Synthesis, characterisation and Microscale Electrolytic Decomposition of Hydroxylammonium Nitrate (HAN) Solution

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Hydroxylammonium Nitrate, (HAN) is known as a new synthetic green monopropellant with chemical formula $NH_3OH^+NO_3^-$. In recent years, it has provided an attractive alternative for space researchers in attempt to replace conventional liquid monopropellant such as hydrazine and hydrogen peroxide (HTP) due to various advantages such as post combustion species are non-toxic and friendly [1], low freezing point low viscosity, easier storage and handling issues. In 2009, a microscale thruster using improved HAN combustion technique via electrolysis was proposed by *Wu and Yetter* in low temperature co-fired ceramic technology, (LCCT) [2].

In this paper, we first of all evaluated synthesis, purification and characrerisation of HAN solutions, follow by decomposition of high concentrated HAN solution in a carefully designed micro-combustion chamber.

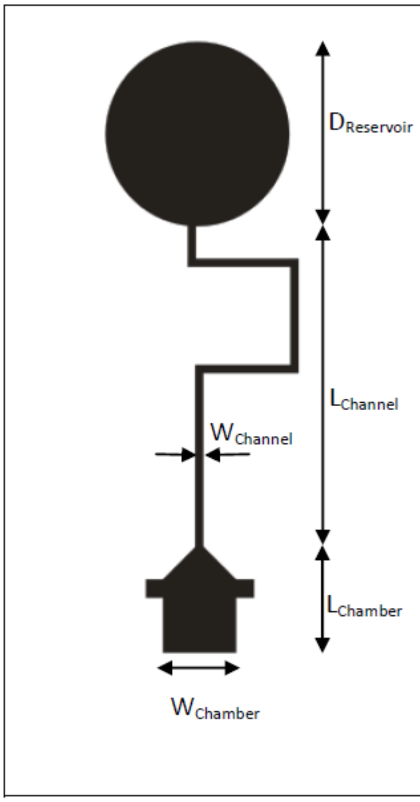
The solution was synthesised through titration of nitric acid and hydroxylamine solution, which can be further purified to high concentration of above 80 wt%. *Charles et.al* characterized HAN solution based on a density-HAN mass fraction driven empirical equation (1) [3]:

$$\rho = \frac{107.85}{96.045 - w(HAN) \times 30.99} \qquad (1)$$

By combining results from re-titration, TGA analysis and density measurement, a new empirical equation which can increase the accuracy in the prediction of solution density was obtained, as shown in Equation (2). Preliminary result shows that the characterisation highly depends on concentration of nitric acid.

$$\rho = \frac{103.9}{100.5 - w(HAN) \times 42.9} \qquad (2)$$

A microcombustion chamber as shown in Figure 1 was designed and fabricated with Polydimethylsiloxane (PDMS) using modified soft-molding method. The design consisted of a reservoir, a microchannel connecting the reservoir to the micro-combustion chamber. Copper wires were poked through the polymer to act as electrodes to initiate electrolytic decomposition of concentrated HAN solution.



Microcombustion System Dimension		
Reservoir Diameter	5 mm	
Microchannel	Width	0.225mm
	Length	15mm
Microcombustion Chamber	Width	2mm
	Length	3mm

Remark:

- The extra edges at the microcombustion chamber are the markers to insert microelectrode.
- The design thickness of the microcombustion device is 100µm.
- The diverging design from channel exit to chamber is to avoid dead volume formation of HAN in the chamber.

Figure 1: Design of a micro-combustion chamber to demonstrate electrolytic decomposition of 8- wt% of HAN solution

In the micro-combustion chamber, at *Re* = 0.18, gas bubbles of oxygen and hydrogen, diameter in the range of 50 - 480µm were formed immediately at anode and cathode respectively when the power was switched, with the formation of bubbles at anode was milder than the cathode. However, the bubbles cannot be displaced to the ambient immediately resulted in choking at the exit, as shown in Figure 2. As the flowrate was increased to *Re* = 0.53, gas released from the reaction was significant that occupying 45 – 75% of space in the chamber, causes the wall effect becomes insignificant. The flow phenomenon in the chamber was more completed with rapid and random formation of large bubbles at the chamber exit. When the flowrate was further increased to 50µl/min, at *Re* = 0.88, The overall decomposition was more rigorous that only few small bubbles formed near the electrodes while the two phase flow in the chamber is smooth without serious problem of choking at the exit.

In conclusion, the micro-combustion system was only effective in relatively high *Re* in which choking was reduced significantly. The un-decomposed HAN solution caused complex two phase flow in the chamber which is difficult to predict and reducing efficiency of the devices. The qualitative result also demonstrated that more detailed study required in order to develop more efficiencient microcombustion system based on concentrated HAN solution.

Figure 2: Electrolytic decomposition of concentrated HAN solution in the micro-combustion chamber.

Reference:

1. N. Klein, Gun Propulsion Technology. In: L. Stiefel, M. Summerfield (Eds.), (1988) Progress in Astronautics and Aeronautics, vol.109 pp.473

2. Wu, M. H. and Yetter, R.A., (2009) A novel electrolytic ignition monopropellant microthruster based on low temperature co-fired ceramic tape technology, *Lab on Chip*, vol.9, pp910-916

3. Decker, M. M., Klein, N., Freedmen, E., Leveritt, C.S., Wojciechowski, J. Q., (1987) HAN-based Liquid Propellant: Physical Properties, available: http://www.dtic.mil/cgi-bin/GetTRDoc?AD=ADA196225 [accessed 2nd November 2011]