INTRODUCTION

Filtration is utilized in many applications in the polymer and chemical industries. Selection of a specific filter medium depends on application and process conditions. For those applications where metal filters are used, cleaning provides a way of reducing costs by reusing filters. When carbon is the contaminant, special cleaning problems may be encountered. Removing carbon from filter media can be accomplished to varying degrees by physical and/or chemical means. Depending on the complexity of the media and the degree of carbon removal necessary to satisfy quality requirements, either of the methods can provide effective carbon removal. However, as the degree of carbon removal becomes more critical and the structure of filter media becomes more complex, *effective* implies nearly 100% carbon removal from media composed of an intricate array of fine fibers. A process designed for complete carbon removal has the potential for damage to small fibers such as those used in fiber metal felt media. As a result, the aggressiveness of the cleaning process must be carefully controlled and monitored to effectively remove carbon while minimizing the potential for damage. This paper explores and compares the theories of several cleaning methods designed to remove carbon and their possible effects on the filter media.

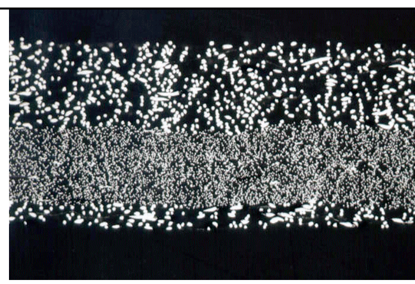


Figure 1: **Cross-sectional view of 5 µm sample of sintered fiber felt depth media.**

FILTER SELECTION AND CLEANING

When choosing a filter for a polymer or chemical application, a decision must be made to clean and reuse or to discard the contaminated filter. If the filter is to be cleaned, it must not only withstand process line conditions but also the cleaning process. It is important that persons involved with filter selection understand cleaning requirements for the contaminant and media prior to selecting the filter.

In ***Figure 1,*** a cross-sectional view of sintered fiber felt depth media shows that the fiber diameters on the upstream side (top of picture) are larger than that on the downstream side. When the downstream fiber matrix becomes plugged with contaminant, it is unlikely that physical methods will provide effective cleaning - especially if the media is tightly pleated. If a suitable chemistry can be developed to dissolve or react with the contaminant, then flushing processes can be very effective in removing contaminant-laden solutions from surface and depth media. If, however, insoluble particulate remains trapped within the matrix, the method of removal must be determined by the composition of the residuals. Other, and possibly more aggressive, cleaning methods must be used for removal. If carbon, carbon containing scale, or degraded polymer remains in the media after normal cleaning, it may not be possible to chemically remove the contaminant from the media by the use of solvents or aqueous solutions. However, there are cleaning methods that either physically remove the carbon from the media or chemically oxidize it to a form that can be removed.

Physical methods include bumping, flushing, and ultrasonics. Depending on the type of media being cleaned, physical methods may be very successful. Physical methods may provide adequate carbon removal for 80µm wire mesh surface filter but not for 5µm fiber felt depth media. Although physical methods can be used, 100% carbon removal cannot be expected, especially in graded depth media. By relying on the chemistry involved with high temperature oxidation processes in combination with physical removal methods, an effective cleaning process can be developed. Since there is potential for damage in any high temperature oxidation process, it is important to understand those processes.

HTO

A high temperature oxidation (HTO) process is defined as a process that subjects contaminants (reactant) to temperatures which will provide the activation energy necessary to cause the reactant to alter its oxidation state. The activation energy and the resulting product will be dependent on the HTO process used and whether other chemicals such as oxidizing agents or catalysts are present. If the reaction involves carbonaceous residuals, once activated, the oxidation reaction is exothermic.

The amount of energy released is stoichiometrically related to the amount of carbon being oxidized. HTO processes can be designed so that safe operating temperatures for the metal substrate can be preset. However, temperature spikes during oxidation of the carbon may exceed 1000^o F. These high temperatures and the time at which the metal is exposed to these temperatures can provide the potential for problems such as sensitization which will result in broken sintered bonds and media loss. The length of time that the metal is subjected to elevated temperatures and the location of the oxidation will be dependent on the HTO process.

Two HTO processes that can be very effective in removing carbon from fiber metal media involve ovens and molten salt baths. Both processes are similar in that each supplies the energy needed to initiate the reaction; however, there are major differences between the methods. To determine which process is best suited for a particular situation, the mechanics of each process and the pre-cleaning and post-cleaning requirements for the filter must be considered.

EFFECT OF HTO PROCESSES ON FIBER METAL MEDIA

STUDY 1 & 2: OBJECTIVE

To determine whether oven and/or molten salt bath processes have detrimental effects on the metal substrate, two studies were performed:

Study 1: Uncontaminated fiber felt media coupons of different micron ratings were processed using different methods and through numerous cycles. After processing, magnified photographs were taken of the processed media and new media. The photographs were compared for evidence of differences in scale characteristics and mechanical damage to the fibers.

Study 2: Historical test data for filters from different applications, manufacturers, configurations, micron ratings, and cleaning processes were examined to determine whether HTO processes shorten the useful life of the filters.

STUDY 1: *Effect of cleaning processes on uncontaminated metal media as indicated by scale formation and mechanical damage to fiber matrix.*

Study 1: Experimental Design

Bekaert AL3-Series samples were cut into twenty-one (21) coupons and processed as described in ***Table 1***. The salt was a formulation of nitrates and nitrites.

Each coupon type (micron rating) was processed by the given method up to 4 cycles.

Coupons were labeled as

1. Micron rating (5, 10, 20, or 30)
2. Process Method (A, B, C, D, E, or F)
3. # of Processing Cycles (0, 1, 2, 3, or 4)

Observations and notes were made as to visual appearance and color of the media. Coupons were sent to the Bekaert facility in Belgium for photographs showing magnified sections of each sample.

Table 1: **# coupons of each micron rating & process description**

Groups	Number of Coupons	Process
Method A	1 @ 5µm 1 @ 10µm 1 @ 20µm 1 @ 30µm	Control Group (No process)
Method B	4 @ 5µm 4 @ 10µm 4 @ 20µm 4 @ 30µm	Scale Removal Chemicals only
Method C	4 @ 5µm 4 @ 10µm 4 @ 20µm 4 @ 30µm	Salt Bath Scale Removal
Method D	4 @ 5µm 4 @ 10µm 4 @ 20µm 4 @ 30µm	Salt Bath No Scale Removal
Method E	4 @ 5µm 4 @ 10µm 4 @ 20µm 4 @ 30µm	Oven Scale Removal
Method F	4 @ 5µm 4 @ 10µm 4 @ 20µm 4 @ 30µm	Oven No Scale Removal

Study 1: Results

After processing coupons in either the oven or salt bath, the metal lost its luster due to the formation of scale. Oven scale tended to be dark with bluing, whereas, scale formed in the salt bath was a "golden" color (See ***Figures 2*** and ***4***). Chemical formulations designed to remove the scale formed during the oven/salt bath runs were used on samples as indicated in ***Table 1***.

The figures show photographs of media samples of various micron ratings that have been through four (4) oven and salt process cycles.

(On black & white copies, the color variations cannot be seen)

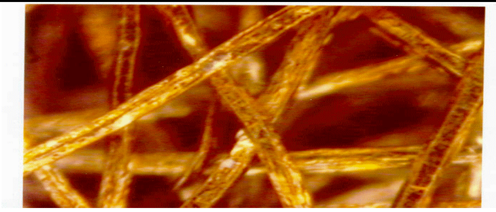


Figure 2: **30µm media sample cleaned 4 times in oven. No post-cleaning.**

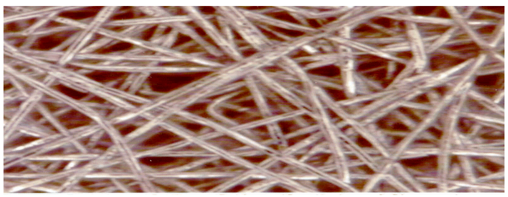


Figure 3: **20µm media cleaned 4 times in oven. Post-cleaning after each run.**

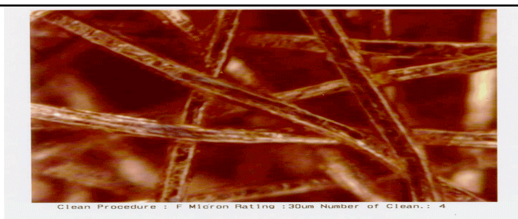


Figure 4: **30 µm media sample cleaned 4 times in molten salt bath. No post-cleaning.**

Figure 5: **5µm media sample cleaned 4 times in molten salt bath. Post-cleaning after each run**

Figure 6 shows an example of the appearance of mechanical damage evidenced by broken fibers. This type of damage would present itself by media migration or shedding.

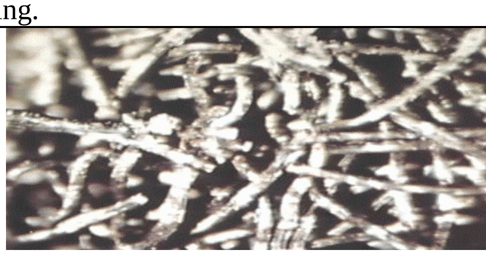


Figure 6: **Damaged media**

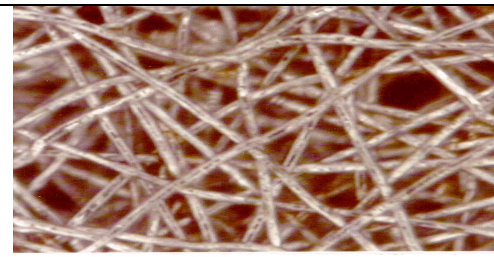


Figure 7**: 5µm media sample with no cleanings**

In ***Study 1***, the oven scale required a more aggressive post-cleaning scale removal process than the salt bath due to the formation of higher oxide scales. If the media had been contaminated, there would have been scale formed as a result of the "baking-on" of inorganic components in the contaminant. However, whether there was post-cleaning scale removal or not, the fiber matrix integrity appears unchanged and similar to that of the control in ***Figure 7.***

STUDY 2: ***Effect of cleaning processes on contaminated metal substrate as indicated by test data trends.***

Study 2: Experimental Design

In this study, historical data consisting of Bubble Point and Flow Test pressures were used to determine whether the cleaning process, specifically HTO processes, shortened the useful life of the filters. Bubble Point is the pressure required to force a stream of bubbles through the largest pore in the media when the filter is submerged under a specific depth of testing fluid. Bubble point pressure can be correlated to the largest pore size in the test sample. In the Flow Test, the submerged filter is challenged with a set air flow and the resulting pressure drop recorded. The Flow Test gives a quantitative (pressure) and a qualitative (flow pattern) measure of the overall integrity of the filter.

Since polymer/chemical process line conditions, catalyst & additive interactions, process line upsets, etc. will also affect useful filter life and may be characteristic of certain polymers or filters, the study was designed to minimize errors associated with a particular filter or product.

Table 2 shows parameters and conditions that were followed when selecting the filters for the study.

Average Bubble Point and Flow Test data, compiled over 3+ years for filters in ***Table 3***, were plotted as a function of the number of cleaning cycles (***Graphs 1 & 2***).

Table 2: **Considerations when choosing filters for *Study 2***		
Parameter	**Description**	**Condition**
Applications	PET, PA, etc. - Additives	3 different applications
Pre-Cleaning	Gross polymer removal	Solvent based, non-aggressive methods
Filter Design	Candle, disc, etc.	Candles and discs were used
Manufacturer	Different manufacturers	3 different manufacturers
Media Type	Fiber Metal	Variations in degree of sintering
Micron Rating	Pore size	Pore size ≤ 40µm

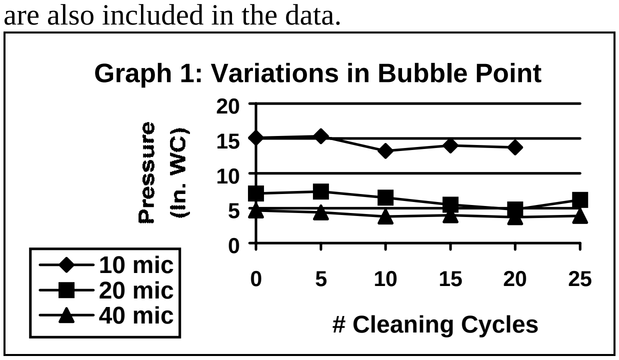
Table 3: **Filter design and micron rating used for evaluation**			
Filter Design	Disc	Candle	Candle
Micron Rating	10µm	20µm	40µm

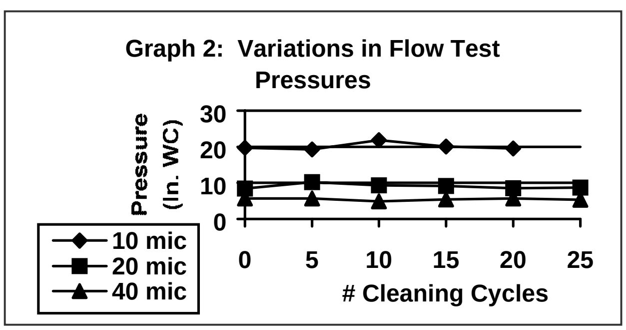
Note: *The air flow used varied with the particular filter being tested.*

Study 2: Results

The following graphs show variations in Bubble Point and Flow Test pressures for filters that have HTO steps in the cleaning process. The graphs represent the mean of all data points in the database for the given number of cleaning cycles.

Test values below acceptable minimum pressures are also included in the data.





STUDY 1 & 2: CONCLUSION

Since the useful life of a filter will be affected by any environment to which it is exposed - *process* or *cleaning*, it is necessary to identify the effects that various cleaning methods may have on the filter. The studies described in this paper were designed to:

- identify potential problems with HTO processes designed to remove carbon, and
- evaluate historical data to determine if HTO processes have detrimental effects on useful filter life.

Processing clean media coupons with methods that include chemical cleaning in aqueous solutions and in those processes that involve temperatures up to 800°F did not show any signs of mechanical damage as evidenced by fiber breakage. The tests did, however, demonstrate a difference in the type of scale formed in ovens and salt baths. The differences were apparent both visually and in the post-cleaning chemistry required for scale removal.

Table 4: **Comparison of Oven and Salt Bath Processes**		
Parameter	***Oven***	***Salt Bath***
Initial Temperature	Can be ambient	Higher than molten temperature of salt
Final Temperature	Can be ambient	Varies
Process Time	Determined experimentally	Determined experimentally
Process Temperature	~ 750°F - 850°F	~ 750°F - 800°F
Oxidizing Agent	Oxygen from air	Salt chemicals
Location of Oxidation	Surface of fiber	Surface of salt
Control of Oxidation	Blanketing	Very little
Energy Absorption Medium	Metal part & air	Salt bath medium
Primary Product of Combustion	Carbon dioxide	Carbonates
Type of Scale	Oxides	Carbonates and oxides

Table 4 provides a comparison of oven and molten salt bath cleaning. Environmental and cost considerations are not included. The comparison only addresses effects on the media.

In looking at historical data for filters that have been exposed to HTO oxidation processes over multiple cleaning cycles, the filters hold up to 20+

cleanings with no appreciable drop in Bubble Point or Flow Test Pressures.

Safety Issues: ***It is assumed that only experienced operators perform the HTO processes described. Improper techniques can result in injuries to the operator, damage to the parts, and, possibly, damage to the facility.***

SUMMARY

In an application where carbon residuals are left in the filter media after normal cleaning processes, there are methods to facilitate the removal of the carbon.

Physical methods, such as flushing and ultrasonics, can be used to reduce the amount of carbon in a filter. The effectiveness of removal depends on the type of media and removal method.

Chemical methods, in particular HTO processes, will remove carbon from the media. Ovens and salt baths are commonly used to oxidize the carbon for chemical removal.

As the degree of effective removal increases, so does the potential for damage. In deciding on the degree of carbon removal required, the user should understand

- methods for removing carbon
- degree of cleanliness expected from the processes
- mechanics and chemistry of the processes
- potential for damage associated with the processes

Cleaning filters, as opposed to discarding them, can result in tremendous savings in filter purchases and disposal costs. Using *proper techniques and methods,* carbon residuals and carbon containing scale can be *safely* and *effectively* removed from fiber metal media.

Sue L. Reynolds, Vice President/Technology and Senior Chemist, has been with Carolina Filters for 10 years. Her major duties include technical field support, process development and engineering, and technical support for the production facility. She has a Bachelors Degree in Chemistry from Winthrop University and a Masters Degree in Mathematics from the University of South Carolina.

ACKNOWLEDGMENTS

Special thanks to those companies and persons who provided materials, time, and technology in helping collect and evaluate data for these studies.

REFERENCES

- ASM Handbook Committee, *Metals Handbook, 8th edition, Vol.2, Heat Treating, Cleaning, and Finishing,* American Society for Metals.
- Bekaert Fibre Technologies, Belgium. Johan Vandamme Bekaert Fibre Technologies. Photography work on media coupons for ***Study 1***.
- Barnes, Leonard A., Jr., Bekaert Corporation, Consultation, 1996.
- Committee of Stainless Steel Producers, *Cleaning and Descaling Stainless Steels,* Washington, DC, 1982.
- Carpenter Steel Division, *Carpenter Alloys for Controlling Severe Corrosives*, Carpenter Technology, Reading PN, 1989.
- Duffy, Robert E., "Basic Methods for Removing Polymer from Parts", International Fiber Journal, June 1991.
- Lai, George Y. , *High Temperature Corrosion of Engineering Alloys*, ASM International, 1990.
- Kopietz, Heinz, Ajax Electric Co. Consultation, 1996.
- Malloy, James C., "Polymer Removal from Melt Spinnerettes and Related Fiber Production Components", Kolene Corp., Presented in Charlotte, NC. June, 1989.
- Privitt, Dr. James, PhD. University of South Carolina. Consultation, 1998