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Hazardous Waste Reduction Continuation

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CHEMICAL & LIFE SCIENCE ENGINEERING

Hazardous Waste Reduction Continuation

CAPSTONE DESIGN EXPO 2017

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Project Description

Two-phase waste is produced from a silicone manufacturing process whose aqueous layer contains the following three reactions:

$$((CH_3)_3Si)_2NH + 2H_2O \rightarrow 2(CH_3)_3SiOH + NH_3$$

HMDS + 2 Water → 2 TMS + Ammonia

$$2(CH_3)_3SiOH \rightarrow (CH_3)_3Si\text{-}O\text{-}Si(CH_3)_3 + H_2O$$

2 TMS → HMDSO + Water

$$NH_3 + H_2O \rightarrow NH_4OH$$

Ammonia + Water → Ammonium Hydroxide

The primary contributor to hazardous/flammable classification of the aqueous phase is hexamethyldisiloxane or HMDSO, highlighted in red. The deliverables of this project were thus to:

- Identify a method to extract enough of the slightly soluble HMDSO from the aqueous phase to render it for disposal as non-hazardous waste.
  - Flash Point > 60°C
  - Develop a Method of HMDSO Quantification
- Design and build a small-scale prototype industrial process to carry out the method identified.

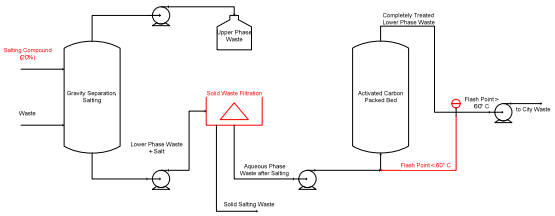


Figure 1: Proposal of wastewater treatment method from previous year. Highlighted in red are its problematic features.

Experimental Methods

- Waste was titrated with standardized 1 M H_2SO_4 and methyl red indicator to determine ammonia content in waste
- Samples of known HMDSO in water, with and without NH_3, were tested to determine an approximate concentration range which satisfies the flash point criterion
- In addition to gravity separation and an activated carbon packed bed, mixing with bleach and semi-batch stripping with N_2 gas were also tested to determine effective HMDSO removal
- HMDSO quantification was modified from the previous year using the method of standard addition and gas chromatography

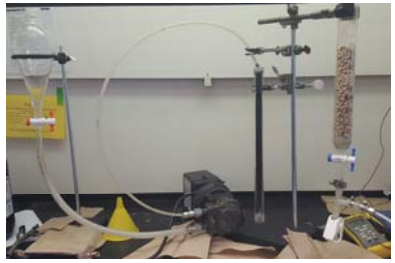


Figure 2: Bench-scale separation prototype

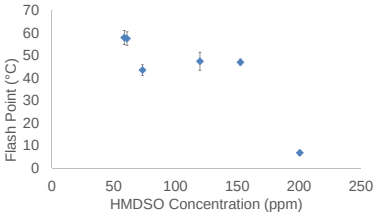


Figure 3: Flash point determinations at various HMDSO concentrations corresponding to the treatment combinations in Figure 4.

- Aqueous phase is approximately 3.26 wt. % NH_4OH
- Flash point criterion is satisfied within the window of approximately 30 – 50 ppm HMDSO
  - This threshold is given greater leniency with presence of NH_4OH
- Treatment combinations IV and VI very nearly satisfy the flash point criterion with some variation
  - Mixing with bleach gives negligible results and poses an explosive risk on reaction with residual NH_3
  - Semi-batch method may be adjusted to accommodate this

Results

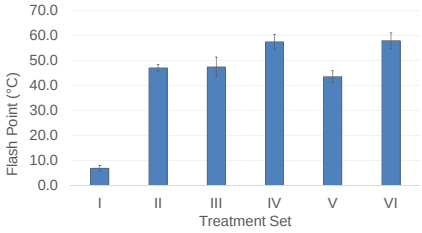
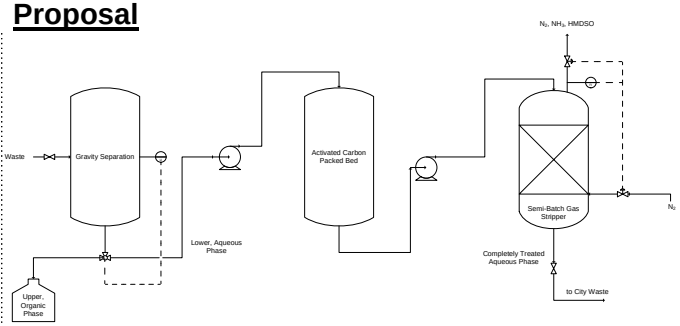


Figure 4: Flash point determinations after various treatment combinations:

- I – After Phase Separation
- II – I + Activated Carbon Packed Bed Treatment
- III – I + II + Mixing with Bleach
- IV – I + II + III + Gas Stripping
- V – After Gas Stripping Alone
- VI – IV without Bleach Mixing

Proposal



- Gas-stripping as a semi-batch operation gives flexibility in HMDSO removal to accommodate differences between bench and pilot scales
  - Testing was done with 2.5 SCFH for 1 hour
- Explosion rated equipment necessary
  - HMDSO also slowly degrades certain plastics
- Estimated scale-up process according to comparable residence times
  - $15,000 investment
  - $10,000 / year savings
  - 2 year payback period

Future Recommendations

- Time-dependent mass transfer analysis for the packed bed column and the gas stripping
- Counter-current gas stripping through the packed bed
  - Use of air in addition to nitrogen
- Explore effects of gas-stripping before packed bed given results of step V in Figure 3
- Explore effect of other packing materials (hydrophobic polymeric beads, etc.)
- Automated sample preparation must be implemented for standard addition given the strong sensitivity of the method
- Quantify NH_3 similarly to HMDSO
  - Determine strength of its effect on flash point increase
  - Determine rate of removal from gas stripping

Acknowledgments

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