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Functional Additives to Enhance PFAS-Free Fire Suppressants

WP22-3284 Project Outbrief

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The research objectives of this project are as follows:
- Identify commercially available additives for existing PFAS-free fire-fighting foams that can improve their fire-fighting ability, with an ultimate goal of meeting requirements in MIL-PRF-24385
- Metrics for success: improved physical properties of foam-additive mixtures; improved fire suppression and burn back tests in large scale testing (28 ft2 pan)

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WP22-3284: Functional Additives to Enhance PFAS-Free Fire Suppressants

Performers

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Jerry Back, *Jensen-Hughes*

Technology Focus

- *Due to the DoD requirement to stop use of PFAS containing AFFF by Oct. 2024, it is urgent to find a drop-in solution for AFFF that can meet the MIL-PRF-24385*
- *This effort focuses on evaluating existing PFAS-free firefighting foams, which do not meet MIL-PRF-24385, and finding functional additives to enhance their performance*

Research Objectives

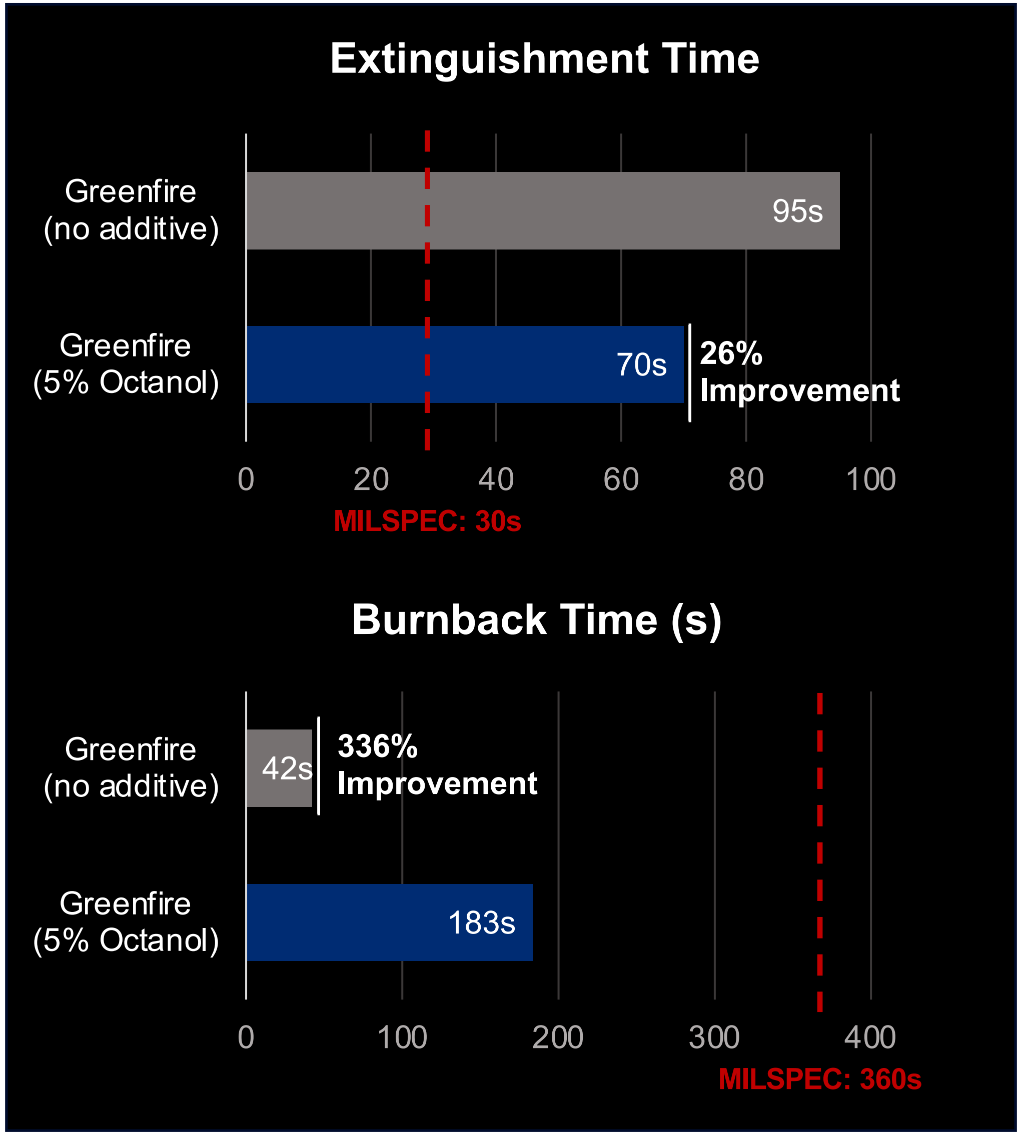
- *Identify commercially available additives for existing PFAS-free fire-fighting foams that can improve their fire-fighting ability, with an ultimate goal of meeting requirements in MIL-PRF-24385*
- *Metrics for success: improved physical properties of foam-additive mixtures; improved fire suppression and burn back tests in large scale testing (28 ft^2 pan)*

Project Progress and Results

- *To date, this work has identified two promising COTS additives, octanol and biochar, that function in two different PFAS-free foams to improve their fire fighting performance*
- ***5% octanol in Greenfire** improved time to extinguish by 26% and burnback time by 336%*
- ***10% biochar in National Foams** improved time to extinguish by 12% and burnback time by 15%*

Technology Transition

- *Future efforts will focus on continuing to optimize promising formulations and additives. We will accelerate optimization by leveraging computational modeling to understand behavior of foam at the molecular scale, connecting fire suppression and burnback performance to materials properties and identifying new additives to further enhance performance in multiple use-case scenarios*
- *We will establish partnerships with PFAS-free foam manufacturers, via APL's Office of Technology Transfer*

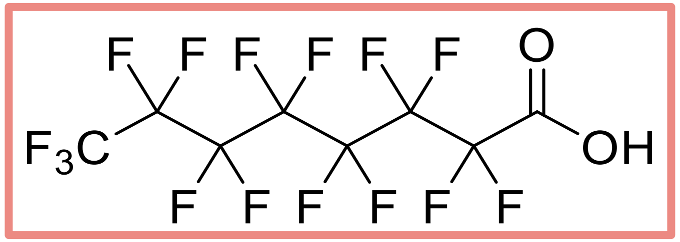


AFFF and Finding ‘Green’ Alternatives

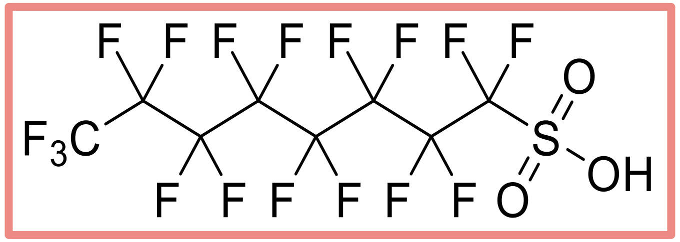
AFFF are water-based fire suppressing mixtures typically containing a **hydrocarbon-based surfactant** (sodium alkyl sulfate) and a **fluorosurfactant** (PFOA, PFOS) used to extinguish liquid-fuel based fires

High thermal and chemical stability, highly **oleophobic** – promotes continuous film formation over the liquid-fuel and enhances stability once foam is formed; essential to fire fighting efforts

PFAS have now been identified as extremely toxic, including association with developmental issues, cancers, liver damage, etc.



Perfluorooctanoic acid (PFOA)



Perfluorooctanesulfonic acid (PFOS)

AFFF and Finding ‘Green’ Alternatives

AFFF are water-based fire suppressing mixtures typically containing a **hydrocarbon-based surfactant** (sodium alkyl sulfate) and a **fluorosurfactant** (PFOA, PFOS) used to extinguish liquid-fuel based fires

- The US Military and DoD have announced plans to stop using PFAS containing AFFF by October 2024
- No PFAS free foams currently meet the requirements for military use specified in MIL-PRF-24385*
- Alternatives typically attempt to mimic oleophobicity and high temperature stability of the compounds
  - *What are some additives that could help improve these properties on an accelerated timeline?*

Project Goals:

Identify additives for existing PFAS-free fire-fighting foams that can improve their fire-fighting ability and meet the Mil-Spec

Task	Milestones
1) Literature Review	• Identify major components of COTS Class B foam concentrates • Identify and analyze COTS additives
2) Foam down-selection and characterization	• Evaluation of existing foams to set standard • Down-select additives for testing • Evaluate additive loading, water, content and commercial foam on performance • Study effect of individual additives on foam system
3) Small scale testing of foam extinguishing performance	• Assemble test apparatus • Test selected foams
4) Testing at Jensen Hughes	• Create quantities of foam needed for large scale testing • Test foams under Jensen Hughes supervision
5) Stability Testing	• Create quantities of foam needed for testing • Set up foams for aging study • Analyze effect of aging on firefighting performance

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Task 1. PFAS-free concentrate identification and enhancement through the introduction of oleophobic compounds

Non-fluorinated additives	Reasoning
1-dodecanol	foaming agent, emulsifier
1-octanol	viscosity controller
octanoic acid	hydrophobic, film forming
octane-1-sulfonic acid	foaming agent
dodecanoic acid	freezing point depressor, surface active agent
dodecane-1-sulfonic acid	ion-associating reagent

Green: high priority, Yellow: medium priority, Red: low priority

Non-fluorinated additives	Reasoning
biochars	edge testing, research indicates as good fire retardant, phosphorus research shows that production of char suffocates fire
salt	literature shows use with AFFF concentrate in the form of salt water is common
baking soda	large quantity needed to put out large scale fires
siloxanes	promising results when used in AFFF as PFAS replacement
silicones	referred to as siloxane in lit
silica aerogels	phenolic silica aerogels are great fire retardants: low thermal degradation, low heat release rate, retain structure for extended time periods at >1000 deg C temps
phosphates	(RDP) super effective FR additive
betaine compounds	no info found
oleophobic coatings	most oleophoic coatings contain PFAS, PFPE etc. (surfactis, aculon)
Magnesium oxide (dolomite)	Has been used as a flame retardant and smoke suppressor

Task 1. PFAS-free concentrate identification and enhancement through the introduction of oleophobic compounds

PFAS-free foams considered in this study:

- Firestopper, *discontinued due to inconsistent results and documented issues with product*
- **Greenfire Firefighting Foam** (GFFF)
- **National Foams**

PFAS-free Foam	Surface Tension (mN/m)		Foam Expansion Ratio (no units)		Viscosity (cSt)	
	3%	6%	3%	6%	3%	6%
Greenfire	34.9	35.0	10.44	9.29	1.52	1.19
National Foams	35.53	ND	8.59	ND	1.05	ND

*NF only tested at 3% due to time and budget constraints

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Task 2. Identify and characterize selected additive and foam mixtures

Impact of additives on different foam formulations were tested:

- Additives were tested at 1, 5, and 10% volume/weight loading in green foam
- Combinations that performed poorly or resulted in ”non-ideal” chemical characteristics were removed from consideration

Requirement	Values	
	Type 3	Type 6
Viscosity, centistokes Maximum at 5℃ Minimum at 25℃	 20 2	 10 2
pH	7.0 to 8.5	7.0 to 8.5
Spreading coefficient, minimum	3	3
Foamability Foam expansion, minimum Foam 25% drainage time, minutes, minimum	 5.0 2.5	 5.0 2.5

From MIL-24385

Task 2. Identify and characterize selected additive and foam mixtures

Key properties related to firefighting capability were assessed for each foam-additive mixture

Viscosity: *The resistance of a fluid to a change in shape or movement of neighboring portions relative to one another, utilizing capillary viscometers*

$$\eta = \frac{\tau}{\gamma}$$

η = dynamic viscosity
τ = shear stress
γ = shear rate

Goal: 2-20 Cs, temp dependent

Spreading coefficient: *Measure of of a liquid's tendency to spread on a second liquid (here, cyclohexane)*

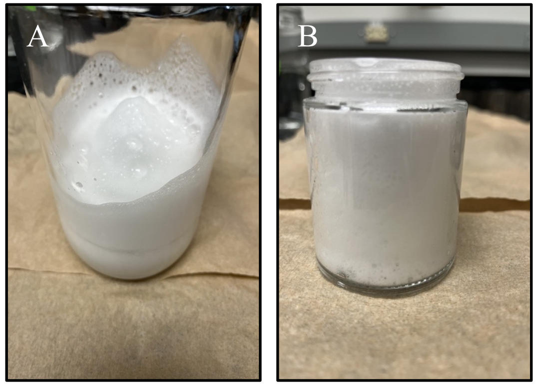
$$\gamma_{a/b} = \gamma_b - \gamma_a - \gamma_i$$

γ_b = surface tension of cyclohexane
γ_a = surface tension of AFFF solution
γi = interfacial tension between the two solutions

Goal: >3 abu

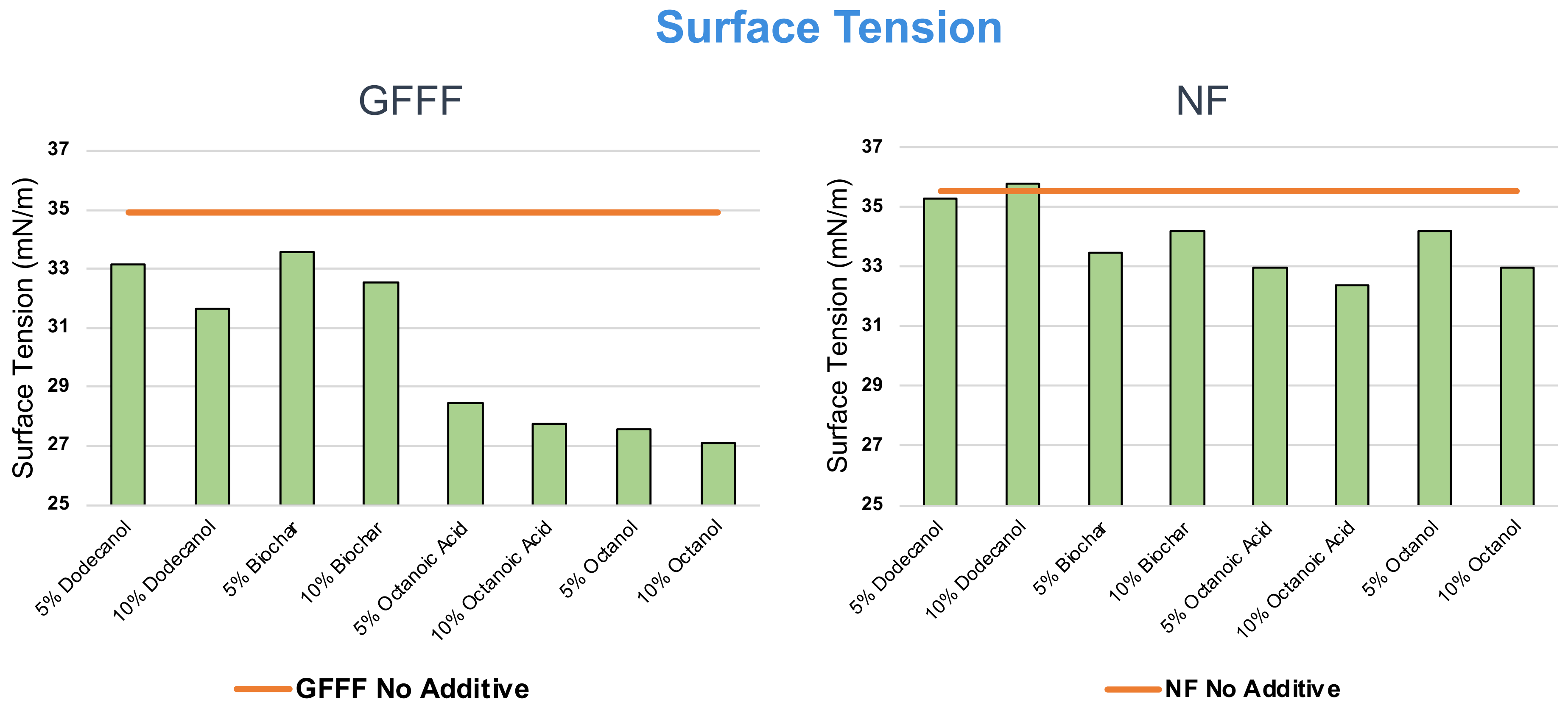
Expansion ratio: *The ratio of volume of foam formed to the volume of solution used to generate the foam*

$$\text{Ex. Ratio} = \frac{V_\rho}{M_2 - M_1}$$



Goal: between 7-10 abu

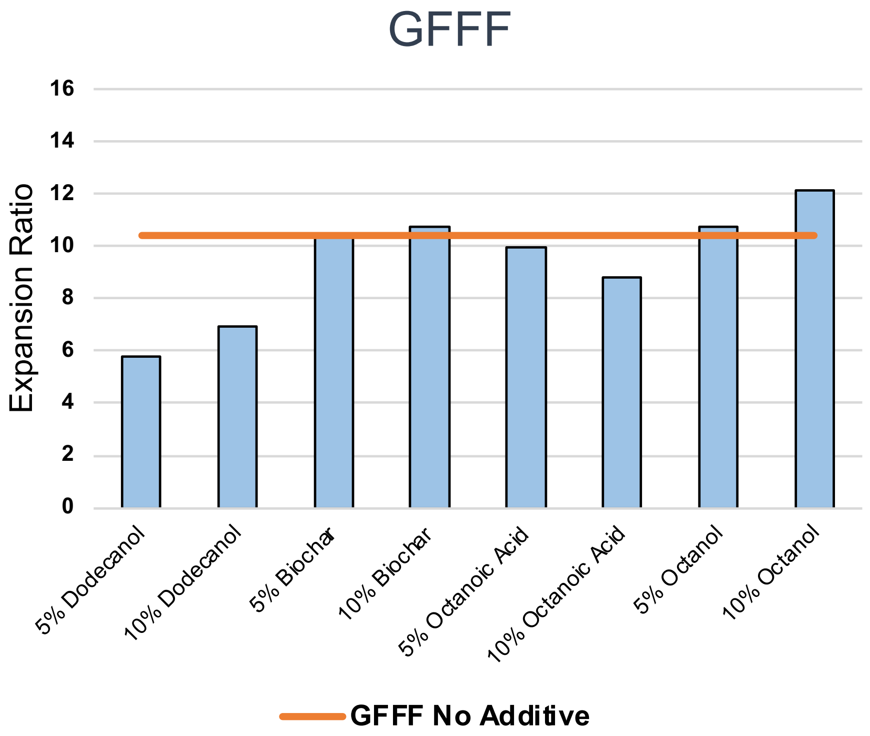
Task 2. Identify and characterize selected additive and foam mixtures

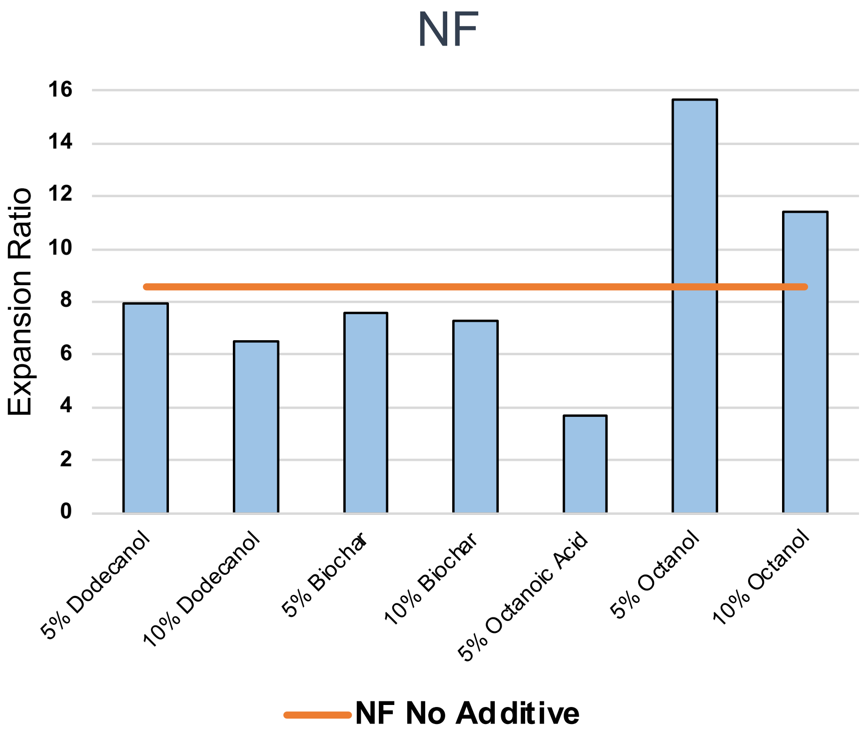


- Additive's influence on surface tension is larger for GFFF
- The same additives yield positive results in both GFFF and NF

Task 2. Identify and characterize selected additive and foam mixtures

Foam Expansion Ratio

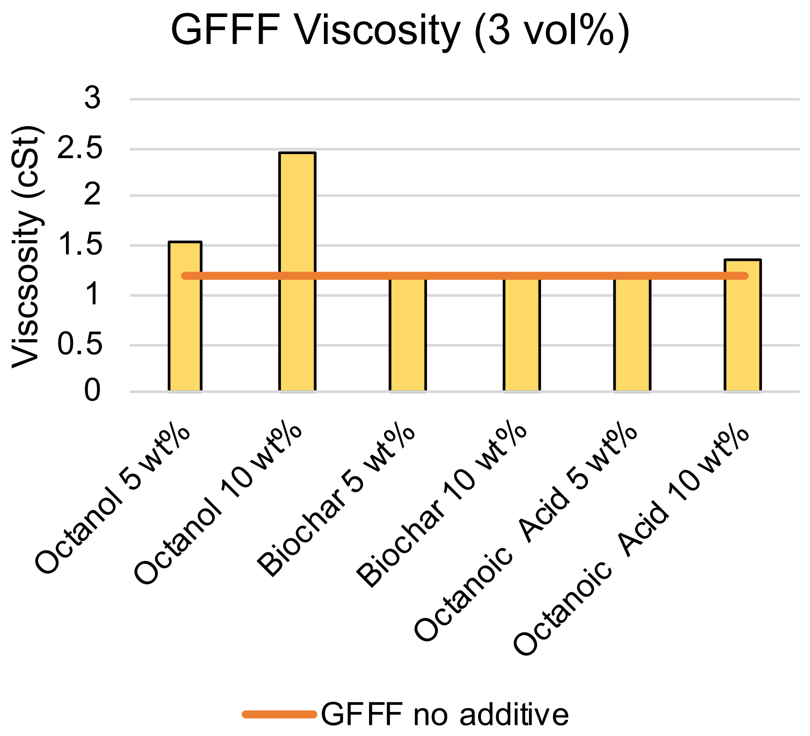


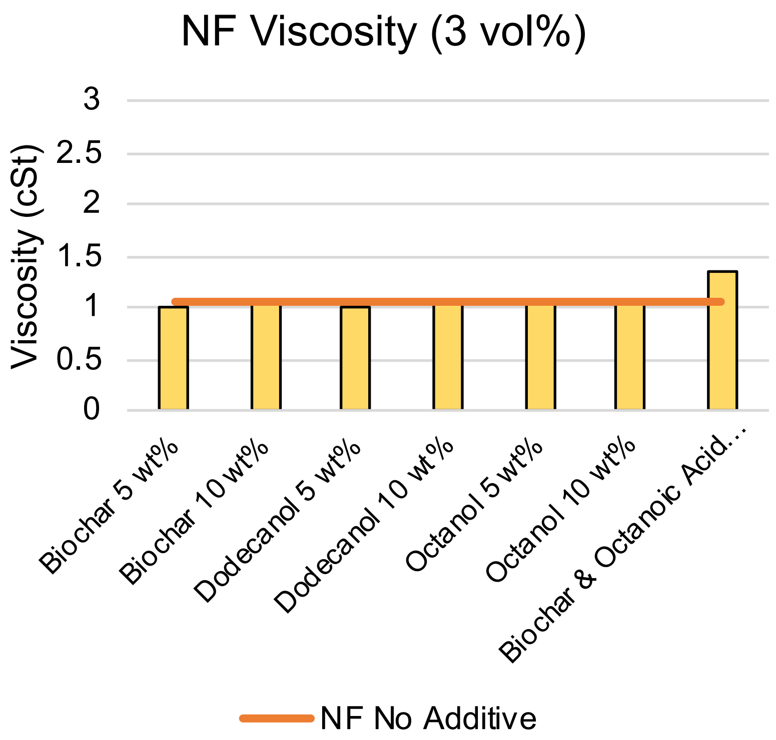


- Foam expansion ratio generally remains within ideal range
- Some additives, for example octanoic acid, severely hinder ability to foam

Task 2. Identify and characterize selected additive and foam mixtures

Viscosity





- Viscosity remained relatively unchanged, except for octanol in GFFF
- Surface tension changes are expected to have the strongest influence on fire suppression performance

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Identify additives for existing PFAS-free fire-fighting foams that can improve their fire-fighting ability and meet the Mil-Spec

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Task 3. Small-scale testing of foam extinguishing performance

Preparation of foam mixture

Pouring foam mixture over fire, measuring time to extinguish

Fire completely extinguished by foam mixture

Task 3. Small-scale testing of foam extinguishing performance

Task 3. Small-scale testing of foam extinguishing performance

GFFF	(seconds)	(seconds)
Mixture:	Time to extinguish:	Burnback time:
GFFF	13.0	107.5
GFFF	10.85	67.2
5% octanol	5.85	73.7
5% octanol	6.9	97.0
10% octanol	7.93	116.5
10% octanol	17.54	130.6
5% biochar, 5% octanol	10.46	89.1
10% biochar, 10% octanol	9.92	94.2

NF	(seconds)	(seconds)
Mixture:	Time to extinguish	Burnback time:
NF 3%	2.72	199.1
NF 3%	3.22	129.4
5% biochar	7.35	199.9
5% biochar	4.03	129.4
10% biochar	5.82	124.6
10% biochar	4.83	199.1
5% biochar, 5% octanoic acid	5.63	206.8

- **Octanol in GFFF, and biochar in NF** influence fire suppression performance
- It can be difficult to differentiate success with small scale testing, development of more consistent system for testing is needed

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Task 4. Fire-fighting performance large scale testing at Jensen-Hughes



28 ft² surface area – industry standard in testing fire extinguishers for liquid-fuel based fires (Class B)

From MIL-24385

Requirement	AFFF solutions, percent		
	1.5% of Type 3 3% of Type 6	3% of Type 3 6% of Type 6	15% of Type 3 30% of Type 6
Foam application time to extinguish (s), maximum	45	30	55
Burnback time of resulting foam cover (s), minimum	300	360	200

Test procedure

- Foam should be generated at 23℃ ± 5 ℃
- Fuel is 10 gallons of heptane
- Fuel is dumped within a 30 second period, ignited within 30 seconds of fueling and allowed to burn freely for 10 seconds
- Fire attacked and extinguished as fast as possible and the fire extinguishing time is recoded at the exact cessation of flame but foam application continues for a total of 90 seconds

Burnback procedure

- Within 60 seconds of foam application, 1 gallon of heptane in a burning pan is placed in the center of the 28 ft² pan and a timer started
- When fire has spread outside of the pan so that burning will continue after pan removal, the pan shall be removed
- Burnback time is the time at which 25% of the total area is in flame

Task 4. Fire-fighting performance large scale testing at Jensen-Hughes

Preparation of foam solution

Ignited fuel in 28 ft^2 pan,
Application of foam-additive mixture

Task 4. Fire-fighting performance large scale testing at Jensen-Hughes

Extinguished fire

Burnback testing

Task 4. Fire-fighting performance large scale testing at Jensen-Hughes

GFFF no additive

GFFF + 5% octanol

Additive improved extinction time by 25 sec, improved burnback by 183 secs

Task 4. Fire-fighting performance large scale testing at Jensen-Hughes

Concentrate	Additive	Extinguishment Time (s)	Burnback Time (s)
GFFF	N/A	95	42
GFFF	5% Octanol	70	183
GFFF	10% Octanol	87	90
GFFF	5% Biochar 5% Octanol	88	46
NF	N/A	49	260
NF	5% Octanoic Acid	Did not extinguish	n/a
NF	5% Biochar	50	270
NF	10% Biochar	43	300
MILSPEC		*30*	*360*
**PFAS-Free MILSPEC*		*60*	*240*

- Best performers: **5% octanol in GFFF, 10% biochar in NF**
- Performance of 10% biochar in NF is approaching MILSPEC
- Promising early research results, a “win”

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Identify additives for existing PFAS-free fire-fighting foams that can improve their fire-fighting ability and meet the Mil-Spec

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Task 5. Evaluate storability and shelf life of highest priority formulations

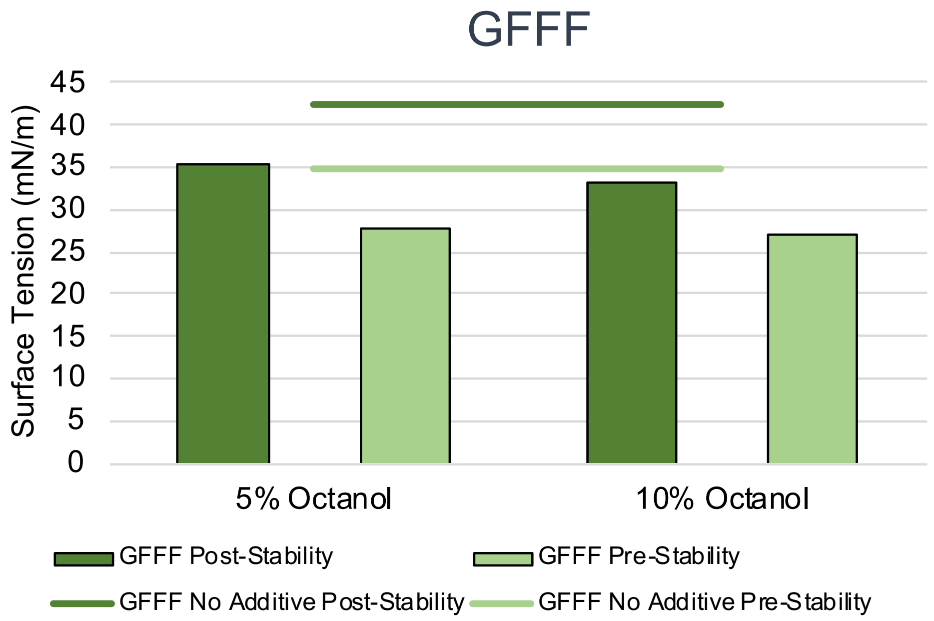
Followed procedure based on the MIL-PRF-24385, with recommendations and collaboration from Jensen Hughes

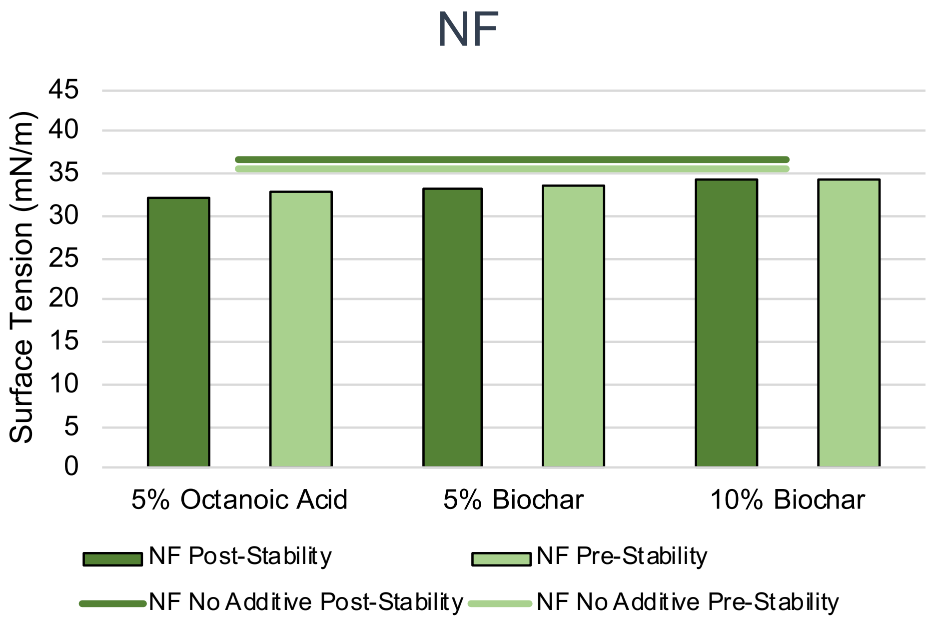
Procedure:

- 1 liter of each mixture shall be placed in lightly stoppered glass cylinders
- The samples shall be stored at 65℃ ± 2 ℃ for 10 days
- The samples will then be tested for the following parameters
  - Foamability
  - Film formation and sealability
  - Fire performance (28 ft^2)
  - Stratification
  - Precipitation

Task 5. Evaluate storability and shelf life of highest priority formulations

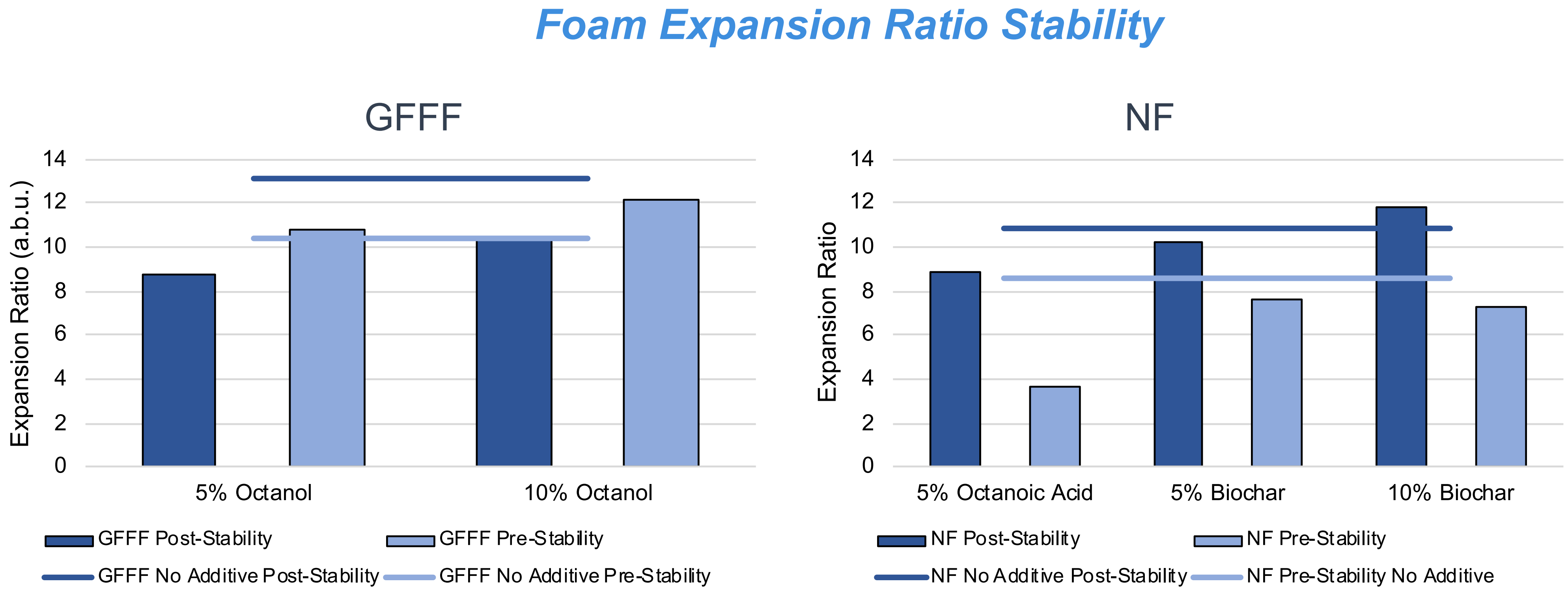
Surface Tension Stability





- In GFFF, aging causes an increase in surface tension
- In NF, aging has very little influence on surface tension

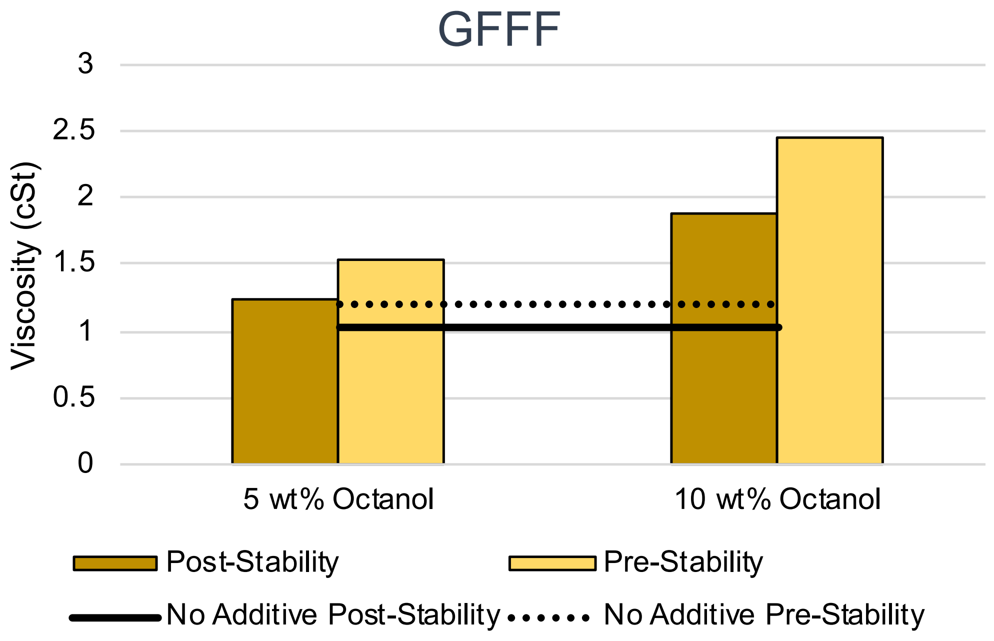
Task 5. Evaluate storability and shelf life of highest priority formulations

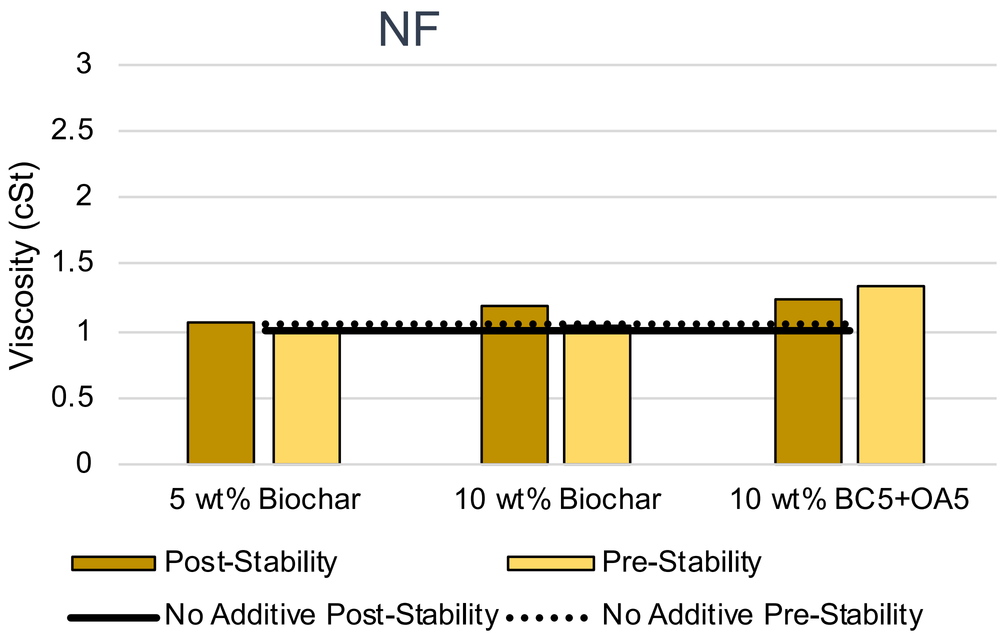


- In GFFF, some loss in foam expansion ratio, but retained within ideal window
- In NF, foam expansion actually increased after aging process – could temperature or longer time for additive-foam interaction help performance?

Task 5. Evaluate storability and shelf life of highest priority formulations

Viscosity Stability

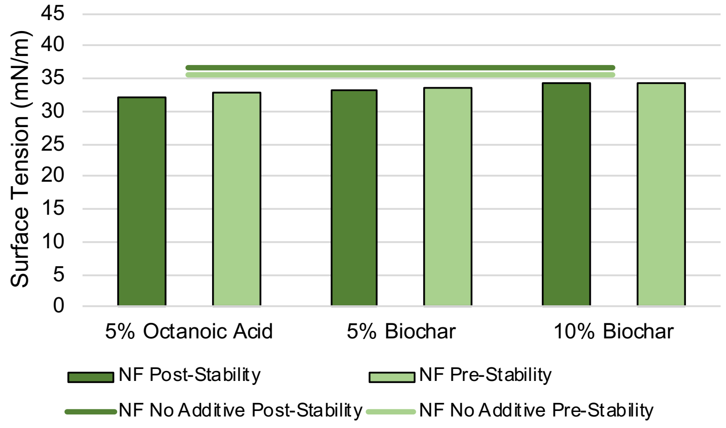


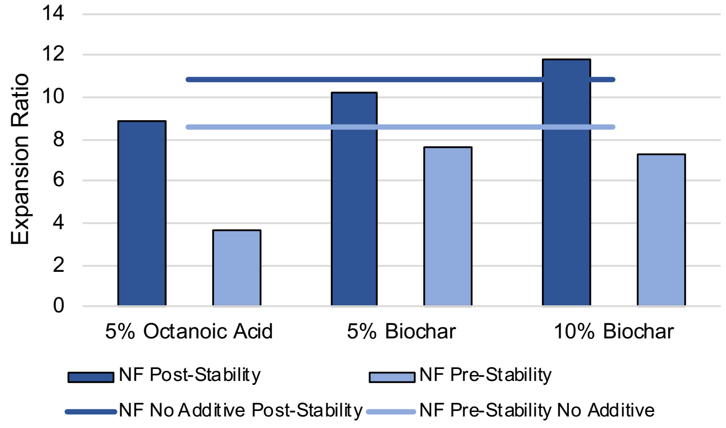


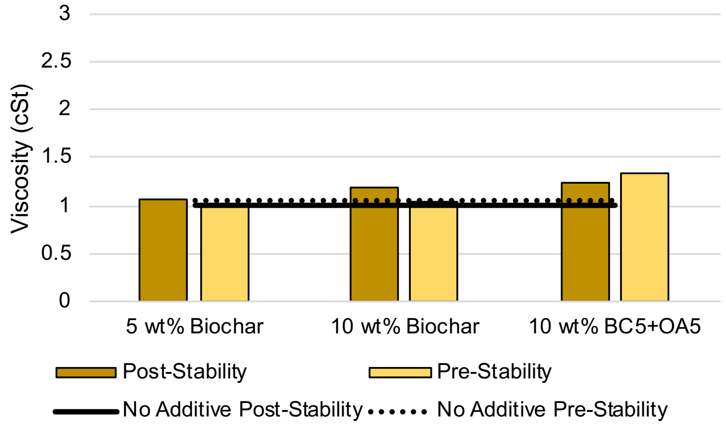
- In GFFF, again causes some loss in viscosity
- In NF, again causes negligible change in viscosity

Task 5. Evaluate storability and shelf life of highest priority formulations

Stability Tests for NF







- NF seems more resilient to long term stability, and addition of additives does not impair stability chemical/physical properties
- Next steps: test fire fighting performance after aging

Summary: Functional Additives to Enhance PFAS-Free Fire Suppressants

Through testing of chemical and physical properties of potential additives in PFAS-free firefighting foams, it was possible to find two potential foam-additive mixtures that improve fire suppression ability

- GFFF with 5% octanol improved time to extinguish by 25 seconds and improved burnback time by 141 second, coming much closer to MIL-SPEC requirements
- NF with 10% biochar improved time to extinguish by 6 seconds and burnback time by 40 seconds, both coming much closer to MIL-SPEC requirements

Both biochar and octanol are more eco-friendly due to their ability to more easily degrade and reduced toxicity to humans and the environment. Additionally, they are readily available and inexpensive as a drop in solution for the urgent needs of the DoD.

Plan to present this work at the 2023 SERDP Symposium

Next challenges:

How can we utilize this preliminary success to inform future PFAS free foam formulations?

What are the relevant use-case scenarios that must be tested for success?

A successful 3-year project would provide a PFAS free formulation that meets the MIL-SPEC under various use-case scenarios, leveraging COTS materials with custom additives

Challenge A

Molecular scale testing and modeling of foam-additive mixtures

- Analytical techniques for evaluation of foam-additive chemical interactions to inform mixing ratios and additive modifications
- *Option:* Develop a computational approach to model interactions and help identify additive modifications

Challenge B

Evaluation of PFAS-free foam use-case scenarios influence on fire suppression

- Evaluation of the following use case scenarios: SEAWATER, FUEL MIXES, AND TEMPERATURE

Challenge C

Evaluation of potential for scaled-up manufacture and distribution

- Storability and compatibility testing for foam-additive mixtures utilizing accelerated aging
- *Option:* Partner with PFAS-free foam manufacturer to develop best forming formulation and analyze ability to scale-up and distribute

At all points of this process testing will be conducted for physical and chemical properties and fire suppression performance

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Next steps: challenges to address

- What is the molecular basis for the chemical interactions between additives and the PFAS-free foams that influence their fire suppression ability? Through this challenge we will:
  - Utilize JHU/APL expertise in molecular scale analytical techniques (NMR, FTIR, and HRMS) to identify the chemical interactions that occur between additives and PFAS-free foams in varied mixing ratios and develop a model of the interactions
  - *Option:* Analytical determination of chemical interactions will be utilized with JHU/APL expertise in molecular scale property metadynamics simulations to develop a model to explain the behavior of additives in varied PFAS-free foams
  - The combined experimental and computational approach will provide the ability to identify proper mixing ratios of additives in a wider variety of PFAS-free foams and will identify additional analogues of high performing additives determined in the Limited Scope Portion of the work. For example, studies have shown that the starting material for the pyrolysis process can significantly alter the molecular characteristics of the biochar material, resulting in a wide range of functional groups and chemical make-up.[53–55] Classes of these different 'biochar' materials may further improve fire suppression and should be reviewed as potential candidates for further testing.

Next steps: challenges to address

- How do varied use case scenarios influence properties and fire suppression ability?
  - Down-selected, high performing additive-foam mixtures must be evaluated for their performance in relevant use case scenarios, listed here:
    - SEAWATER: The DoD has not ruled out use of PFAS on board ships due to concerns over using seawater as the water source for creating the foam with PFAS-free firefighting foams. Our additive-foam mixtures will be tested with relevant seawater concentrations of salt (and with acquired seawater itself) to determine if there is a change in fire suppression performance.
    - FUEL MIXTURES: Firefighting foams may be used to put out fires resulting from a variety of fuels or fuel mixtures. Testing conducted in the Limited Scope effort focused on heptane for small-scale tests and gasoline for large-scale tests. Additional testing is required with a variety of fuels and fuel mixtures in order to determine how fire suppression may change with varied additive-foam mixtures as a result of different fuel sources (alcohol, ether, gasoline, etc.).
    - TEMPERATURE: Additive-foam mixture properties are likely to vary significantly depending on external temperatures at the point of use. Our highest performing additive-foam mixtures must be tested at varied temperature conditions to determine if there are changes to fire-suppression performance.

Next steps: challenges to address

- How can novel additive-foam mixtures be manufactured and distributed at scale?
  - The best performing foam-additive mixtures identified from property testing and fire suppression testing should be considered for manufacture to address the urgent need for drop-in AFFF replacements.
    - Conduct storability and compatibility testing with accelerated aging experiments followed by property and fire suppression testing to determine how long term storage of additive-foam mixtures may influence stability of the product.
    - *Option*: Partner with a PFAS-free foam manufacturer whose product we have done testing with and work with them to supply a new formulation based on our test results. The new formulation will be tested as previously described for chemical and physical properties and fire suppression ability. The PFAS-free foam manufacture, with vast expertise on producing a product at scale, will help JHU/APL to conduct an analysis of materials cost, manufacture cost and ability, and determine ease of producing this new product for DoD use.

Project Overview

- Total Funding: 250k (seedling)
- Start Date: 29-April-2022 (actual technical kickoff was end of June 2022)
- End Date: 28-April-2023

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Aqueous Film Forming Foam (AFFF)

AFFF are water-based fire suppressing mixtures typically containing a **hydrocarbon-based surfactant** (sodium alkyl sulfate) and a **fluorosurfactant** (PFOA, PFOS)

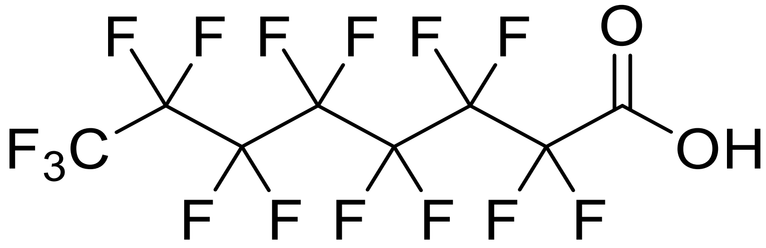
Class B foams – used to extinguish liquid-fuel based fires, such as gasoline, oil, and jet-fuel (used most commonly in DoD installations)

Foam blankets the fuel surface, smothers the fire, and isolates the fuel source from oxygen

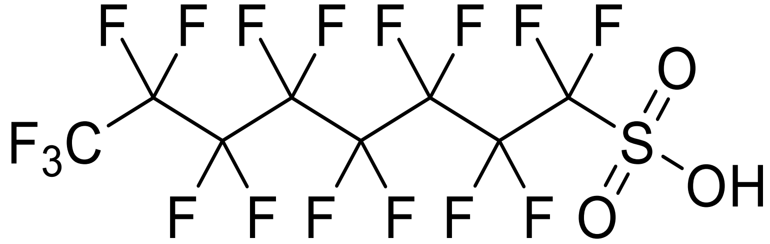
Water content cools the fuel, suppressing release of flammable vapors

Concentrate that is mixed into either 3% or 6% solution with water

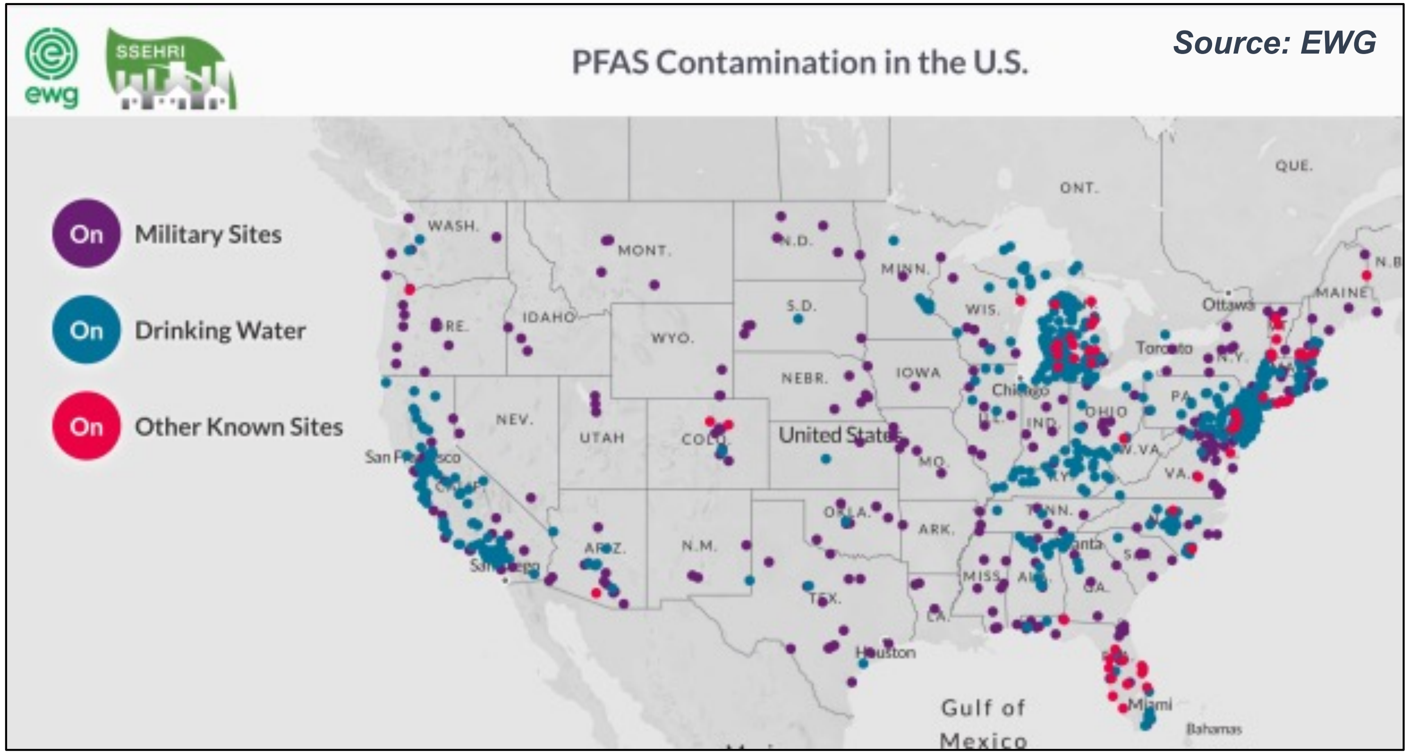
Perfluoroalkyl substances (PFAS) in AFFF



Perfluorooctanoic acid (PFOA)



Perfluorooctanesulfonic acid (PFOS)



- High thermal and chemical stability, highly **oleophobic** – promotes continuous film formation over the liquid-fuel and enhances stability once foam is formed; essential to fire fighting efforts

- PFAS have now been identified as extremely toxic – including association with developmental issues, cancers, liver damage, etc.

AFFF "Green" Alternatives

- *The US Military and DoD have announced plans to stop using PFAS containing AFFF by October 2024*

- **No** PFAS free foams currently meet the latest requirements for military use specified in MIL-PRF-24385
  - PFAS free foams are unstable at high temperature and in the presence of oil vapors due to lack of C-F bond

- Alternatives typically mimic oleophobicity and high temperature stability of the compounds
  - What are some additives that could help improve these properties?

JOHNS HOPKINS

APPLIED PHYSICS LABORATORY