Wastewater-derived Ammonia for a Green Transportation Fuel

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Mineta Transportation Institute

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Abstract
The energy-water nexus (i.e., availability of potable water and clean energy) is among the most important problems currently facing society. Ammonia is a carbon-free fuel that has the potential to reduce the carbon footprint in combustion related vehicles. However, ammonia production processes typically have their own carbon footprint and do not necessarily come from sustainable sources. This research examines wastewater filtration processes to harvest ammonia for transportation processes. The research team studied mock wastewater solutions and was able to achieve ammonia concentrations above 80% (nanofiltration) and 90% (reverse osmosis). The research team also investigated the influence of transmembrane pressure and flow rates. No degradation to the membrane integrity was observed during the process. This research used constant pressure combustion simulations to calculate the ignition delay times for NH₃-air flames with expected impurities from the wastewater treatment processes. The influence of impurities, such as H₂O, CO, CO₂, and HCl, were studied under a range of thermodynamic conditions expected in compression ignition engines. The team observed carbon monoxide and water vapor to slightly decrease (at most 5%) ignition delay time, whereas HCl, in general, increased the ignition delay. The changes to the combustion chemistry and its influence of the reaction mechanism on the results are discussed. The experimental wastewater treatment study determined that reverse osmosis produced higher purity ammonia. The findings of the combustion work suggest that ignition delays will be similar to pure ammonia if HCl is filtered from the final product.
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# CONTENTS

Acknowledgments .................................................................................................................vi

List of Figures..................................................................................................................... viii

1. Introduction....................................................................................................................... 1

2. Methodology ..................................................................................................................... 3

   2.1 Ammonia Extraction from Synthetic Wastewater........................................................ 3

   2.1.1. Membrane Filtration ............................................................................................... 3

   2.2 Combustion Simulations ............................................................................................. 4

3. Results and Discussion....................................................................................................... 6

   3.1. Ammonia Extraction from Synthetic Wastewater....................................................... 6

   3.1.1 Pure Water Flux Measurements................................................................................ 6

   3.1.2. Impact of Transmembrane Pressure on Ammonia Recovery................................. 7

   3.2 Combustion Simulation Results................................................................................... 7

   3.2.1. Mechanism Study .................................................................................................... 7

   3.2.2 Ignition Delay Study................................................................................................. 8

   3.2.3 Detailed HCl Impurity Study ................................................................................. 10

4. Summary & Conclusions ................................................................................................. 15

Bibliography ........................................................................................................................ 16

About the Authors............................................................................................................... 18
LIST OF FIGURES

Figure 1: Illustration of Experimental Setup for Ammonia Recovery from Synthetic Wastewater Solution ................................................................. 3

Figure 2: Ammonia Measurement Using Hack Spectrometry ................................................................. 4

Figure 3. Example of Typical Temperature Rise in Ignition Simulation with an Illustration of the Calculated Ignition Delay Time. ........................................ 5

Figure 4: Measured Pure Water Flux of Tested NF Membrane at Room Temperature ........ 6

Figure 5: Measured Pure Water Flux of Tested RO Membrane at Room Temperature........ 6

Figure 6. Influence of Transmembrane Pressure on Ammonia Recovery................................. 7

Figure 7. Ignition Delay Based on Initial Temperature for Each Impurity at 10,000 ppm..... 9

Figure 8. Influence of Pressure on the Ignition Delay. All Impurities are Shown with Concentrations of 10,000 ppm ............................................................ 9

Figure 9. Effect of Equivalence Ratio on the Ignition Delay for Each Impurity at Levels of 10,000 ppm ................................................................. 10

Figure 10. Ignition Delay Dependence on HCl Impurities Level................................................. 11

Figure 11. ClOH Mole Fraction Profile Normalized for Initial Concentration of HCl........ 12

Figure 12. ClO Mole Fraction Profile Normalized for Initial Concentration of HCl.......... 12

Figure 13. OH Mole Fraction Profile Normalized for Initial Concentration of HCl ......... 13

Figure 14. Early Time Evolution of Normalized OH Profiles ...................................................... 13
1. Introduction

Goods and supplies are often transported via vehicles using internal combustion engines. These engines burn hydrocarbon fossil fuels, producing greenhouse gases such as carbon dioxide. Since 2000, transportation sources have produced over 160 million tons of carbon dioxide each year. Approximately 40% of the carbon dioxide emissions in California during 2017 were from transportation sources (California Greenhouse Gas Emissions for 2000 to 2017: Trends of Emissions and Other Indicators). These values do not include the emissions from refinery processes that supply the fuel, thus increasing the total contribution and reducing the sustainability of petroleum-derived fuels. Bio-derived fuels have been investigated to improve sustainability, but these hydrocarbon fuels will also produce gases, such as carbon dioxides. As such, a fuel without carbon content is needed to reduce carbon dioxide emissions. Furthermore, this fuel should be from a sustainable source and have the ability to limit other pollutants.

Because ammonia (NH₃) does not contain any carbon, it is a candidate to replace hydrocarbon fuels. The products of NH₃ combustion are water and nitrogen gas. Zamfirescu and Dincer (Zamfirescu & Dincer, 2008) showed that the cost per energy content of ammonia is less than half of other commonly used hydrocarbons and has similar energy density to compressed natural gas. The higher latent heat of evaporation compared to other hydrocarbon fuels means that ammonia may be used to cool the engine as well. However, challenges with the combustion performance of ammonia flames have led researchers to study mixtures of hydrocarbon fuels and ammonia. Small amounts (10%, by mole) of Diesel fuel added to ammonia have been shown to decrease the ignition delay within a compression ignition engine (Reiter & Kong, 2008). This approach of using mixtures can directly result in carbon dioxide emissions with acceptable combustion properties in engines. In addition, recovering and using ammonia from wastewaters would reduce the human impact on the planet’s nitrogen cycles (Angus, 2019).

The sustainability of ammonia fuel can be realized by recovering ammonia from the wastewater streams. The possibility of capturing ammonia from wastewaters represents a means of both reducing damaged aquatic environments and supporting the reduction of greenhouse gas emissions, resulting from a reduction in the need for ammonia production via traditional methods. Generally, wastewater contains a high amount of organic materials and minerals, such as nitrogen, phosphorus, and potassium. Recently, anaerobic membrane bioreactor (AnMBR) and microbial electrochemical (ME) processes have become the main emerging technologies to recover energy from wastewater. However, the significant escape of nutrients (mainly ammonia and phosphate salts) in their effluent streams impede their capabilities as recovery processes (Wichtpan, Jaewoo, Kunli, Karahan, & Tae-Hyun, 2018). Thus, there is a need to design and implement a process that can eco-efficiently recover high purity ammonia from the AnMBR and ME effluents with minimum chemical and energy demand.

For use as a fuel, purification of ammonia is desirable to minimize unwanted contaminants that could be pollutants and to provide a consistent energy-dense fuel. Contaminants may change the
energy density, ignition delay, and overall combustion chemistry. Thus, it is of prime importance to design a green process that can recover ammonia with minimal impurity and operational costs. While there is a significant body of knowledge pertaining to the assessment of the physical and biological methods to eliminate the impact of ammonia contamination and water pollution, little is known regarding the viability of capturing high purity ammonia and its application as a green fuel. In recent years, a number of studies have documented that membrane-based processes exhibited some potential for ammonia recovery from wastewater and production of ammonia-based fertilizers (Zhang, Ma, He, & Waite, 2018). Among the examined conventional and membrane-based processes, membrane contactors and membrane distillation show promise for recovery of high purity ammonia from wastewaters, as they are fairly simple in concept and enjoy a high selectivity and productivity, moderate cost to performance ratio with compact modular design, and considerably less environmental footprints (Darestani, Haigh, Couporthwaite, Millar, & Nghiem, 2017).

Even though purification is ideal for consistent combustion performance, removing these materials requires additional hardware and cost energy. There may be benefits to minimizing the degree of purification of the ammonia solution after passing through the filter. The energy saving associated with removing the purification step is straightforward, but one contaminant—water—is known to aid in the reduction of nitrogen oxides (NOx). The addition of water vapor to combustion engines has been shown reduce NOx emissions. Nitrogen oxides can form via the thermal mechanism, which is temperature dependent, or the prompt mechanism, which is more prevalent at lower temperatures. Injection of water vapor into combustors has resulted in substantial decreases in NOx emissions (Glassman, 2015). Water molecules react with atomic oxygen to create OH radicals. The OH molecules are less effective than the O atoms in attacking N2, thus reducing the NOx concentration.

Water and other impurities, such as carbon oxides and hydrogen chloride, may also be present in the captured ammonia. Although the influence of water vapor on combustion has been documented, the effect other impurities has not been investigated previously. Similarly, details of the separation process, such as ammonia purity, influence filtration conditions, etc., are not known. The goal of this project is to determine how these potential impurities influence ignition and how the filtration will influence ammonia purity. To this end, we report initial work filtering synthetic wastewater solutions to quantify the amount of ammonia recovered and conduct a computational study on the ignition of ammonia with impurities expected from the wastewater treatment process. Discussion on how impurities influence the combustion chemistry is included.
2. Methodology

This project had two tasks: (1) the simulation of combustion chemistry and (2) the recovery of ammonia from wastewater. The former was led by Dr. Kalman and the latter by Dr. Haddad.

2.1 Ammonia Extraction from Synthetic Wastewater

2.1.1. Membrane Filtration

Synthetic solutions containing ammonia chloride and ammonia bicarbonate (10 g/L) were used to study the impact of membrane type, transmembrane pressure, and cross flow velocity on ammonia recovery (Figure 1). All experiments were done in batch mode and cross flow filtration at room temperature. To maintain the feed characteristics, constant, permeate, and concentrate streams were sent back to the feed tank. Nanofiltration and reverse osmosis membranes were used, and pure water flux was measured prior to and after each experiment to check membrane integrity.

The transmembrane pressure varied from 10–14 bar, while the crossflow velocity varied from 0.2–0.4 m/s. The concentration of ammonia in feed, concentrate, and permeate lines were measured using Hach spectrometer (Figure 2).
The amount of ammonia recovered in each experiment calculate as:

\[ R (\%) = \frac{C_{\text{feed}} - C_{\text{permeate}}}{C_{\text{feed}}} \times 100 \]

where R represents the ammonia recovery and C shows the concentration of ammonia.

2.2 Combustion Simulations

Simulations were all conducted using Cantera (version 2.4.0) (Goodwin, Speth, & Moffat, 2018), an open-source combustion code. The goal of this work is to investigate the effect of ignition delay in compression ignition engines (CI). In CI engines, combustion occurs at an approximate isobaric manner. As such, all studies were conducted with constant pressure, zero-dimensional reactor with the initial pressure and temperature range being representative of the thermodynamic conditions expected in an IC engine. Pressures were varied from 30–80 atm and Initial temperatures from 1050–1200 K. The equivalence ratio of NH₃-air mixtures also varied from 0.5 to 2.5, representing a wider range than what would typically be expected in practice. Impurities of H₂O, CO, CO₂, and HCl were investigated from 0–10,000 ppm (compared to ammonia, not the overall mixture).

The ammonia combustion mechanism published by Dai et al. (Dai, Gersen, Glarborg, Levinsky, & Mokhov, 2020) was used to simulate the detailed chemistry. This mechanism includes both nitrogen and hydrocarbon chemistry. It should be noted that this mechanism was chosen due to a combination of its recent development, inclusion of hydrocarbons for the water, CO, and CO₂ chemistry, and studied with experimental conditions similar to those in the current work. The influence of all impurities, except HCl, were included in the mechanism. Additional reactions and species were added to the mechanism in order to account for the chlorine chemistry resulting from HCl. These reactions were taken from the mechanism published by Gross and Beckstead (Gross & Beckstead, 2011).
All ignition delays were calculated based upon the rise in temperature. A representative time-temperature plot is shown in Figure 3. The temperature is approximately constant at the initial set temperature for a short time before a steep rise is observed. The time at which the temperature reaches 300 K greater than the initial temperature is taken to be the ignition delay. Additional details, such as mole fraction profiles, rate of progress for elementary reactions, etc. were investigated to understand changes to the combustion chemistry due to the impurities.

Figure 3. Example of Typical Temperature Rise in Ignition Simulation with an Illustration of the Calculated Ignition Delay Time
3. Results and Discussion

3.1. Ammonia Extraction from Synthetic Wastewater

3.1.1 Pure Water Flux Measurements

Figures 4 and 5 depict the pure water flux of NF and RO membranes measured prior to and after each ammonia recovery test. As expected, for all the applied transmembrane pressure, the pure water fluxes of the NF membrane were significantly higher than the RO membrane. This result is because NF membranes are porous while RO membranes are dense.

Figure 4: Measured Pure Water Flux of Tested NF Membrane at Room Temperature

Figure 5: Measured Pure Water Flux of Tested RO Membrane at Room Temperature
3.1.2. Impact of Transmembrane Pressure on Ammonia Recovery

More than 90% of ammonia was recovered when the reverse osmosis membrane (RO) was used, while more than 80% ammonia recovery was achieved once the filtration was done with the nanofiltration membrane (NF), as seen in Figure 6. Increasing the transmembrane pressure slightly increased the ammonia recovery for both of the examined membranes. The higher recovery of ammonia with RO membranes can be related to the membrane’s properties, as they are effectively non-porous and apply a higher pressure than the osmotic pressure, which resulted in water transfer across the membrane. NF membranes, on the other hand, are porous, and the mass transfer across the membrane was due to the ion size as well as Donnan exclusion.

![Figure 6. Influence of Transmembrane Pressure on Ammonia Recovery](image)

3.2 Combustion Simulation Results

The results from the filtration work demonstrate that up to ~20% of the product may contain impurities. Although the composition has not been completely identified, it is clear that impurities may be on the order of 1 percent without additional purification processes. It is necessary to investigate the effect this impurity level has on ignition.

3.2.1. Mechanism Study

Prior to conducting calculations, the sensitivity of the kinetics of the modified Dai et al. (Dai et al., 2020) mechanism was conducted. The added reactions from Gross and Beckstead (Gross & Beckstead, 2011) were compiled for ammonium perchlorate combustion and thus may not be valid for the conditions of the current effort. The calculated ignition delays with the added reaction shortened as much as 80% compared to the original Dai mechanism. A sensitivity analysis

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identified several reactions that were responsible for those changes. The following reactions (R1–R3) were identified by Ermolin et al. (Ermolin, Korobeinichev, Tereshchenko, & Fomin, 1982) for ammonium perchlorate combustion.

\[
\begin{align*}
\text{HNO} + \text{O}_2 & \iff \text{NO}_2 + \text{OH} \quad \text{(R1)} \\
\text{NH}_2 + \text{NO}_2 & \iff 2 \text{HNO} \quad \text{(R2)} \\
\text{NH}_2 + \text{NO} & \iff \text{H} + \text{N}_2 + \text{OH} \quad \text{(R3)}
\end{align*}
\]

These reactions decreased the ignition delay by artificially increasing the radical concentrations (e.g., H, OH). Reaction R1 decreased ignition delay by ~10%, whereas R2 and R3 each decreased ignition delay by about 60%. Reaction R3, in particular, seems unlikely to occur under the current conditions, but several branches of the NH$_2$ + NO reaction are possible. The relatively low temperatures, in a combustion sense, make it unlikely for H and OH to be produced as suggested by R3. It is more reasonable that H$_2$O is produced. This initial study included these three reactions; however, these findings are important beyond the initial motivation of this effort.

3.2.2 Ignition Delay Study

The ignition delay time was calculated for a range of conditions and impurities (10,000 ppm shown). The effect of the initial temperature is shown in Figure 7. All cases show an expected decrease in ignition delay, as the temperature rises due to the Arrhenius reaction rates. A similar ignition delay decrease was observed in Figure 8, as initial pressure increases due to the higher rate of molecular collisions. Figure 9 shows the effect of equivalence ratio. All conditions, except the HCl impurities, show a similar dependence for decreasing ignition delay, as the mixture becomes more fuel rich.

Many cases (concentrations of each impurity) did not result in a change to ignition delay of more than 0.5%. Impurities of HCl at 10,000 ppm increased ignition delay time by as much as ~50%, whereas H$_2$O and CO decreased ignition delay time by less than 5%. Carbon monoxide likely reduces the ignition delay since it is an additional fuel itself. The addition of CO$_2$ showed negligible change over a range of temperature, pressure, and equivalence ratios. Differences regarding the influence of each impurity become minimal at higher temperatures. The effect of radical consumption by Cl-based species is likely offset by the increased reaction rates by the higher temperatures. Part of that reasoning is due to the consumption of OH by ClOH. The reaction rate coefficient for ClOH + OH is constant with temperature (activation energy is zero), whereas NH$_3$ + OH has a non-zero activation energy. The reaction rate coefficient for ClOH + OH is greater at lower temperatures and thus more readily consumed OH. This depletion of the radical pool delays ignition and hinders H-abstraction from NH$_3$. The longer ignition delay for fuel rich mixtures with HCl is affected in a similar fashion. Fuel rich mixtures will have lower concentrations of OH, which makes the consumption of OH by ClOH more critical.
Figure 7. Ignition Delay Based on Initial Temperature for each Impurity at 10,000 ppm

Figure 8. Influence of Pressure on the Ignition Delay. All Impurities are Shown with Concentrations of 10,000 ppm
3.2.3 Detailed HCl Impurity Study

The large change in ignition delay with HCl led us to explore the process in more detail. The concentration of HCl was varied from 0 to 1000 ppm in 1 ppm increments. Stoichiometric mixtures were all studied under fixed pressure (50 atm) and initial temperature (1150 K) conditions. The ignition delay initially decreased before monotonically increasing for large concentrations of HCl. Interestingly and unexpectedly, a critical value of HCl concentration exists that results in a minimum ignition delay at 49 ppm, as illustrated in Figure 10.
Figure 10. Ignition Delay Dependence on HCl Impurities Level

Chlorine-based species profiles were studied to try to understand the cause of this observation and are shown in Figures 11–14. Mole fractions of the species were normalized by the initial concentration of HCl for comparison purposes. An initial decrease in HCl mole fraction coincided with an increase in Cl₂ and ClOH. The first step is likely formation of ClOH from the reaction of HCl+O₂. Chlorine monoxide can then be produced (with H₂O) through the reaction of ClOH + OH, which will occur faster than H-abstraction of NH₃ by OH at lower temperatures. The mole fraction of ClO is much less likely due to its reactivity. ClO reacts with Cl or ClOH to produce Cl₂. Those reactions occur earlier in the process to build the mole fraction of Cl₂. ClO also abstracts an H-atom from NH₃, which can also build the concentration of ClOH. Eventually, ClO concentration increases and likely starts to form HCl through reactions with either H₂O or NH₂. At this point, the OH radical pool increases so that the reaction with NH₃ becomes more prevalent and the oxidation process continues with temperature increasing. The increase in temperature further increases the reaction rate of NH₃ + OH but does not have the same effect on the ClOH + OH reaction, as discussed earlier. Thermal runaway proceeds at this point.
Figure 11. ClOH Mole Fraction Profile Normalized for Initial Concentration of HCl

![Graph showing the normalized ClOH mole fraction profile for different initial concentrations of HCl.](image)

Figure 12. ClO Mole Fraction Profile Normalized for Initial Concentration of HCl

![Graph showing the normalized ClO mole fraction profile for different initial concentrations of HCl.](image)
The cause for the minimum value of ignition delay observed in Figure 10 is unclear. Figures 12 and 14 show that ClO and OH mole fractions, respectively, increased at an earlier time for the 49 ppm case. This development will cause the concentration of ClO to be large enough to reform HCl. Moreover, the peak normalized mole fraction of OH (Figure 13) is greatest for the 49 ppm condition, suggesting that the ignition occurred earlier due to a larger radical pool. This observation highlights the importance of the OH radical and the competition between OH reacting with chlorine-based species instead of NH₃.
There is concern that the chlorine chemistry used may be limited in accuracy. If that is the case, then the actual changes to the combustion chemistry may differ. The current work is limited by the data available in the literature, as developing a more detailed chlorine mechanism is outside the scope of this effort. Regardless, it is likely that HCl will increase the ignition delay over most conditions. Thus, future wastewater-derive ammonia will likely need to be further filtered or purified for quality control purposes (i.e., consistent ignition in batch-to-bath variations) and/or to minimize the degradation on ignition performance. The presence of other impurities studied do not have a substantial influence on the ignition properties and are thus not a concern from that perspective.
4. Summary & Conclusions

This work focused on two aspects of developing wastewater-derived ammonia: (1) the influence of membrane conditions for filtration and (2) how impurities from the process alter the ignition delay time. The pressure across the membrane and membrane type was observed to influence the flow rate and will affect the output rate. The purity of the product ammonia was constant for a given membrane. Reverse osmosis membranes were able to produce higher-purity (90 vs. 80%) ammonia due to internal structure. Importantly, the process did not affect the integrity of the membranes. Combustion simulations showed that CO and H₂O impurities from the separation process will slightly decrease the ignition delay due to CO acting as an additional fuel and water vapor, helping produce OH radicals. This aspect shows promise for ammonia solutions dominated by those impurities, despite the reduction of the energy density. Mixtures with HCl showed an overall increase in ignition delay but, under (i.e., 49 ppm of HCl for stoichiometric mixtures initially at 1150 K and 50 bar), may decrease them. This aspect needs further investigation since chlorine chemistry under these thermodynamic conditions are not well known. Finally, several reactions related to NH₃ oxidation and NOₓ formation were identified to unrealistically decrease ignition delay times. This work shows the impurities (with the exception of HCl) expected from wastewater-derived ammonia will have minimal effect even in high (~1%) concentration levels. Further work is needed to understand the effect of HCl if this impurity cannot be filtered out of the solution with minimal energy input.
Bibliography


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Dr. Kalman is an Assistant Professor of Mechanical and Aerospace Engineering at California State University, Long Beach, since 2018. He received his B.S. (Rutgers University, 2008), M.E. (Rutgers University, 2010), and Ph.D. (University of Illinois at Urbana-Champaign, 2014) in Mechanical Engineering. Dr. Kalman spent four years as a researcher at the Naval Air-Warfare Center-Weapons Division, China Lake before joining CSULB in 2018. His expertise is in the areas of particle and multiphase combustion, non-intrusive diagnostics, and energetic materials. He has published 18 peer-reviewed articles and over 30 conference papers on those topics.

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