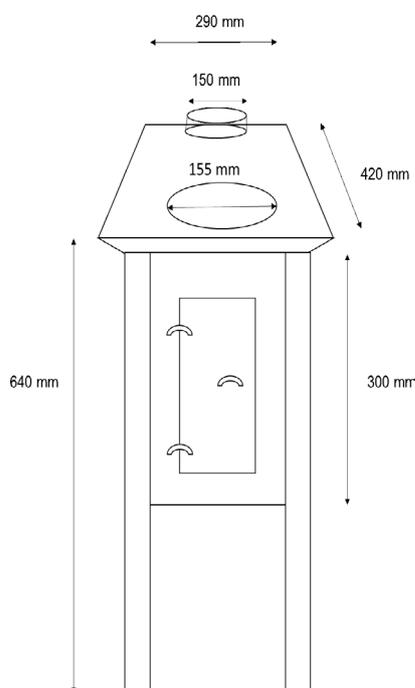


Figure 2: Diagram of the one-plate stove employed in testing of the catalyst

Bulk catalysts were produced using precipitation and wet impregnation. The catalyst was positioned at the base of the

flue where the highest flue temperatures were expected, as laboratory testing had indicated that the catalyst was only effective temperatures above 250°C (Gao et al, 2020).

For each test run, 100 g of soft wood sticks approximately 150mm in length and 20 x 20 mm diameter were utilized and the combustion process was started using a single wax based firelighter ball. The soft wood was selected as fuel to improve the reproducibility of the test runs as the wood is more homogenous in size and composition when compared to hard wood or coal. The test runs were conducted under similar ambient conditions at the same time each day to improve reproducibility. The fuel used for the runs was sized, weighed and bagged to reduce fuel variability and to ensure consistent fuel moisture content.

Measurement Methodology

The flue gas flow rate was measured using a vane type digital anemometer (Benetech, China). The flue temperature was measured using an infrared thermometer (Shenzen FLUS Technology Company, China). Measurements were taken at 290mm from the flue exit point to allow for the cooling of the flue gas.

Particulates were collected for gravimetric analysis using 37 mm polystyrene cassettes, with 37 mm polytetrafluoroethylene (PTFE) filters with a pore size of 2 µm (Environmental Express, South Carolina, USA). PTFE filters were selected as they are temperature resistant up to 260°C. A Gilian (GilAir Plus) personal sampling pump was used to collect particulates onto the filters at a sampling flow rate of 600 mL/min for 10 min. The positioning of the sampling probe could potentially influence the size distribution of the particulate matter sampled, favouring the capture of finer particles. For this study non-isokinetic sampling was considered adequate as the study was interested in whether the catalyst affected the particulate matter formation in a relative manner, and not to quantify the emissions or derive emission factors. Sample filters were weighed using a Mettler Toledo microgram analytical balance in a temperature (21±1°C) and humidity (50±5%) controlled environment.

Catalyst and PTFE Filter Analysis

The catalyst samples produced were examined using Scanning Electron Microscopy (SEM) using a Zeiss Crossbeam 540 SEM at 20 kV to determine the morphology and particle size of the catalyst and to determine the catalyst coverage on the mesh substrate.

The catalyst samples were analysed using Energy Dispersive X-Ray (EDX) (OXFORD Link-ISIS-300 Zeiss, Germany) analysis to determine the semi-quantitative composition of the catalysts.

The PTFE filter samples were examined using the same SEM instrument at 2 kV. A section of the center part of each filter was removed using a steel blade, mounted on an aluminum stub and sputter coated with Au to improve conductivity.

Results and Discussion

Catalyst Synthesis

Synthesis using precipitation

The synthesis was optimized by changing the initial concentrations of manganese acetate tetrahydrate and TEG used to prepare the reaction mixture. The test sample was produced utilizing a 5 x 5 mm piece of mesh suspended in the reagent mixture. Although the precipitate formed on the wire mesh, the precipitate was patchy and did not cover a large percentage of the mesh surface. The Energy Dispersive X-Ray (EDX) analysis confirmed that the precipitates formed during the precipitation runs were manganese oxides with carbon inclusions.

The test run was repeated with an increased manganese acetate tetrahydrate mass of 1 g to improve the catalyst coverage on the mesh. The mesh was calcined directly after synthesis and the calcining time was reduced to 2 hours to ensure that the catalyst was calcined directly after synthesis. The precipitate on the mesh could clearly be seen at 100 X magnification as shown in Figure 3. The precipitate covered the mesh well and formed clusters of precipitate in places. The utilization of higher concentrations of manganese acetate tetrahydrate greatly improved the precipitate coverage on the mesh.

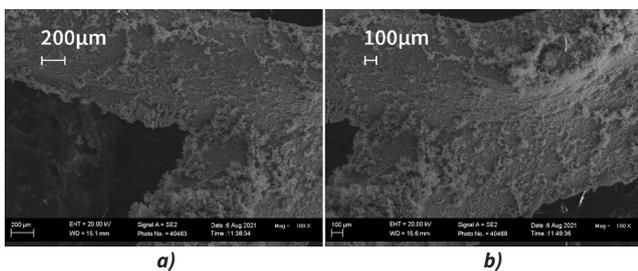


Figure 3: SEM micrographs of the catalyst synthesized using increased manganese acetate tetrahydrate concentrations showing precipitate on the mesh at 100 X magnification

To investigate the impact of the addition of TEG on the coverage and morphology of the catalyst, the previous test was repeated without the addition of TEG. The precipitate formed had good coverage, as shown in Figure 4.

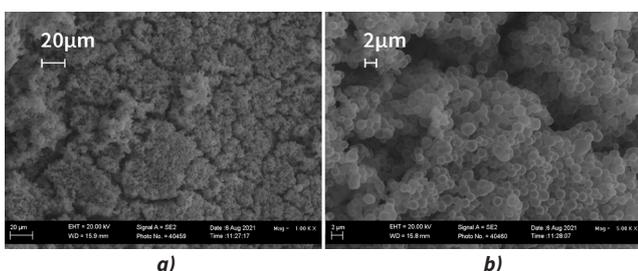


Figure 4: SEM micrographs showing the precipitate comprised of spherical particles at 1000 X (a) and 5000 X (b) magnification for the catalyst produced without the addition of TEG

The addition of TEG did not appear to improve coverage of the mesh with catalyst particles and the precipitates appeared to have similar morphologies as shown in Figure 5.

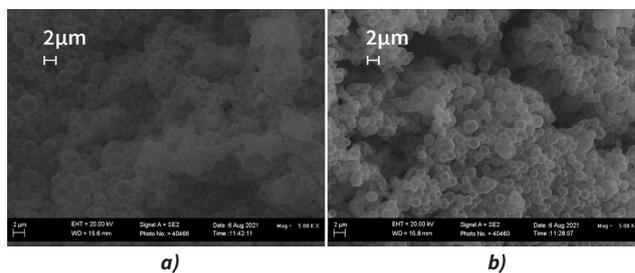


Figure 5: SEM micrographs at 5000 X magnification comparing the samples prepared with the addition of TEG (a) and samples prepared without TEG (b)

Synthesis using wet and dry deposition

A relatively smooth manganese oxide deposit was formed using wet precipitation and good coverage of the catalyst on the mesh was achieved as shown in Figure 6.

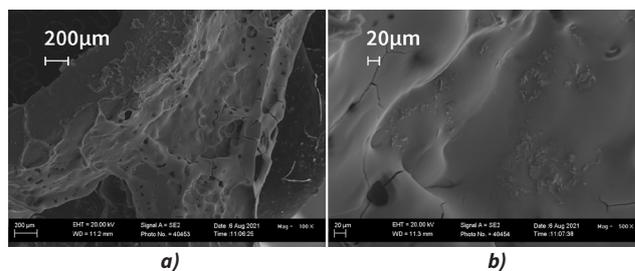


Figure 6: SEM micrographs showing the catalyst synthesized using wet impregnation at 100 X (a) and 500 X (b) magnification

The morphology of the catalyst changed significantly depending on the synthesis method used. At higher magnifications, the surface of the wet impregnation sample had a plate-like structure compared to the spherical, agglomerated particle type structure of the sample prepared using precipitation as shown in Figure 7.

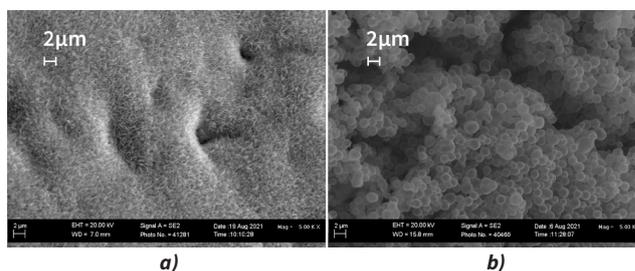


Figure 7: SEM micrographs comparing the surface of samples prepared using wet impregnation (a) and precipitation (b) at 5000 X magnification

The catalyst prepared using dry impregnation did not achieve good coverage and the mesh substrate was still clearly visible.

Bulk catalyst preparation

Both the optimized precipitation synthesis and wet impregnation techniques achieved good coverage of the mesh substrate. The wet impregnation technique is a simpler process

involving dipping the mesh into a concentrated solution of manganese acetate tetrahydrate prior to calcination, whereas the precipitation technique involves the gradual heating of the reagents to form a precipitate. The morphologies of the samples prepared were significantly different, as shown in the paragraph 'Synthesis using wet and dry deposition'.

The sample was analysed using SEM. The results indicated that that the procedure can be successfully scaled up and that similar coverage and morphology was achieved to that obtained in the smaller test scale tests as shown in Figure 8.

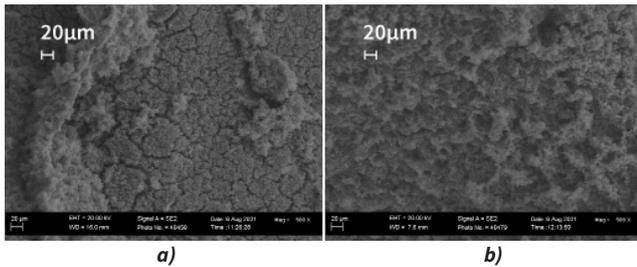


Figure 8: SEM micrographs at 500 X magnification comparing the catalyst synthesized from the optimized test run (a) and synthesis of the bulk sample (b)

Catalyst Testing

Bulk catalyst samples were prepared for testwork using both techniques and were then field tested.

The gravimetric analysis results indicated that relatively higher mass concentrations of particulate matter were recorded for the catalyst runs compared to the baseline runs as shown in Figure 9. It was expected that due to the oxidation of VOCs over the catalyst, secondary particulate matter would be reduced during the catalyst runs.

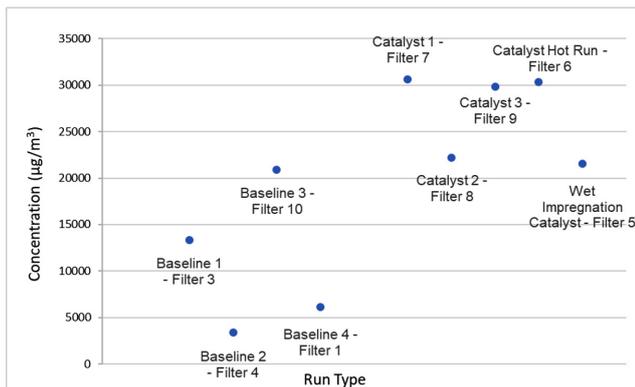


Figure 9: Comparison of the gravimetric results for the catalyst and baseline runs

To investigate the cause of the increased particulate matter, Scanning Electron Microscopy (SEM) analysis was conducted on five of the samples. Two baseline samples (Filters 3 and 10), two catalyst samples (Filters 6 and 9) and the field blank sample (Filter 2) were selected for analysis.

For the catalyst runs, clusters of particles could be seen within the pores of the filters, as shown in Figure 10. Both filters had particle clusters within the pores, but the Catalyst Hot Run which was sampled only during the flaming phase of the run and therefore for a shorter period (Filter 6) had fewer of these clusters.

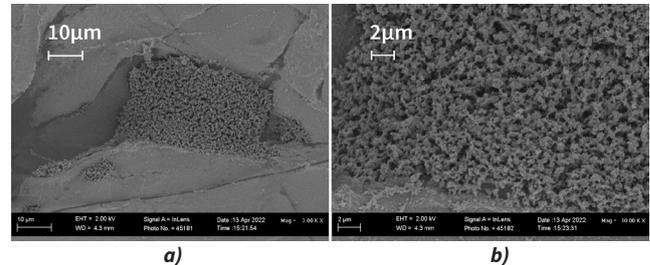


Figure 10: SEM micrograph at 3000 X (a) and 10000 X (b) magnification showing spherical particles smaller than 1 µm in size for the Catalyst Hot Run (Filter 6)

Particulate clusters can clearly be seen in the pores of the filter for the catalyst run. Very few similar clusters could be seen on the baseline sample filter as shown in Figure 11.

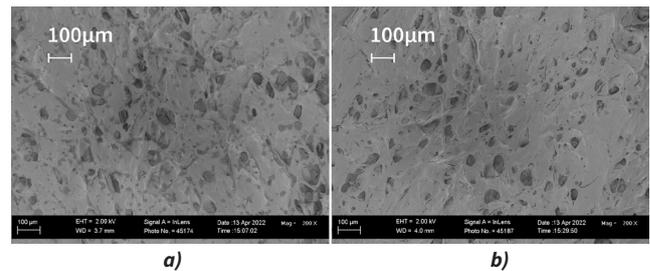


Figure 11: SEM micrographs comparing the filters from catalyst Run 3 (Filter 9) (a) to baseline Run 3 (Filter 10) (b) at 200 X magnification

Energy-dispersive X-ray spectroscopy (EDX) analysis was conducted at 15 kV on the samples to determine whether there was any detectable catalyst lost to the flue gas which would contribute to the PM load on the filters. Carbon and fluoride were detected by the EDX analysis due to the composition of the filter, therefore it was not possible to definitively determine whether the particulates formed were carbon-based due to the background carbon content of the filter. The EDX analysis did not detect any manganese and the increased particulate mass on the filters used for the catalyst runs were therefore not as a result of macroscopic particles of the catalyst detaching from the support.

Conclusions and Recommendations

The testwork showed that an active catalyst can be synthesized in a facile and cost effective manner onto a mesh catalyst support which can be utilized in domestic fuel burning devices. The catalyst is easy to install and can be customised to fit non-standard domestic combustion units. The manganese based catalyst is heat resistant and does not release toxic materials. The catalyst is light in weight and requires no electricity to operate. During the limited test runs conducted, the catalyst

did not clog, the airflow was not restricted and the stove vented as per the baseline runs. It is recommended that the catalyst be tested over a prolonged period to determine whether the catalyst remains self-cleaning or whether it clogs over time. The number of consecutive cycles it can be used over should also be determined (in this study four cycles were successfully tested in this regard).

The total particulate concentrations were measured gravimetrically and the particle mass concentration of the catalyst runs were found to be higher than for the baseline runs. EDX analysis indicated that the increased particulate concentration on the catalyst runs was not likely to be as a result of macroscopic particles of the catalyst being dislodged from the support, as no manganese was detected. The catalyst therefore had good adherence to the catalyst support.

SEM analysis showed that the catalyst run filters had particulate clusters comprised of spherical particles in the pores of the filters. The baseline runs had very few, if any, of these particle clusters. The increased particulate matter could result from the dislodgement of very small metal particles from the catalyst which served as nucleation nodes for particle growth. The metallic nuclei may have had a non-metal coating which may lead to non-detection by EDX. The increase in particulate matter could also be caused by the impingement of particulate matter precursors on the catalyst. Particle growth occurs on the catalyst surface. If the catalyst does not oxidize the impinged pollutant efficiently, particle growth can occur until the particles formed are dislodged from the catalyst by the flue gas, resulting in chain like aggregates. Measuring total particulate matter mass concentrations does not provide information on the morphology, surface chemistry, composition and size characterization of the particulates. It is thus recommended that more detailed analysis be conducted on the particulate matter to provide further insight into secondary particulate formation.

Although the reaction mechanism of oxidation over a manganese oxide based catalyst is generally understood as being through the supply of oxygen from the crystal lattice, a detailed understanding of the elemental reactions are not well understood, particularly for multicomponent systems (Khaskheli et. al. 2022). An improved understanding of the reaction mechanism could lead to the optimisation of the catalyst activity.

Various studies have shown that manganese oxide catalysts are able to oxidize CO, however previous studies investigating the use of manganese oxide catalysts for soot, CO and VOC reduction have mostly been conducted at laboratory scale and not under typical practical (real world) conditions. During laboratory testing, the soot or gas mixture has sufficient time to react and sufficient contact is achieved between the catalyst and the soot or gas (Khaskheli et. al. 2022). In a practical application, there will be much less contact time between the catalyst and the flue gas, which may impact on the efficiency of the catalyst. It is therefore recommended that a range of optimized, potentially active catalysts be tested to improve the oxidation of particulate

matter precursors to CO₂. In addition, it is recommended that further testwork include the analysis of gaseous components such as CO, VOCs and CO₂ to determine the extent of oxidation of the particulate matter precursors.

The testwork showed that an active catalyst can be synthesized onto a mesh catalyst support which can be utilized in domestic fuel burning devices. The potential use of an optimised, cost effective catalyst to reduce domestic fuel burning emissions by oxidising particulate matter precursors would enable the continued use of locally available solid fuel with reduced health and environmental impacts.

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