Development of novel broad-spectrum sorbent to extend the lifetime of collective protection filters

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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 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 to maximize service life of collective protection filters.

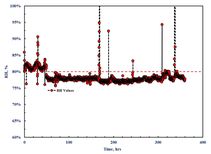
A novel process is used to co-precipitate metal-oxides onto blended amounts of carbon and $Zr(OH)_4$ powders. A binder is used to make beaded granules and then loaded with triethylenediamine (TEDA). Physical properties such as porosity, density, and hardness are similar to traditional granular filtration media (gfm).

The beaded granules are then subjected to accelerated aging involving humidity or battlefield contaminants (fuel vapors, SO_x/NO_x). Following aging, materials were tested for filtration performance against, dimethyl methylphosphonate (DMMP), hydrogen cyanide (AC), and cyanogen chloride (CK).

A Zn-Fe-Si (ZFS) precipitated metal oxide material with a 70:30 mix of carbon:$Zr(OH)_4$ proved optimal. Production of this media advanced to pilot scale manufacturing to produce 200 lb batches. Using this media, different degrees of accelerated aging were tested to develop aging profiles. This media was also put into guard bed style filters that fit into the M98 plenum and deployed to get realistic field aging.

ACCELERATED AGING

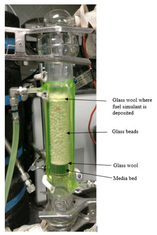
Humidity



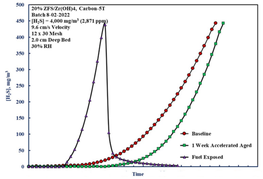
Accelerated Aging Temperature	Weeks to Achieve 5 Simulated Years
60°C	19
65°C	13
70°C	9.3
75°C	6.6
80°C	4.7

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.



CHEMICAL FILTRATION TETSTING

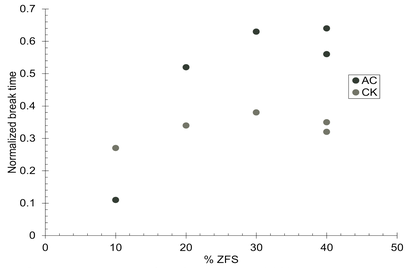


Breakthrough testing was performed using DMMP, AC, and CK on 2 cm beds on baseline, humid aged, and fuel exposed samples.

DMMP: Measures physical adsorptive capacity
AC: Acid gas that reacts with metal phase to measure reactive capacity
CK: Reacts with TEDA to form HCl. HCl either poisons TEDA or reacts with metal phase. Measures the ability of media to retain TEDA near active metal sites.

RESULTS

Material	DMMP	AC	CK
10% ZFS/Carbon, Zr(OH)4 70-30	1.00	0.39	0.72
humid aged		0.30	0.63
fuel exposed	0.21	0.11	0.27
20% ZFS/Carbon, Zr(OH)4 70-30	0.94	0.73	0.85
humid aged		0.52	0.60
fuel exposed	0.16	0.56	0.34
30% ZFS/Carbon, Zr(OH)4 70-30	0.94	0.79	0.99
humid aged		0.69	0.86
fuel exposed	0.21	0.63	0.38
40% ZFS/Carbon, Zr(OH)4 70-30	0.86	0.77	0.95
humid aged		0.76	0.75
fuel exposed	0.18	0.56	0.32
40% ZFS/Carbon, Zr(OH)4 60-40	0.80	1.00	1.00
humid aged		0.70	0.75
fuel exposed	0.21	0.64	0.35
40% ZFS/Carbon-5T	0.49	0.74	0.58
humid aged		0.60	0.69
fuel exposed	0.10	0.25	0.16



- Optimum loading of ZFS is around 30%
- Results similar for broad range of formulations so there is wiggle room for manufacturing variation.
- Due to higher cost of $Zr(OH)_4$, the 70:30 formulation was selected for scale up.

PILOT SCALE-UP

500-gallon reactor tank

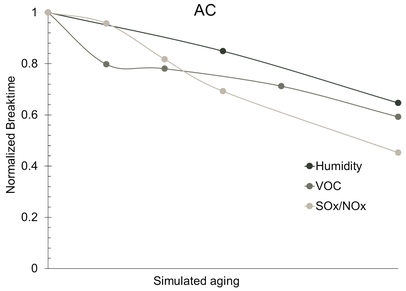
Filter press for washing

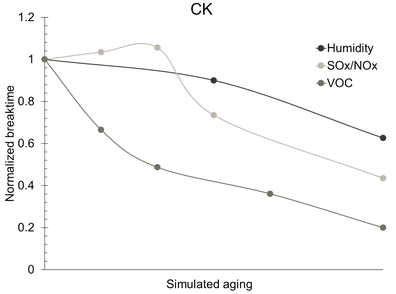
V-blender for TEDA sublimation

- Successfully produced two 100 lb batches and two 200 lb batches of media
- Physical characteristics and filtration performance of pilot-scale media is similar to bench top scale.

AGING STUDIES

With a greater quantity of media from pilot batches, simulated aging curves were developed for humid aging, VOC, and SO_x/NO_x exposure.





- Both AC and CK curves show little media degradation due to humid aging revealing that the metal phase is stable
- SO_x/NO_x has a greater impact than humidity due to reaction with the metals.
- VOC has a greater impact for CK due to pore blockage preventing access to TEDA.

Guard bed style filters that fit in the plenum of M98s have been made with the new media and deployed to get real field aging data.

CONCLUSIONS/FUTURE WORK

- A new media has been produced with better performance after simulated collective protrection aging environments
- The media was successfully scaled-up to pilot scale production levels
- Combination of media cost and longer filter life could provide significant life cycle cost savings

- Media is currently deployed in the field to obtain realistic aging data
- Broader chemical testing are ongoing to optimize media formulation

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