Extended life filter for chemical biological radiological and nuclear (CBRN) protection

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BLUF: Collective protection (COLPRO) filters are used to protect spaces such as buildings from CBRN threats. Many of these COLPRO systems operate full time, causing the filters to lose their chemical capacity due to the degrading effects of continual air flow. Additionally, the cost and man hours required to replace COLPRO filters places a significant burden on the operation of COLPRO systems. This effort is intended to develop and prototype a new extended life media and filter bed for incorporation into COLPRO filters. This should in turn decrease the frequency of filter replacement and reduce the associated life cycle cost.

ABSTRACT

Broad spectrum filtration is of critical importance for protection from toxic chemicals. This application requires materials with both high porosity for physical sorption and metal sites for chemical reactions. While these challenges have been well met, aging due to exposure to humidity or contaminants limit the fieldable lifespan. The objective of this effort is to develop improved filtration media and to incorporate the media into scaled filter designs that maximize service life.

A novel process is used to make beaded granules of blended powder materials. The powders include carbons with different porosities, precipitated metal oxides, and zeolites. In addition to the base materials, co-precipitated media with metals deposited onto the carbon powder can be made and then built into beaded granules. Beaded granules with different ratios of powders are made and then loaded with triethylenediamine (TEDA). Physical properties of the filtration media are evaluated to determine porosity, density, particle size distribution, and ball pan hardness.

The beaded granules are then subjected to accelerated aging. Humid aging was performed by delivering air at 80 °C and 80% RH to media at flow rates representative of collective protection filters. Under these conditions, 5 simulated years of aging can be achieved in 2 weeks. To simulate contaminant exposure, discrete loadings of a simulated fuel mixture was loaded onto the beaded granules. Following aging, materials were tested for filtration performance against cyanogen chloride (CK), dimethyl methylphosphonate (DMMP), CH_3Br, and H_2S, with results compared to those of the baseline material.

A co-precipitated Zn/Carbon beaded granule was prepared and evaluated for filtration performance prior to and following accelerated aging. For all blended media with carbon, TEDA retention and utilization is good as measured by CH_3Br breakthrough. However, CK performance is greatly reduced for blended beaded granules of carbon and metal oxides. This is due to separation of TEDA in the carbon pores and the reactive metal sites needed to keep the TEDA from being poisoned. When co-precipitated Zn/Carbons are employed, CK performance remains essentially unaltered after aging. This is due to stabilization of active metals sites within the carbon pores near the retained TEDA. In addition to CK, there is no impact of either fuel or humid aging on the AC performance of Zn/Carbon co-precipitated materials. The durability of the beaded granules versus AC is attributed to the stabilization of the metal phase. In addition to Zn, a promising Zn-Fe-Si (ZFS) metal oxide was also successfully deposited on carbon.

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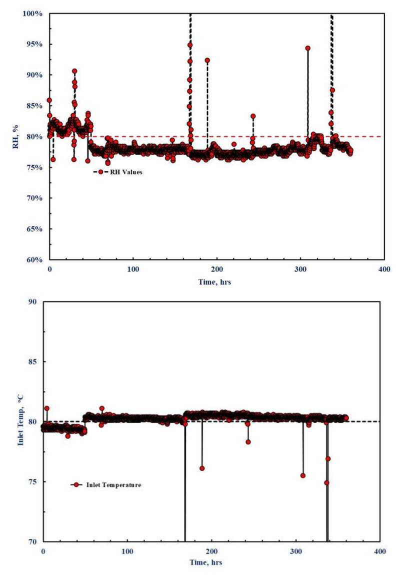
MEDIA PREPARATION

A novel method to blend powder material into beaded granules has been developed to create improved media. Powders of carbon, zeolite, and precipitated metal oxides can be mixed to create a single material with adsorptive properties that can be tailored by varying the ratio of the powders used. The resulting materials are formed into 12x30 mesh beads via spheronization which enhances particle density without significant impacts on porosity. The media created by this process also displays the appropriate ball pan hardness required for use in COLPRO filters. In addition, co-precipitated media have been developed in which metal oxides are precipitated in the presence of microporous powders. These powders can then also be blended with others in the beaded granules process.

ACCELERATED AGING

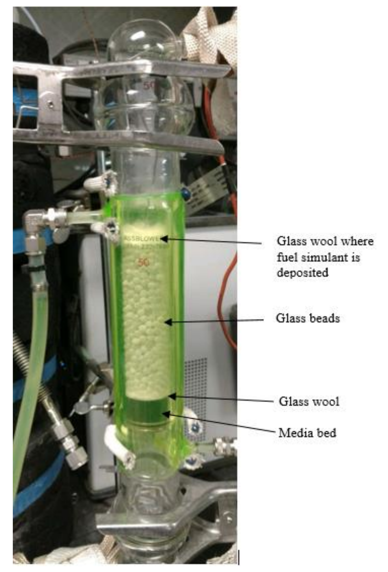
Humidity



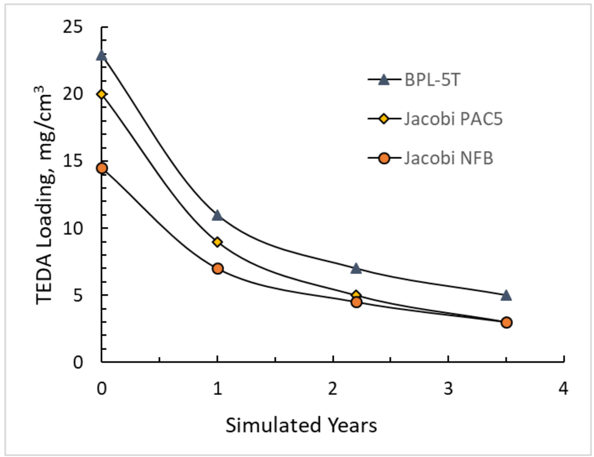
Accelerated Aging Temperature	Weeks to Achieve 5 Simulated Years
40°C	75
45°C	53
50°C	37
55°C	26
60°C	19
65°C	13
70°C	9.3
75°C	6.6
80°C	4.7

Fuel Contaminants

A blend comprised of 60% decane, 20% diethylbenzene, 15% dodecane and 5% naphthalene by weight was used to simulate diesel fuel. The exposure involved placing a plug of glass wool with a discrete amount of fuel simulant above a sample Air is passed through the system overnight (16 hours) to vaporize the fuel simulant and adsorb it within the pores.



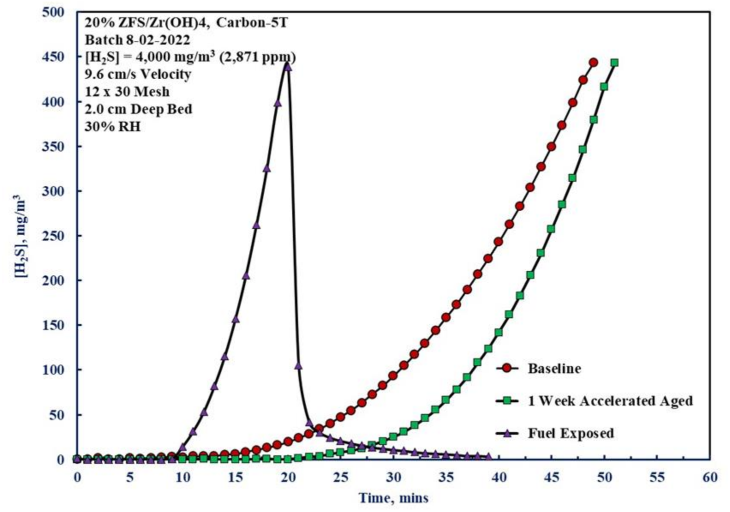
TEDA EXTRACTION



A solvent washing method was used to assess the ability of different carbons to retain TEDA after accelerated aging. All carbons lose significant amounts of TEDA, with higher density carbons being able to retain TEDA the best. ZSM-5 was also tested and showed to retain TEDA strongly, however, much of the TEDA in zeolite pores is inactive based on CH_3Br testing.

Material	Density g/cm³	mg TEDA/cm³ As-Prepared	mg TEDA/cm³ Weathered
BPL-5T	0.54	22.9	5
Jacobi NFB-5T	0.30	14.5	3
Jacobi PAC5-5T	0.43	20.1	3

CHEMICAL FILTRATION TETSTING



Breakthrough testing was performed using DMMP, CH_3Br, H_2S, and CK on 2 cm beds on baseline, humid aged, and fuel exposed samples.

DMMP: Measures physical adsorptive capacity

CH_3Br: Reacts 1:1 with TEDA to measure TEDA retention/reactivity

H_2S: Acid gas that reacts with metal phase to measure reactive capacity

CK: Reacts with TEDA to form HCl. HCl either poisons TEDA or can react with metal phase. Measures ability of media to retain TEDA near active metal sites.

RESULTS

Material	DMMP	H_2S	CK	CH_3Br*
25% Zn/Carbon	158	13		79%
humid aged		17		44%
fuel exposed	80	8		
50% Zn/Carbon	154	28	8	89%
humid aged		32	10	84%
fuel exposed	63	9	4	
70% Zn/Carbon	125	15	12	85%
humid aged		17	9	50%
fuel exposed	35	3	4	
25% MFI/Carbon	58	13		59%
humid aged		4		12%
fuel exposed	55	12		
ZFS/Carbon		26	24	102%
humid aged		28	18	40%
fuel exposed				
ZFS/Carbon/MFI		36	16	
humid aged				
fuel exposed		28	6	
ZFS/Zr(OH)4, carbon	125	18		93%
humid aged				
fuel exposed	49	11		47%

Several interesting trends show up in the data:

1. Varying amounts of Zn on carbon showed good activity towards DMMP, H_2S, and CK after humid and fuel exposure. This is likely due to the stabilization of the metal phase in the co-precipitation process. Higher levels of Zn allow enhanced acid gas reactivity, but too much can block pores. All Zn/Carbon are negatively impacted by fuel exposure.
2. MFI/Carbon shows good resistance to fuel but does not resist humid aging.
3. A Zn-Fe-Si (ZFS) co-precipitated carbon showed enhanced CK removal when compared to just Zn.
4. A tri-blend material, ZFS/carbon/MFI shows excellent retention of acid gas reactive capacity after exposure to fuel. This material has been identified as a potential media for scale up and testing in mixed or layered bed configurations.
5. Continued efforts for novel media development include ZFS/$Zr(OH)_4$ and Zn-Zr/Carbon.

LAYERED BED CONFIGURATIONS

Layered bed configurations are currently being explored as an option to enhance filter lifetime. As fuel vapors are the primary airborne contaminant, materials that resist degradation after fuel exposure will be tested in tandem with current filter media. Two strategies to explore in layered bed filter design are the volume ratio of media and inlet vs outlet configuration. As promising materials are discovered, new layered beds will be tested to provide optimized protection for fresh and aged beds.

CONCLUSIONS/FUTURE WORK

Novel media have been developed to provide better filter performance after exposure to humid aging and fuel contaminants. A new co-precipitation and blending method allow for a high degree of tailoring and optimization. Best results have been with a Zinc-Iron-Silica metal oxide layer deposited onto highly microporous carbon. The new media has been tested against reactive gases and maintains performance after aging.

- Continue development and optimization of blended media
- Incorporate new media into layered filter bed designs
- Explore regeneration of fuel dosed media
- Down select material and bed design for prototype filter fabrication

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