Influence of relative humidity conditioning on aerosol and liquid chemistries in electronic cigarettes

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Meeting a Need

- With rising popularity of vapor products, effective methods for analyzing these products must be developed

- Vapor products typically contain formulations with water, propylene glycol (PG), and/or glycerin (Gly)
  - Nicotine can be included, but is not required

- PG + Gly = hygroscopic
  - Relative humidity (RH) and temperature is an important consideration when developing methodology

Current Methods

- ISO Standard for combustible products recommends RH 60% (± 2%), and 22 °C (± 1 °C) (ISO 3402:1999, 2010, ISO 3308:2012, 2012)

- CRM81 “Routine Analytical Machine for E-Cigarette Aerosol Generation and Collection – Definitions and Standard Conditions” (June 2015)
  - Specific RH and temperature not defined (only limitations)
  - Relative Humidity limit = ± 5%
  - Temperature limit = ± 2 °C

- CORESTA E-Cig Task Force collaborative study in progress to produce a recommended method for aerosolizing vapor products
  - RH = 40 – 70%
  - Temperature = 20 – 25 °C

Two Relevant Questions:

How does relative humidity affect vapor chemistry (water, nicotine)?

How does relative humidity affect liquid formulations in cartridges over time?

Experimental

E-Liquid Formulations

- Generic formulations: constant nicotine, constant PG:Gly ratio, and varied water
- E-liquids added to commercially available atomizer with commercially available battery
- Range of water concentration based on 2014 Market Survey of commercially available e-cigalike products

Sample Designation	Water (w/w %)	Nicotine (w/w %)	PG:Gly (w:w)
0% H_2O	0	2	50:50
5% H_2O	5	2	50:50
10% H_2O	10	2	50:50
15% H_2O	15	2	50:50

Vapor Analyses

- Conditions
  - 60% RH, 22°C ± 1.0°C
  - 40% RH, 24°C ± 1.0°C (actual RH = 47.4% ± 1.0)

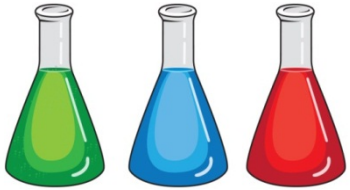
- Method
  - Weighed cartridges before and after aerosolizing
  - Samples aerosolized 24 hours post-cartridge fill
  - Puff parameters: 55 mL puff volume, 3 second duration, 30 second intervals, square wave puff profile
  - Collected ***first 100 puffs*** for each formulation (5 reps each)
  - Measured water, nicotine, PG, Gly, and TPM (using Cambridge filter pad)

Cartridge Chamber Studies Setup

Used the same generic formulations in commercially available cartridges as described previously (0, 5, 10, and 15 % water)

Experimental for Chamber Studies

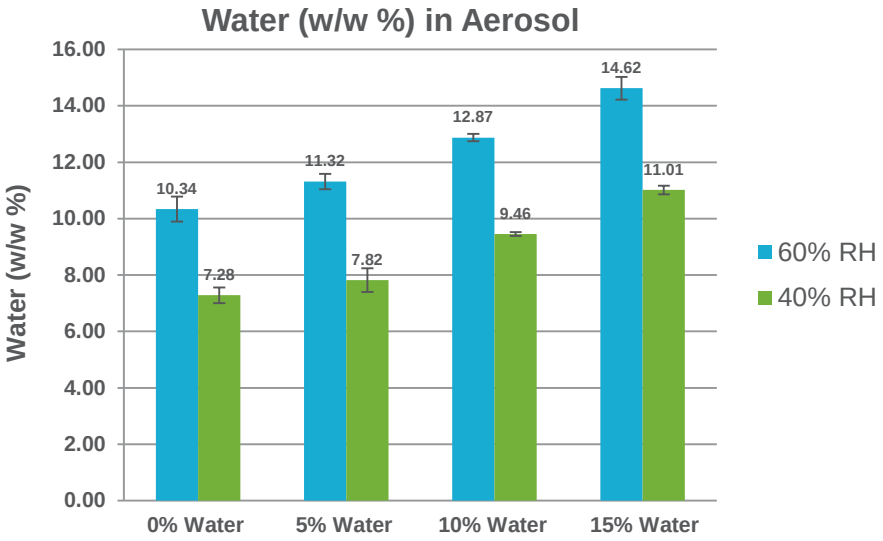
- Cartridges weighed before and after time in chamber
- Removed liquid from cartridge (centrifugation)
  - Determined % by weight based on mass of liquid removed
- Analyzed for water, nicotine, PG, and Gly

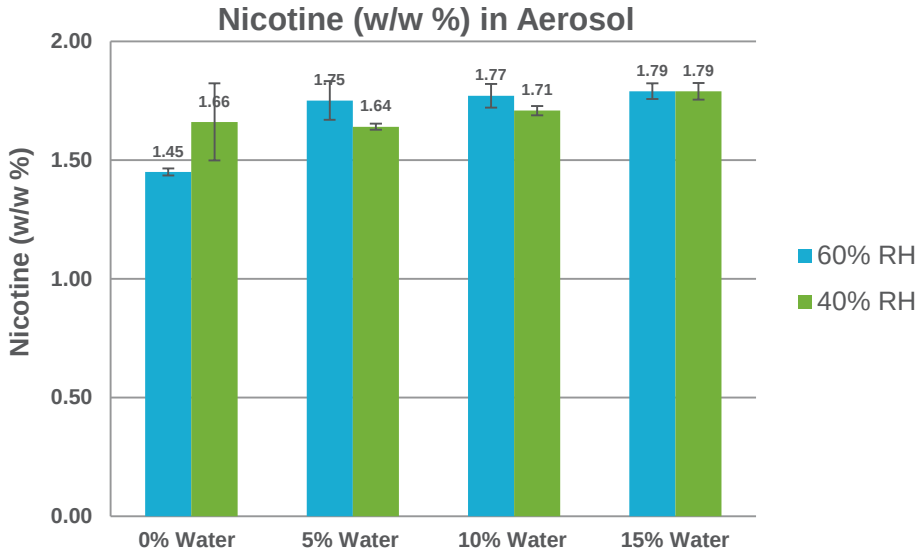


Results

Water (w/w %) and Nicotine (w/w %) Results in Aerosol

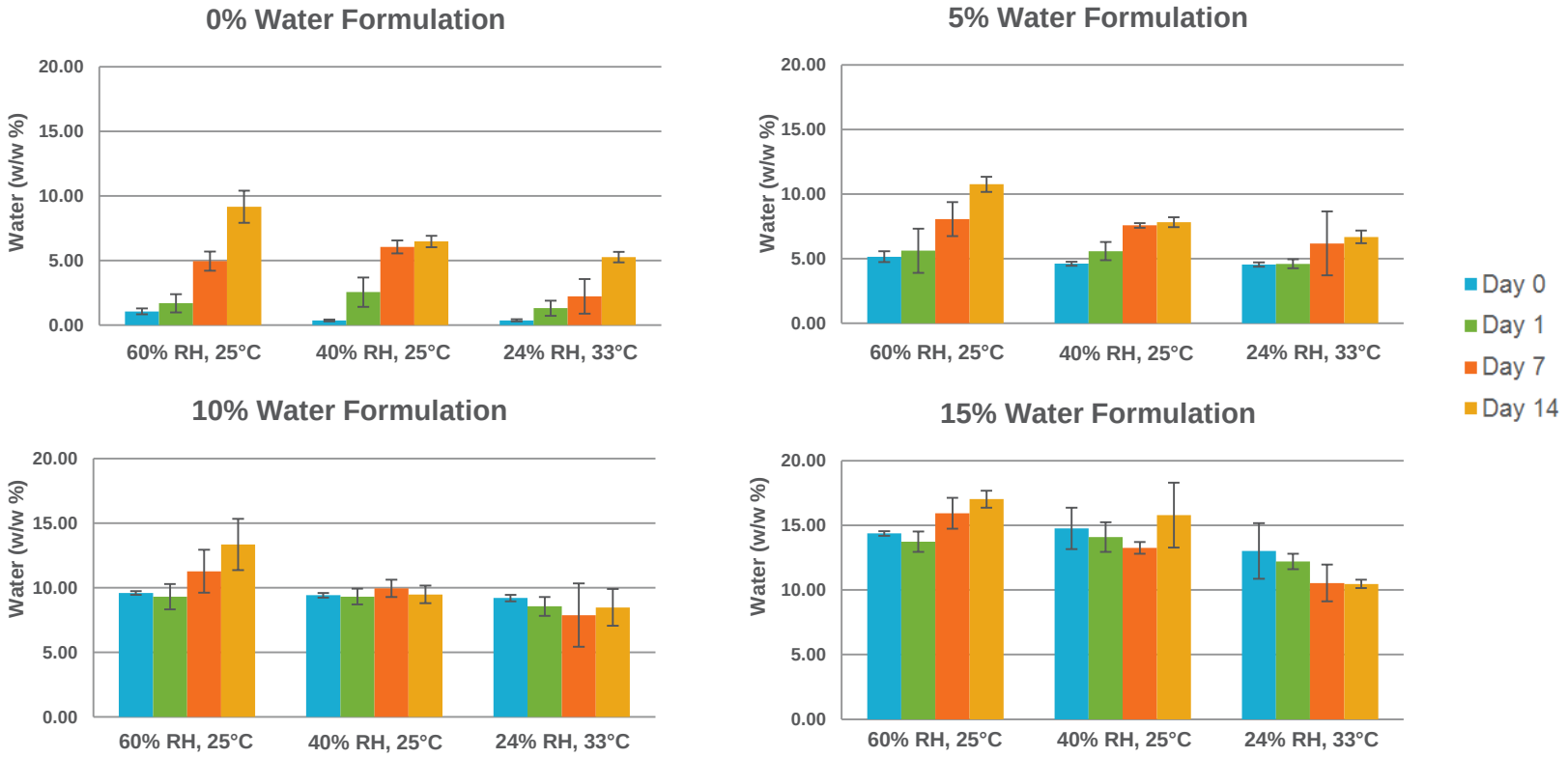
(first 100 puffs)



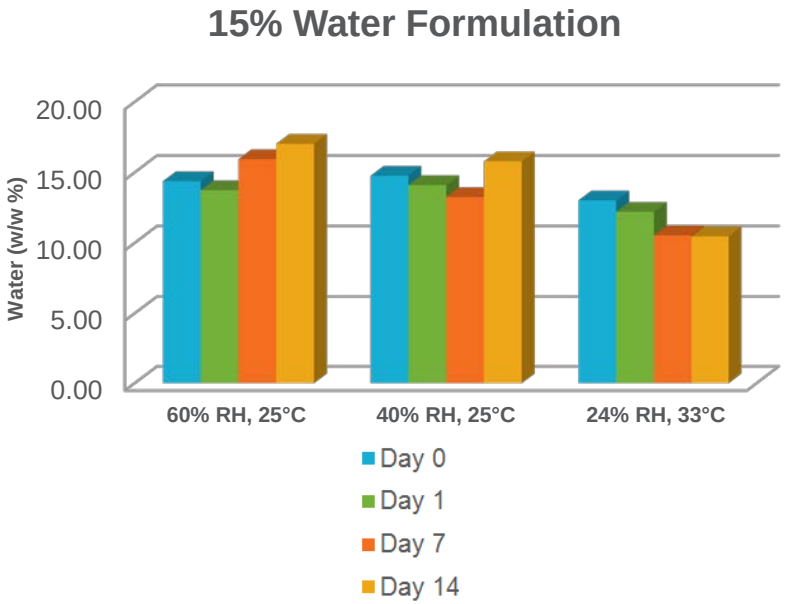


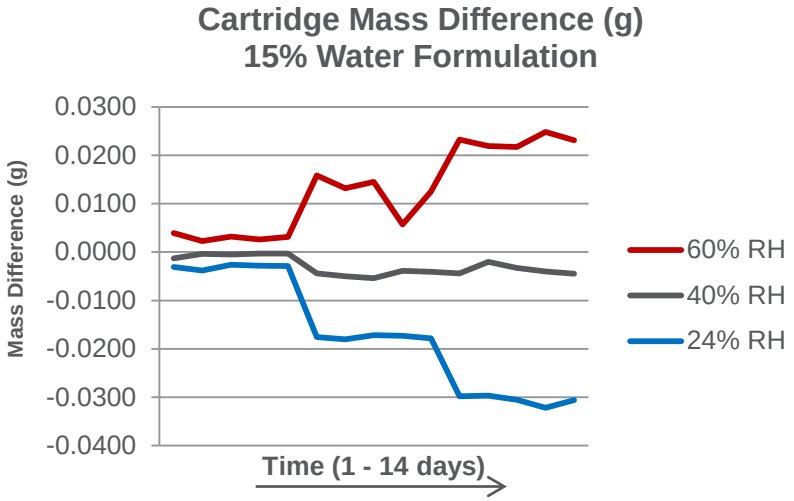
*$[nicotine]_{as\ made}$ = 2.04 ± 0.04%

Time Dependent E-Liquid % Water Results



Chamber Studies – Mass Difference





Time Dependent E-liquid Nicotine (w/w %)

The more things change…

	0% H_2O		5% H_2O		10% H_2O		15% H_2O	
Time (40% RH)	PG	Gly	PG	Gly	PG	Gly	PG	Gly
Day 0	48.72	48.51	45.84	46.02	43.34	43.78	40.40	40.78
Day 1	46.60	47.00	45.08	45.73	43.58	44.53	41.42	42.40
Day 7	44.72	45.49	43.57	44.62	42.45	43.76	40.53	41.85
Day 14	43.38	44.96	42.80	44.54	41.40	43.43	41.14	41.14

Conclusions

- Relative humidity has an affect on water concentration in both aerosol and e-liquid samples

- Storage conditions play a part in changes of samples over time (packaging not included)

- Changes appear dependent on the original water concentration of the e-liquid for both aerosol and e-liquid samples
  - difficult to “standardize” a method for every product with wide range of water concentrations

Future Investigations

- Repeat (in the winter) to achieve 40% RH for aerosol studies
- Longer time course for e-liquid chamber studies (equilibrium?)
- Different formulations: varying ratios of PG:Gly
- Aerosolize to depletion to monitor changes over life of cartridge
- Analyze for other chemicals of interest (aerosol and e-liquid)
  - Carbonyls
  - TSNAs
  - SVOs

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