Development of a Biobased Carbon Adsorbent for Treating Emerging Contaminants in Recycled Water on the International Space Station

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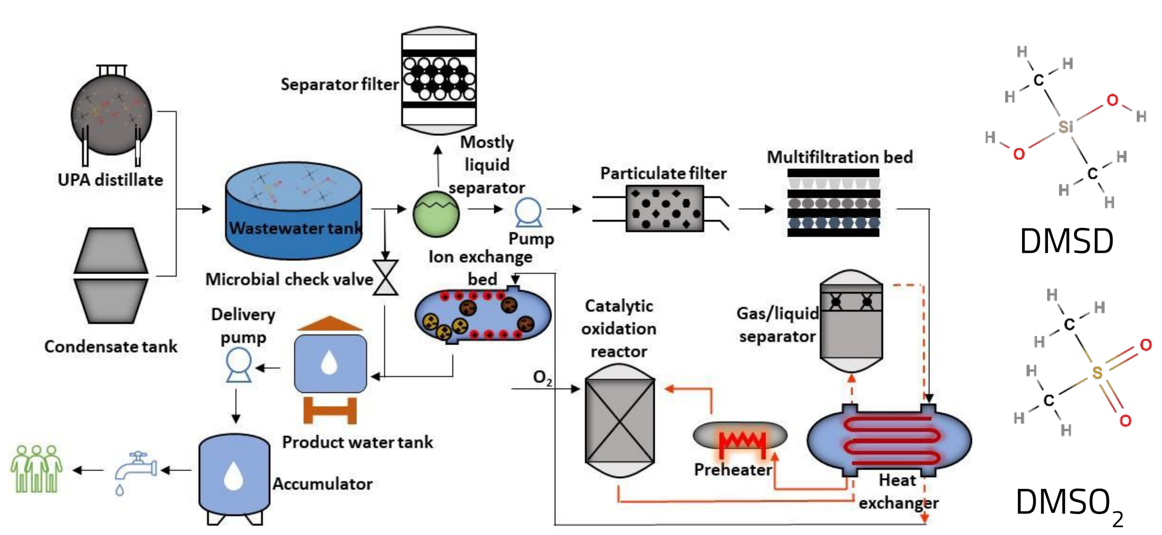
Introduction

- The International Space Station (ISS) operates as a closed system
- Water is recycled from humidity and urine and held in a wastewater tank; part of the Water Processing Assembly (WPA)
- Potable treated water is used indirectly in the Sabatier Reactor
- The Sabatier Reactor is a key component in the Environmental Control and Life Support System (ECLSS)
- Three main functions of ECLSS: water recovery, air revitalization, and oxygen generation
- Sabatier Reactor recently failed due to contamination of $DMSO_2$, dimethyl sulfone, and DMSD, dimethylsilanediol
- $DMSO_2$ and DMSD are emerging contaminants that are not removed by the media used in the WPA due to their low affinity
- $DMSO_2$ and DMSD are introduced into the water system through personal hygiene products such as lotion, conditioner, and wipes, which all contain volatile methyl siloxanes (VMS)
- VMS are decomposed into $DMSO_2$ or DMSD and are found in urine



International Space Station Water Processing Assembly - Graphic Credit: Dr. Khan's Lab

- Ambersorb 4652, a styrenic polymer adsorbent that is not biobased or sustainable, is the current media used in the multifiltration beds in WPA on the ISS
- Costly and unsustainable, Ambersorb 4652 is challenged to remove DMSD and $DMSO_2$ from recycled water
- Granular activated carbon (GAC) works similarly to a styrenic polymer adsorbent and can be made from biobased materials
- Ionic liquids are liquid molten salts at temperatures < 100 ℃ that are typically composed of large and unsymmetrical organic cations and organic or inorganic ions
- Composed of a hydrogen bond acceptor (HBA) and a hydrogen bond donor (HBD)
- Remarkable solvation ability for a broad range of natural and synthetic materials and compounds

Granular Activated Carbon (GAC)

Synthesized Ionic Liquids

Research Objectives

Objectives:
- Synthesize at least 3 biobased ionic liquids
- Analyze and quantify how much $DMSO_2$ is removed by the ionic liquids through liquid-liquid extractions
- Coat biochar with the most effective biobased ionic liquids
- Analyze and quantify how much $DMSO_2$ is removed with uncoated GAC and ionic liquid coated GAC through batch adsorption testing

Prior Work

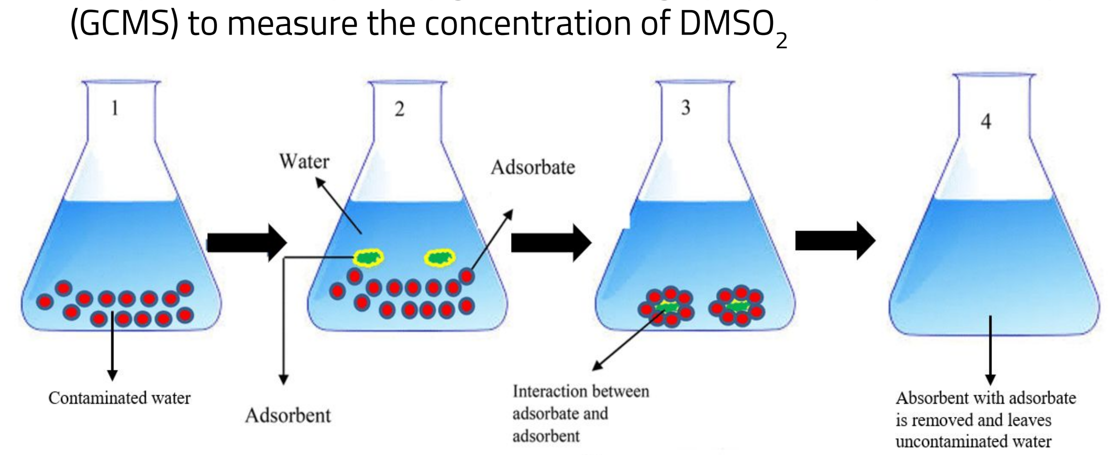
- The following ionic liquids were synthesized:

HBA	HBD	Molar Ratio	Result
Thymol	Decanoic Acid	1:1	Liquid, oil-like viscosity
Thymol	Decanoic Acid	2:1	Liquid, oil-like viscosity
Thymol	Decanoic Acid	1:2	Liquid, water-like viscosity
Thymol	Undecylenic Acid	1:1	Liquid, water-like viscosity
Thymol	Undecylenic Acid	2:1	Liquid with some recrystallization
Thymol	Undecylenic Acid	1:2	Liquid, water-like viscosity
Thymol	Dodecanoic Acid	1:1	Completely recrystallized solid
Thymol	Dodecanoic Acid	2:1	Liquid with some recrystallization
Thymol	Dodecanoic Acid	1:2	Completely recrystallized solid

Synthesized Ionic Liquid Results

Methodology

Equilibrium Batch Adsorption Testing with Uncoated GAC:
- 100 mg of coconut granular activated carbon ranging in sizes from 400-595 microns is added to a 45 mL vial containing 800 ppb $DMSO_2$
- Solution is shaken until reaching equilibrium
- Equilibrium time is determined by taking samples over time - 24, 48, 72, and 96 hours
- Samples are filtered to separate GAC from water
- Samples are analyzed by gas chromatography mass spectrometry (GCMS) to measure the concentration of $DMSO_2$

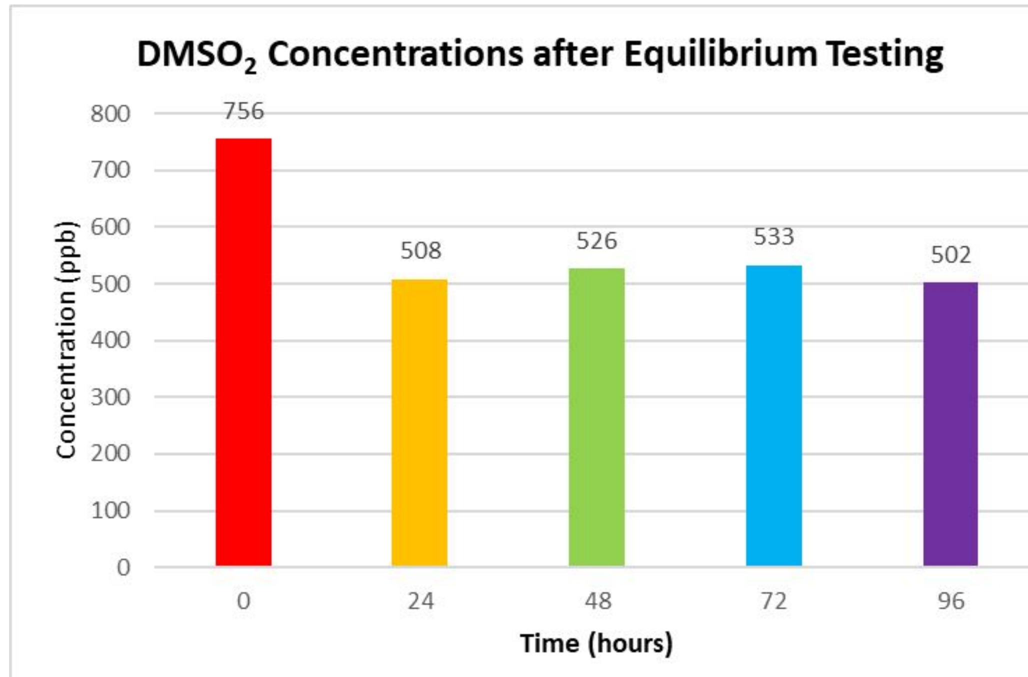


Batch Adsorption Testing

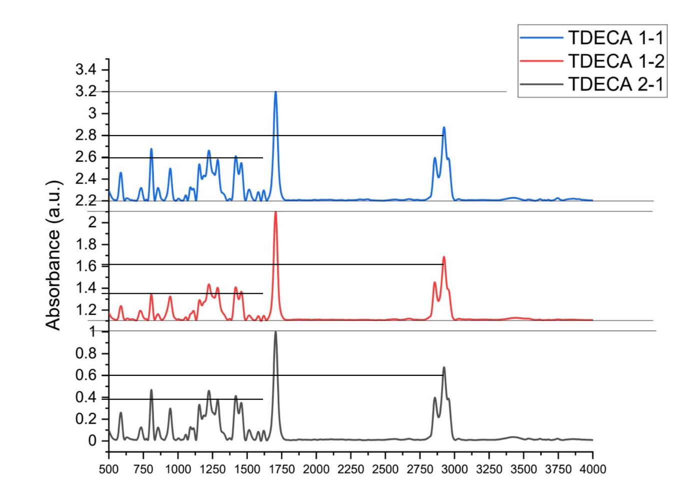
Extractions:
- In order to run analysis on the samples, the water must be absorbed (by $MgSO_4$) and the contaminant must be transferred to tetrahydrofuran (THF)
- 0.5 mL of sample, 1 mL of THF, and 0.5 g of $MgSO_4$ are added to a vial
- Remaining liquid is removed from the vial and moved to another vial containing 0.2 g of $MgSO_4$
- Wait for 10 minutes to ensure complete adsorption of water
- The remaining liquid is removed using glass syringes into vials ready for gas chromatography mass spectrometry (GCMS) analysis

Extraction Vials

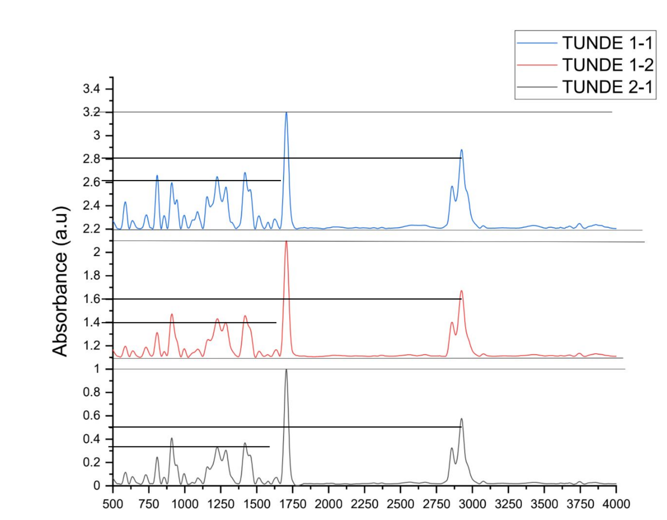
Results



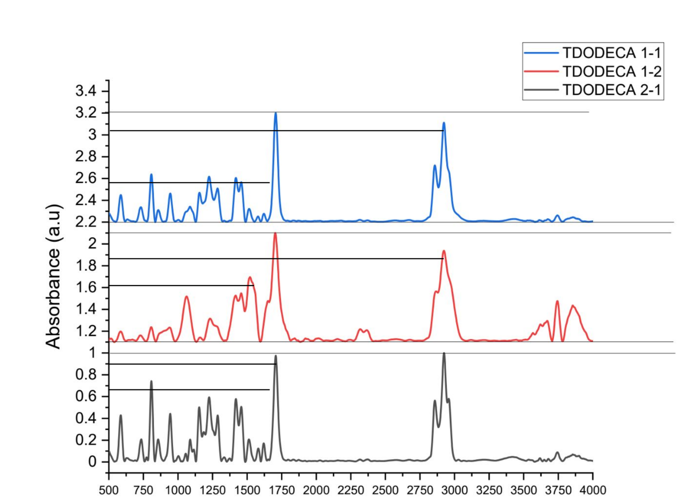
Uncoated GAC Equilibrium Batch Adsorption Test Results



Thymol and Decanoic Acid:
- Absorbance intensities differ at 2800 and 2900 peak (C-H stretching)
- Similar absorbance intensities at 1735 peak (C=O stretching)
- Small differences in intensity overall, 2:1 ratio more intense



Thymol and Undecylenic Acid:
- Higher absorbance at 2800 and 2900 peak for 1:1 ratio (C-H stretching)
- 2:1 ratio shows very low to almost negative intensity, and was an unsuccessful ionic liquid
- Absorbance intensity is higher in 1:1 ratio than 2:1 ratio



Thymol and Dodecanoic Acid:
- Much higher absorbance for all compared to other ionic liquids
- 2:1 ratio shows the highest absorbance for every peak
- All synthesized ionic liquids were not successful

Discussion/Conclusion

Batch Adsorption Testing:
- 0 hours started with 756 ppb of $DMSO_2$
- Over 24-96 hours, concentration stayed relatively the same around 515 ppb $DMSO_2$
- 24 hours can be determined as the equilibrium time with only a 32% removal
- Demonstrates a need for ionic liquid coated GAC since uncoated GAC is not sufficient in removing $DMSO_2$ from water

Extractions:
- Water was successfully absorbed by $MgSO_4$, contaminant was transferred to THF phase for GCMS analysis

FTIR Characterization:
- Peaks show that all ionic liquid trials contained both a HBD and HBA
- X-axis location of peaks is consistent with ionic liquid formation across all thymol and decanoic acid liquids
- X-axis location of peaks is not consistent with ionic liquid formation across all thymol and dodecanoic acid liquids

Future Work

- Analysis by GCMS will be completed to determine the $DMSO_2$ adsorption by liquid-liquid extractions
- The best two ionic liquids, determined by analysis, will be coated onto biochar
- The two ionic liquid coated GACs will go through batch adsorption tests - equilibrium, isotherm, and kinetic testing
- The results of the adsorption experiments will be analyzed to determine the removal of $DMSO_2$
- If removal was successful, the ionic liquid coated biochar would be a good recommendation to fix the Sabatier Reactor

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References

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