SYNERGETICS

ENVIRONMENTAL ENGINEERING

Review of literature on gasification derived nanoparticles

for

New Energy Corporation Pty Ltd

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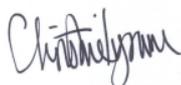
Review of literature on gasification derived nanoparticles

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1   Introduction

The National Environment Protection (Ambient Air) Measure (NEPC 2003) has been established to provide regulation of a range of air pollutants, including particulate matter up to 10 micron in size (PM_{10}).[1] The 2003 NEPM includes an advisory standard for $PM_{2.5}$.[2] These measures provide a means to assess and regulate particulate matter pollution to protect particular aspects of the environment.

However, with the evolution of technology towards nanoscale particles interest in the health impacts of the both engineered and incidental particulate matter in the sub $PM_{2.5}$ fractions has increased. As an example, the toxicological effects of nanoparticles (engineered and incidental) are discussed in a comprehensive review by Madl and Pinkerton (2009).   Those authors found that a better understanding of the physicochemistry and the fate of the particles within the body and the cellular responses was required in order to predict the toxicity of nanoparticles and establish a framework for human health risk assessment. From there, measures to monitor and regulate nanoparticle emissions can be considered for future control of those emissions to ensure protection of community health.

The role of nanoparticles in the health impacts of emissions from industry has become topical in recent times and material from groups opposed to new industrial developments referring to the health impacts of nanoparticles has been presented at public forums throughout the world and is readily available on the internet (Connett 2011).

Recent presentations to community groups in Western Australia have directly alerted those groups to claims of adverse health impacts from nanoparticles from waste incineration.[3] Knowledge of the generation of nanoparticles from waste gasification is of interest to the New Energy Corporation, to establish an understanding of the potential for nanoparticle generation from the Entech process. This is necessary to ensure that any nanoparticle generation is controlled to the maximum extent achievable to ensure protection of public health, as well as to have full understanding of the emissions profile of the technology required to address concerns that may be raised by community groups.

To that end, NEC has asked Synergetics to conduct a review of primary and secondary literature on the formation chemistry and emissions of nanoparticles from gasification processes.   This report describes the key findings from the review and recommendations for further work specifically aimed at addressing the potential for nanoparticle formation and emission from the Entech gasification technology.

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[1] PM_{10} is defined as particulate matter with an equivalent aerodynamic diameter of 10 micrometres or less.
[2] $PM_{2.5}$ means particulate matter with an equivalent aerodynamic diameter of 2.5 micrometres or less.
[3] As presented by Dr Paul Connett at venues in Midland, Kwinana and Perth, February 2011, hosted by the Alliance for Clean Environment.

2 Definition of a nanoparticle

In 2006, the ASTM International (formerly know as the American Society for Testing and Materials) released a document to define the terminology used in the field of nanotechnology (ASTM International 2006). Contributions to this document came from "nano-researchers" from different fields including the American Institute of Chemical Engineers (AIChE), the American Society of mechanical Engineers (ASME), the Institute of Electrical and Electronic Engineers (IEEE), the Japanese National Institute of Advance Industrial Science and Technology (AIST), NSF International and Semiconductor Equipment and Materials International (SEMI) (McCarthy 2007).

The following definition of a nanoparticle was presented:-

"nanoparticle, n—in nanotechnology, a sub-classification of ultrafine particle with lengths in two or three dimensions greater than 0.001 micrometer (1 nanometer) and smaller than about 0.1 micrometer (100 nanometers) and which may or may not exhibit a size-related intensive property."

The following discussion accompanied the definition of a nanoparticle:-

"This term is a subject of controversy regarding the size range and the presence of a size-related property. Current usage emphasizes size and not properties in the definition. The length scale may be a hydrodynamic diameter or a geometric length appropriate to the intended use of the nanoparticle."

Nanoparticles also fall within the definition of an ultrafine particle:-

ultrafine particle, n—in nanotechnology, a particle ranging in size from approximately 0.1 micrometer (100 nanometers) to 0.001 micrometers (1 nanometer).

The International Standards organization (ISO) also has a technical specification for the terminology associated with nanotechnologies (ISO 2008). Nanoparticle is specified as a nano-object with 3 dimensions in the nanoscale where the nanoscale is defined as the size range from approximately 1 nm to 100 nm. Ultrafines are defined by ISO as particles with an equivalent diameter less than 100 nm and it is noted that most nanoparticles are also ultrafines as defined by their geometrical dimensions.

Some authors have a different definition of nanoparticles and ultrafines, however these are not explicitly stated in all publications. Therefore, where the terms nanoparticle or ultrafine are used in the literature but not otherwise defined, it is assumed that the international definitions have been adopted and particles in the 100 nm range are being described. While conducting the review, all studies concerning particles in the 100 nm range were considered, i.e. ultrafines as well as nanoparticles, since the health effects of these two categories of particles are likely to be similar. The terminology as used by the author of the published work will be used when referring to the individual studies. Hence the terms nanoparticles and ultrafine particles have been used interchangeably in this review, rather than adhering to the strict definition of a nanoparticle requiring at least 2 dimensions to be below 100 nm.

3 Air emissions from thermal processing facilities

Thermal processing may be carried out solely to generate electricity i.e. the processing of purpose produced biomass, or to convert waste to energy in a process to dispose of unwanted materials with the added benefit of co-generation of electrical power or heat for a secondary application (e.g. steam for heating of nearby buildings or for operation of a chemical process). The term thermal processing includes both incineration and gasification processes as well as pyrolysis. Pyrolysis strictly refers to the thermal degradation in the absence of oxygen however the term is often more loosely used in the literature to describe gasification processes i.e. the process is described as pyrolysis with the addition of air (such as in Gómez-Moreno, 2003). Note that some pyrolysis will occur during gasification and combustion due to localised competition for oxygen to fuel the combustion processes.

Similarities between gasification and incineration processes do exist, such as being able to process the same fuels, employ similar temperatures and use similar plant equipment. However due to the fundamental differences in the chemistry of the processes, the emissions profiles from combustion and gasification are distinct. Therefore, despite the numerous studies in the literature on the formation of nanoparticles from combustion, these cannot necessarily be directly extrapolated to apply to gasification processes. To demonstrate the fundamental differences in the processes, the fate of the major constituents of the air emissions from incineration and gasification are outlined below.

Incineration processes involve the combustion of fuel in an oxidising environment (excess air) resulting in the conversion of the carbonaceous portion of the fuel to CO_2 and H_2O as well as the oxidation of nitrogen and sulfur in the fuel to NO_X and SO_X. Acid gases are formed from any halogens in the fuel. Thermal NO_x is also generated from the nitrogen gas in the air added to facilitate combustion.

Gasification is carried out in a reducing environment where the ratio of oxygen (O_2) to carbon is less than 1 (substoichometric). The fundamental processes that occur during gasification result in the thermal conversion of fuel to form synthesis gas (syngas). Syngas is primarily comprised of CO and H_2 with some CO_2 and CH_4. The formation of SO_X and NO_X does not occur due to the reducing conditions and sulphur and nitrogen are primarily converted to H_2S, ammonia and N_2 (Orr 2000). Syngas generated from the gasification stage is fed to a secondary chamber where air is introduced and the syngas undergoes combustion. During the combustion process any species entrained in the syngas will undergo further transformation i.e. oxidation of nitrogen and sulphur species to NO_X and SO_X prior to release to atmosphere. Thus it is the composition of the syngas that enters the combustion chamber, combined with the pollution reduction achieved by any pollution control equipment employed post the combustion chamber and any further reactions, that will dictate the species ultimately released to the atmosphere in the waste combustion gases.

Minor air emissions constituents from thermal processes include metals, either in gaseous form (i.e. elemental Mercury) or associated with particulates, as well as volatile organics and particulates. Particulate material includes both filterable particles and condensable particles (aerosols) in the stack gas. The exact profile of the emissions will very much depend on the fuel type and the exact specifics of the

process technology employed including factors such as residence time, agitation and O_2 concentration.

The profile of the final emissions depends not only on the upstream gasification process but also on the gas cleaning (pollution control) technology installed, e.g. catalytic particulate filters for the removal of dioxins or electrostatic precipitators for the removal of particulates. To a large degree, the extent of gas clean-up appears to be driven by regulatory requirements and while there is a trend towards removal of sub-PM_{10} particulate, the current technology available to industry does not have the capacity to effectively remove ultrafine or nanoparticle particulates.

4 Review of examples of nanoparticle emissions from gasification processes

The aim of this review is to identify literature precedent for the formation of nanoparticles specifically from gasification processes that can be applied to an assessment of the potential for such emissions from the Entech gasification technology. The review involved keyword based searching of various publically available on-line databases to obtain key publications for examination, then pursuit of relevant papers and articles referenced therein. Time and cost constraints meant that the review was not exhaustive. However, the intention was to identify sufficient publications that provided key information on aspects of nanoparticle formation and emissions from gasification processes including waste gasification.

A summary of the relevant literature identified in this review is presented at the end of this section (Table 1).

The review found that a large number of studies are reported on the emission of nanoparticles or ultrafine particles from combustion and several mechanisms of formation of combustion derived particles, including nano and ultrafine particles, have been described and reviewed (Cernaushi 2009, Cormier 2006, Flegg 2010, Frenlach 2002, Guigliano 2008, Kauppinen 1990, Slama Lighty 2000, Woolridge 1998). As previously described (Section 3) due to the fundamental differences between gasification and combustion processes these studies can not be directly related to gasification process and examples for gasification (where a syngas is formed and combusted) were sought.

Particulates may be formed by simple mechanisms such as the volatilisation of metals at high temperatures in the primary thermal processing chamber and condensation, at lower temperatures downstream in the process, as well as more complex mechanisms, i.e. the PAH theory of soot formation. These mechanisms may also be applicable to gasification, however investigation of this was considered outside the scope of this review.

Less examples of nanoparticle formation from gasification (or pyrolysis) than from combustion are available in the literature, however several gasification examples were found and these are described below. Note that all particle sizes presented are mobility diameters unless otherwise stated.[4] Comparison of the absolute concentration of particulates was not carried out, since this is dependent on many process factors including the fuel type, the gasification conditions and the flue gas treatment equipment employed, as well as in what stage of the process the sampling is conducted and the scale of the process. Gasification of coal was excluded from this review since this is not a waste derived fuel source and was therefore unlikely to be used as a fuel for the Entech gasification technology. A critical review of the sampling and analysis technology employed in the studies reported in the literature was also

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[4] Mobility diameter is the diameter of a sphere that has the same drift velocity in an electric field as the particle under study. The mobility diameter depends on particle size and shape.

outside the scope of this review on the precedent of for nanoparticle formation from gasification.

Lemieux reported a study on the use of a mobile gasification plant for the destruction of animal carcasses (Liemieux 2008, 2010). The process involved the gasification of ground animal carcasses (size unspecified) in a primary chamber followed by firing of the product gases in the secondary combustion chamber. Nearly all of particulate in the stack gases was reported to be below 500 nm with the majority of the mass (99.6 % of the cumulative mass) being less than 1.62 µm and 54.8 % cumulative mass being less than 330 nm diameter(Lemieux 2008). The specifications of the method used to measure the particulate size distribution were not provided, however the results indicated that the lowest cut off was < 330 nm aerodynamic diameter and the largest was 8.79 µm. While the nano-range was not specifically investigated, it is reasonable to assume that a portion of the < 330 nm fraction would fall below 100 nm. However no further information can be gleaned from this paper on that matter .

A study on the particulate emissions from the pilot scale pyrolytic destruction[5] of milled refuse derived fuel was reported by Gómez-Moreno *et al* (2003). A side stream was extracted for particulate analysis prior to the gas treatment components of the process. The characteristics of the particulates emitted were studied throughout the pyrolytic, transition to combustion and combustion process phases. Particle size measurements were conducted using a single-particle light scattering optical spectrometer and a scanning mobility particle sizer (SMPS) for the submicron fraction. The particulate counts dropped following a peak during the transition from pyrolysis-combustion to the combustion phase. The emitted particulate was found to be in the less than 700 nm size range for all phases. A downward shift in the size distribution also occurred in the combustion phase from being predominantly in the range of 100 to 300 nm during the transition phase to 50 to 150 nm in the combustion phase. Elemental analysis was conducted by combustion for C and S and by Flame Emission Spectroscopy, by ICP-ESS, or by ICP-MS for the metals. Analysis of the major constituents of the particulate was reported for 2 experiments (mean for all samples taken throughout the experiment). Carbon, sulfur, Na_2O, K_2O, CaO, Fe_2O_3, MgO, Copper and Lead were found to comprise over 99% of the particulate weight (in varying proportions between the 2 experiments for which data were reported).

Caponero *et al* describe the emissions from the laboratory scale pyrolysis of waste tyre chips (in the absence of air). The study compares pyrolytic (in nitrogen) and oxidising condition prior to entry into the secondary chamber (Capnero 2005). The experiments were conducted by mixing either oxygen or nitrogen into a Venturi mixer prior to exiting the primary pyrolysis chamber. The addition of oxygen resulted in a flame from the pyrolysates. Sampling was conducted at the exit of each chamber. Particulate sizing was conducted using an Andersen impactor (1 ACFM nonviable ambient particle sizing sampler) which simulates the human respiratory system. The results showed that under pyrolysis-pyrolysis conditions, the emissions of particulates ranged from 10 to 50 times higher, depending on primary furnace temperature, than those detected with the oxidising step (PAH emissions were also found to be 5 to 10 times higher varying with temperature from the pyrolysis-pyrolysis conditions). Under pyrolysis-oxidation conditions, 64% of the particulates exiting the secondary chamber

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[5] Note air was introduced into the chamber to control the pyrolysis rate, therefore O_2 is present in the pyrolytic chamber and thus this process is regarded as gasification

were found to be sub-micron. Unfortunately the lowest particle size cut had a lower bound of 400 nm therefore the production of particles falling within the ultrafine fraction was not characterised. No particle size results for the pyrolysis-pyrolysis experiment were reported. With respect to a gasification process the results suggest that while sub-micron particulates and PAHs will be formed in the gasification chamber, the concentration in the final emissions are likely to be reduced by the firing of the syngas in the oxidising conditions of the combustion chamber.

The particulates in the syngas generated from a 20 kW wood pellet bubbling fluidised bed gasification (using oxygen and steam introduced into the bottom of a magnesite bed) were characterised in a study reported by Gustafsson (2007). In this paper it was noted that the handling of the syngas from gasification of biomass can be complicated by the presence of tars which can condense upon cooling of the syngas and subsequently contribute to the particulate fraction. The particulate sampling methodology employed was subsequently designed to ensure condensed tars were not analysed as part of the particulate matter. Particulate sampling was conducted from the process at steady state from the top of the gasifier thus the particulate in the syngas was characterised as opposed to in the waste gases post-combustion. A bimodal particle size distribution was measured for particle counts in the range 15 to 670 nm (measured by SMPS) with a large peak at approximately 20 nm and a smaller peak at 400 nm (middle of the trough at 105 nm). Particles up to 10 μm aerodynamic diameter were measured using a thermogravimetric analyser (TGA), however upon conversion of the SMPS results for direct comparison with the TGA results, some disagreement for the fractions below 500 nm aerodynamic diameter was reported. This highlights the need for caution when comparing measurements made using different technologies. The composition of the particulates was investigated. Carbon, quantified by scanning electron microscopy with energy-dispersive spectroscopy (SEM-EDS), was identified to be the dominant component in the particulates greater the 1 μm. Unfortunately Carbon could not be effectively quantified in particles less that 1 μm diameter due to the EDS beam size of 1 μm. Potassium and chlorine, expected by the authors to be present in the particulate was not detected in the study. Elemental analysis by inductively coupled plasma mass spectrometry/atomic emission spectra (ICP-AES/MS) of the sub 5 μm aerodynamic diameter fraction identified elements making up 9% of the mass (~14wt % if oxides were present) and it was postulated that much of the remaining mass would be contributed by carbon. Of the elements detected by ICP-AES/MS it was found that Calcium, Sodium and Sulfur dominated. Particle Induced X-ray Emission (PIXE) was also used to determine the presence of Calcium, Manganese, Potassium, Iron, Chlorine and Titanium in the particulate fractions from 0.04 to 8.38 μm. Calcium was found to dominate comprising up to 75 mass percentage of these elements in the particulates below 130 nm and Manganese contributed approximately 20 mass percentage of the elements detected by PIXE. It was noted that as the particle size increased the elemental composition changed with the larger fractions being less dominated by Calcium and having higher percentages of Iron and Potassium.

Gaegauf conducted a study to determine the emissions of nanoparticles from biomass combustion (Gaegauf 2001). The aim of this study was to characterise the particulate formed from 14 different wood combustion systems using a range of wood based fuel types. One of the systems was a gasification system unfortunately the data for this appliance was not presented separately from the combustion systems. Particulates from the stack gases of all systems were determined using a SMPS for the 10 to 600 nm range and reported typically to be predominantly in the 30 to 300 nm range. The

particle size distribution was found to vary according to the phase of the burn cycle and data were presented for one of the combustion systems showing a shift in the particle size distribution showing an increase in the mode mobility diameter with a reduction in excess air (from 11.6% to 2.7% O_2).

Hindsgaul reported the characterisation of particulates from a two stage (pyrolysis and gasification) down-draft biomass gasifier (Hindsgaul 2000). The primary chamber was a pyrolysis chamber. In the secondary chamber steam and air were added to the pyrolysis syngas and fixed char bed. Particulate was sampled from the gas stream from pre and post the venturi gas clean up system using a cascade impactor to determine the particle size distribution and a scanning electron microscope (SEM) was used to examine the particulate geometry and inorganic species. It was found that 80% of the particulate captured prior to input into the gas cleaning system had a diameter of approximately 70 nm (no data post the clean up system was presented) and were mono-sized spherical primary soot particles. An x-ray fluorescence (XRF) detector was used to detect carbon, potassium, sulfur, chlorine and oxygen and the carbon fraction was estimated using thermogravimetric analysis for the 220 nm and < 220 nm (glass end filter) fractions. The polarity of the volatile fraction was also investigated using adsorption column chromatography and analysis by gas chromatography.

A comparison of the combustion and gasification of wood products has been carried out by a group at the University of Kuopio in Finland (Sippula 2009). During this study a gasifier was compared with a rotating grate furnace using wood chip (forest residue) fuel producing 7 and 15 MW output, respectively. The rotating grate combustion furnace was equipped with a multicyclone and ESP whereas the gasifier had only a monocyclone to remove coarse particulates. Two other combustion technologies were also evaluated, however an alternative fuel source was used therefore these are not considered directly comparable. Sampling was conducted directly after the cyclone as well as after the ESP for the furnace. The flue gas particle size distribution in the size range from 0.024 μm to 9.5 μm was analysed using an electric low pressure impactor. A Berner low pressure impactor was also used to determine the particle mass concentration as a function of aerodynamic diameter. Carbon analysis was conducted on PM_1 samples using an optical carbon analyser while elemental analysis was conduced via ion chromatography and ICP-MS. Results were presented corrected to 6% O_2 in the flue gases. The gasification process has 4% O_2 in the stack gases while the combustion process was run at 5%. This gives the gasification process a slight advantage (~ 7%) in the comparison of the emissions conducted by the authors of the study. The emissions were normalised to the energy output in order to account for the different capacity of the two processes. It was found that the concentration of pre ESP PM_1 emissions from the combustion plant were in the order of 7 times higher than the PM_1 emissions from the gasifier. The particle number size distribution from the gasifier (aerodynamic diameter) had a mode less than 100 nm. The particle number size distribution of the combustion process showed much lower counts in the ultrafine fraction (measured after the ESP). Composition data from the particulates from gasification were not specifically presented. It was presented (not defined from which process) that the fine fraction was predominantly comprised potassium, sulfate and chloride. Zinc, some organic compounds, Sodium, Calcium and Magnesium were also identified as well as some other metals (not specified). Plots presented for the element size distributions of the particles upstream of any gas treatment installed in the different process omitted plant B therefore it was not possible to compare a combustion and gasification process using the same fuel type. It is however

noteworthy that the gasification process yielded a similar distribution for Cadmium, Thallium, Zinc, Manganese and Lead as the 2 combustion plants utilising an alternative woody fuel type while the profile for Copper was distinct.

Table 1 – Summary of literature on particulate emissions from gasification

Author	Date	Process	Fuel type	Sampling location	Particle size* investigated (µm)	Nanoparticle composition investigated
Lemieux	2008	Gasification	Animal carcasses	Stack (no gas clean up)	< 0.33 – 8.79	No
Caponero	2005	Pyrolysis-pyrolysis and pyrolysis-combustion	Tyre chips	Exit of primary and secondary chambers	<0.4 - 9	No
Gómez-Moreno	2003	Gasification	Refuse derived fuel (particle size < 3mm)	Post-secondary chamber and heat exchanger, pre-gas treatment system	< 0.02 to 20	Yes
Gustafsson	2007	Bubbling fluidised Bed Gasification	Wood pellets	Gasification chamber (product gas)	0.15 to 0.67	Yes
Hindsgaul	2000	Downdraft gasification	Woodchips	Pre and post flue gas venturi scrubber and demister	0.22 to >1.1, SEM used to determine size of ultrafines	Yes (in < 220 nm fraction)
Sippula	2009	Comparison of combustion and gasification	Woodchips or forest residue and bark	Post cyclone to remove coarse fraction and pre and post secondary particulate filtration	0.024-9.5 (aerodynamic diameter)	Yes

* Mobility equivalent particle diameter unless otherwise stated

5 Discussion

Synergetics scientists (Dr. Christine Ingram and Dr. Peter Forster) attended the presentations recently held in Perth by Dr. Paul Connett on incineration based waste to energy (Connett 2011). It is notable that Connett considered the potential health risks from incineration (combustion), gasification and pyrolysis to be the same. Since there are fundamental differences in the basic chemistry, these blanket claims appear not supported by extrapolation of data from combustion processes to gasification processes.

Notwithstanding that issue, the literature clearly shows that particles in the sub 100 nm range can be generated and emitted from gasification processes. While direct comparisons of results from different studies (or different measurement techniques in the case of particle sizing) are not feasible the reviewed literature examples show that process technologies and process conditions can affect the composition and size distribution of emitted particulates. Therefore the impacts of different operating conditions should be considered when evaluating the particulate emissions from a process.

6   Conclusions and recommendations

As discussed, the literature indicates that nanoparticles can be generated and emitted from gasification processes.   However, direct application of those findings to the Entech gasification technology cannot be made until an understanding of the impact of the design and operating conditions that will prevail with that technology on nanoparticulate formation is known.

Although it could be advocated that measurements of particulates including those in the nanoparticle size range be conducted on an Entech facility, such measurements would be premature in light of the absence of a regulatory framework with benchmarks established for acceptable levels of nanoparticle emissions and standardised methods available for the measurements.

However, at some time in the future, it may be appropriate to determine the nature and rates of emissions of nanoparticles from the proposed NEC facility. In that case, careful planning would be required in order to select the most appropriate methodology (regulatory approved or otherwise) to obtain data that is as representative as possible of any nanoparticle emissions for the various types of waste feed. A key issue to be addressed is the potential for changes in the particle size distribution due to agglomeration or deposition of the particular type of particulates obtained from the Entech process, in the sampling apparatus prior to particle counting. A review of the mechanisms of particle formation may be pertinent, once data on nanoparticle formation and emission from the Entech process have been obtained, to determine whether reductions in the level of any ultrafines produced could be realised at a fundamental level to reduce the significance (if any) of those emissions.

Therefore, in the absence of regulatory approved methods and assessment framework, it is recommended that the available (and evolving) technologies for sampling, monitoring and end-of-pipe removal of submicron particulates are reviewed periodically.   That will ensure NEC is well prepared in the event that future environmental regulations include nanoparticle emissions.

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