An optical method for characterizing carbon content in ceramic pot filters

J. Y. Goodwin, A. C. Elmore, C. Salvinelli and Mary R. Reidmeyer

ABSTRACT

Ceramic pot filter (CPF) technology is a relatively common means of household water treatment in developing areas, and performance characteristics of CPFs have been characterized using production CPFs, experimental CPFs fabricated in research laboratories, and ceramic disks intended to be CPF surrogates. There is evidence that CPF manufacturers do not always fire their products according to best practices and the result is incomplete combustion of the pore forming material and the creation of a carbon core in the final CPFs. Researchers seldom acknowledge the existence of potential existence of carbon cores, and at least one CPF producer has postulated that the carbon may be beneficial in terms of final water quality because of the presence of activated carbon in consumer filters marketed in the Western world. An initial step in characterizing the presence and impact of carbon cores is the characterization of those cores. An optical method which may be more viable to producers relative to off-site laboratory analysis of carbon content has been developed and verified. The use of the optical method is demonstrated via preliminary disinfection and flowrate studies, and the results of these studies indicate that the method may be of use in studying production kiln operation.

Key words | carbon content, ceramic pot filters, disinfection, flowrate, kiln operation, optical analysis

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INTRODUCTION

Household water treatment (HWT) is a common way for improving drinking water quality in terms of fecal pathogen disinfection in developing areas. Sobsey *et al.* (2008) and Hunter (2009) identified ceramic pot filters (CPFs) as one of the most effective available HWT methodologies. CMWG (2011) credits Fernando Mazariegos of the Instituto Centro Americano de Tecnología Industrial in Guatemala with developing the modern CPF design. That design consists of forming a mixture of native clay soil, a fugitive pore former (commonly sawdust), and water into a shape resembling a large flowerpot, firing the clay body in a ceramic kiln, and applying a silver compound solution to the final product. Salvinelli & Elmore (2015) reported that a typical CPF raw water volume is 9 L, and CMWG (2011) reported that CPF factories used a range of batch flowrate values of 1–3 L/h as the principal quality control criterion. A major advantage of CPFs is that the raw materials (with the exception of the silver solution) are typically available locally, and the production process can effectively be implemented using local labor.

CPF studies can be generally classified into the following categories based on the type of filter:

- production CPFs manufactured in developing countries (van der Laan *et al.* 2014; Salvinelli *et al.* 2016; and others);
- experimental CPFs fabricated in laboratories using either native clay soils or engineered clay (Plappally *et al.* 2011; Yakub *et al.* 2013; and others);
- disk or other non-pot shaped filters fabricated in the laboratory (Oyandel-Craver & Smith 2008; Ren & Smith 2013; and others).

It is common for researchers to characterize the porous nature of the completed ceramic body (Yakub *et al.* 2013; Soppe *et al.* 2015, and others) using the explicit or implicit assumption that the kiln firing sequence effectively combusts all of the fugitive pore former so that the final filter consists only of the vitrified ceramic body and the applied silver solution. Therefore, the firing sequences (temperatures and times) of factory kilns are not widely reported in the literature. Certainly, the effective construction and operation of a CPF kiln is readily recognized by manufacturers, and CMWG (2011) provides a relatively detailed description of kiln construction and firing variables. That document discusses means of kiln fire control including visual observation of the filters during firing, the use of pyrometric cones, the use of pyrometers and thermocouples, and the development of a written firing log. CMWG (2011) also describes draw trials as a means for confirming that the pore former has been completely removed from the production CPFs without requiring the destructive testing of the production filters. The interior or core of an incompletely fired CPF will include a layer colored black by carbonized pore former. A draw trial includes the insertion of incompletely fired CPFs pieces in a kiln, and subsequent manipulation of the firing parameters until fracturing of a trial shows that all of the carbon (by visual inspection) has been removed. By inference, CMWG (2011) states that proper firing of a CPF includes complete carbon burnout and fugitive pore former removal.

The authors observed the CPF manufacturing process at a factory near Antigua, Guatemala. The sawdust is past through a wire mesh to eliminate particles larger than a No. 8 sieve and is then mixed with clay from Rabinal, Guatemala at an approximate weight ratio of 6:1 clay-to-sawdust and wetted to create a working body. When it appears homogenous via visual inspection, the working body passes through an extruder to finalize mixing. The working body is then formed into a pot shape with a mechanical press. The shaped CPFs are then set to dry completely on shelving in the open-air factory for 1–3 weeks before being fired in a kiln. The filters are soaked in water overnight to saturate prior to a quality control flowrate test. A 1-hour falling head test is conducted in duplicate to ensure that the flowrates of the filters are between 1 and 2 L/h. Filters that pass the test are painted inside and out with a silver solution prior to distribution to consumers.

The Guatemala factory has two crossfire updraft kilns, each of which has a firing zone volume of $8.5\,m^3$ that allows the kiln to be loaded with four CPFs across, nine deep, and six high for a nominal capacity of 200. There are six Ransome venture propane burners per kiln which have a heat capacity of $3.2 \times 10^8\,J$ each. The kiln's walls are constructed of 0.064 m insulating fire brick and the interior is insulated with 0.10 m of fiber module. The firing process has a total duration of 7–8 hours. The kiln burner is operated for 3 hours to raise the temperature to 450–500 °C, then turned off for 1.5 hours with the vents closed, which limits the oxygen in the kiln and induces a reducing atmosphere. The burner is turned on again, increasing the temperature to a maximum of 750 °C before the kiln is turned off and the top vents are opened for cooling. The open vents allow some burnout of carbon beginning from the outside of the CPFs; however, oxygen is limited and the burnout occurs slowly. Once the kiln temperature cools below the combustion point of the sawdust the burnout stops, leaving a carbon core in some unknown percentage of the CPFs, as shown in Figure 1. It is of note that Rayner *et al.* (2013) reported that of the factories included in their worldwide survey, two of 16 respondents aimed to leave a carbon core in their production CPFs. The Guatemala CPF factory was identified as a participant

Figure 1 | Production CPF sampled to show carbon core throughout the body.

in that study, but it was not listed as one of the two facilities that were intentionally producing carbon cores.

The use of activated carbon as a HWT to improve esthetic water quality has been marketed in the USA for several years by several manufacturers. A comparison of commercially available HWT systems by Al-Haddad *et al.* (2015) showed that an unspecified carbon-based filter (available in the USA) was reasonably effective at improving water quality including reducing free chlorine concentrations and providing bacterial disinfection. This study, along with others, may be the basis for the Guatemala CPF manufacturer's marketing statements that the presence of a carbon core in CPFs is a benefit to purchasers of their product even though CMWG (2011) explicitly describes a methodology for eliminating the core.

There are two primary questions associated with the presence of carbon cores in production CPFs such as the one shown in Figure 1: does a carbon core (incomplete firing) impact the efficacy of the CPF, and does the core provide a supplemental treatment benefit? A first step in addressing these questions is the identification of a means to characterize the carbon core at CPF production locations where Western laboratory services may not be available, may be prohibitively expensive, or may be logistically restrictive in terms of sample preservation and shipment. This paper describes an optical methodology that may have the practical potential for characterizing the carbon content of a production CPF without laboratory analyses. The paper also demonstrates the use of the optical methodology in a preliminary study of the impact of the carbon content on filter disinfection efficacy and flowrate.

METHODS

This study used disk-shaped ceramic filters fabricated at the Missouri University of Science and Technology. The composition of the filters was selected to be both representative of the clay soil used by the Guatemala factory and to have a color that would provide a reasonable color contrast with any carbon core.

The clay body used in this study used the formula of the surrogate developed by Hubbel *et al.* (2015) to resemble the clay from Rabinal, Guatemala used in the CPF factory. The lower levels of iron in the surrogate clay gave it a lighter color relative to the native Rabinal clay soil and was beneficial for this study because it provided a contrasting background relative to the dark carbon core. Yellow pine sawdust was sieved through a No. 8 sieve and combined in a 6:1 weight ratio with the clay, then mixed with deionized water until homogenous. Experimental filter disks were formed with a 10 cm diameter manual press set to a thickness of 2 cm which was the typical thickness of the bottom of the filters produced in Guatemala. After pressing and before firing, the filters were allowed to dry for at least a week and spent at least 8 hours drying in a soils oven set at 100 °C. The filters in this study were not applied with colloidal silver in an effort to simplify the focus of the study because CPFs have been found effective even without silver present (Oyandel-Craver & Smith 2008; Clark & Elmore 2011).

The filters were fired in Cone Art Kilns, Inc. Model No. BX2327D electric kiln in three separate firings using identical schedules (temperatures and times) but with different atmospheres in order to develop multiple carbon core thicknesses. The firing schedule slowly increased the kiln temperature to 993 °C over the course of 14 hours before allowing it to cool back to room temperature. Ten filters were fired in each firing. For the mid-range atmosphere firing, oxygen was limited locally by placing the filters in a cordierite sagger box with a partial seal, and placing other combustible material (additional sawdust) in the box to partially scavenge oxygen that entered the box. The resulting atmosphere limited oxygen to result in incomplete burn out and leave some carbon remnants in the completed filter. For the reducing atmosphere firing, the process was repeated with additional combustible material in the sagger box and a thicker seal. The filters in this firing experienced less burnout than the first firing with a thicker carbon core. The oxidizing atmosphere firing was performed in air (without a sagger box), causing all of the pore-forming sawdust to fully combust.

The carbon core decreased in thickness radially away from the center of the mid-range and the reducing atmosphere disks because more oxygen was able to penetrate during firing into the disk edges compared to the disk center. Figure 2 shows how a 5 cm diameter sample was cut from the center of each of the 10 cm disks to avoid these edge effects.
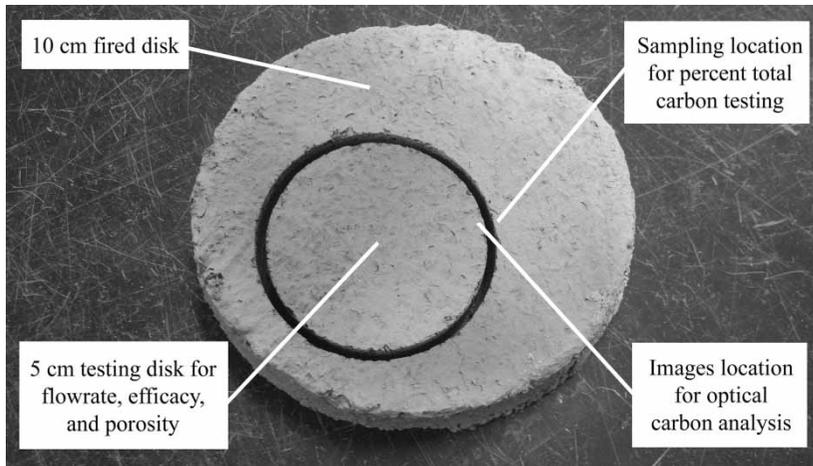
Figure 2 | Experimental filter sampling locations.

The carbon content was analyzed both optically and by laboratory analyses. Laboratory samples were collected by powdering approximately 5 g of the inner perimeter of the 10 cm disks after the previously described 5 cm disks had been removed. The powder was then sent to the University of Missouri Extension Soil Testing Laboratory in Columbia, Missouri for total carbon analysis by combustion using an Elementar Vario MAX Carbon/Nitrogen Analyzer (Pella 1990; Nelson & Sommers 1996). The total carbon by combustion analysis is similar to methods used by Eliche-Quesada *et al.* (2011, 2012). Optical testing was performed by taking three images along the circumference of the 5 cm disks with both a Leica S8APO stereo microscope and a HiROX KH-8700 digital microscope. Two different instruments were used in order to validate the optical methodology. The Leica exposure was set at 12.6 ms with 2.1× gain, 100% saturation, and 1.00 gamma. The HiROX exposure was 1/250 s with 3 dB gain and white balance set at red 184 and blue 139. Each image was cropped to include only the filter material and then converted to greyscale. MATLAB 8.4 code was written to import the images as an array with a value ranging from 0 for black to 255 for white for each image pixel, and then the code returned the median value of the array as the tone value for the image. A paper standard was used to compare the performances of the two instruments. The standard had six printed blocks of color: white, white darker 5%, white darker 15%, white darker 50%, black lighter 25%, and black. An image was taken of each color block on each imaging device to identify a representative tone value for each block.

Seven samples from production filters made at the factory in Guatemala were also analyzed for carbon content. The cross sections of the samples contained a dark carbon core similar to that shown in Figure 1. The presence of the core indicated that complete combustion of the pore former had not occurred even though there was no visual evidence of carbon core on the surface faces of the samples. Three images were taken from the cross-sectional surface of each CPF sample to be representative of a filter that had experienced full burnout. The cross section of each sample was imaged seven times to characterize the carbon core, and the surface face of each sample was imaged three times to provide a background greyscale value for the ceramic body. The images were taken on both the Leica stereoscope and the HiROX microscope, and were processed in the same way as the experimental filter images to obtain a representative tone for each CPF sample.

Apparent porosity was characterized using a modification of the Archimedes method (ASTM C830-00). Two experimental filters, one with complete burnout and one with incomplete burnout with apparent high carbon content were tested. Instead of using a vacuum and pressure chamber to saturate the samples, each sample was left to soak submerged in water for at least 18 hours to achieve saturation. This procedure is similar to the procedure followed by the CPF factory in Guatemala to saturate the production filters before flowrate testing.

Disinfection and flowrate testing were performed by placing six experimental filters from each of the three firings in 5 cm diameter PVC pipes as shown in Figure 3. The perimeter of each filter was sealed against the pipe wall using marine-grade silicone caulk. The filters were disinfected according to the procedure used by Clark & Elmore (2011) with three pore volumes (45 mL total) of bleach water solution (0.08 mL/L chlorine bleach). Once the filters were disinfected, each filter was filled with a sodium thiosulfate solution (0.25 mg/L) to remove any residual chlorine. The effluent water was tested for total chlorine with a Hach Total Chlorine field kit (CN-66T) to ensure that all chlorine was removed. If the test kit result indicated that total chlorine was still present, additional sodium thiosulfate solution was passed through the filter until the test kit results were below the detection limit. A presence/absence Colilert® coliform test analyzed the effluent from each filter to ensure that there were no coliforms present in the effluent from the filters before testing began.

To test the experimental filters, a simulated contaminated surfaced water was produced using influent from the municipal wastewater treatment plant (WWTP) in Rolla, Missouri. The WWTP influent was mixed with tap water to create the testing influent water ('challenge' water) for the filters, which was passed through the filters for 4 weeks. The experiment length of 4 weeks was chosen to allow sufficient time for potential microbial growth using fugitive pore former as a carbon source. The challenge water coliform concentrations were on the order of 10^2 colony forming units per 100 mL (cfu/100 mL) for the first week, on the order of 10^3 cfu/100 mL for the second and third weeks, and on the order

of 10^4 cfu/100 mL for the fourth week. These levels of contamination have been observed in surface water sources in Guatemala (Elmore et al. 2005). Challenge water total coliform and Escherichia coli levels were measured initially with Colilert Quantitray 2000® but after the first week were measured with Coliscan Easygel® in triplicate as the coliform concentrations grew too high to be quantified with the Quantitrays (which read up to 2,419.6 cfu/100 mL). Effluent water was tested for total coliforms and Escherichia coli with Colilert Quantitray 2000® each time the concentration of coliforms in the challenge water increased to check for bacterial breakthrough.

After 4 weeks of testing, more highly contaminated challenge water (on the order of 10^5 cfu/100 mL) was filtered to force failure so that the logarithmic reduction value (LRV) could be calculated. LRV is a measure commonly used to measure the bacteria removal efficacy of point of use (POU) water treatment systems (van Halem et al. 2007; Sobsey et al. 2008; Clark & Elmore 2011). The influent water was the same concentration for all filters and was tested for total coliforms and E. coli using Coliscan Easygel® in triplicate. The effluent from each filter was tested for total coliforms and E. coli with Colilert Quantitray 2000® to quantify the LRV.

RESULTS AND DISCUSSION

Figure 4 is a comparison of the performance of the two instruments used in the optical analysis. The scattergram shows that the two instruments resulted in relatively similar results for the three analyzed sample sets because the data have relatively good straight-line fits. The slopes of both the experimental filters data set and the production CPF samples data set have an absolute difference of 0.13 from the ideal value of 1, and the R^2 values of the paper standards data set and the production CPF samples set are greater than 0.9. The poorest fit was associated with the experimental filters data set where the R^2 value was less than 0.8.

Summary statistics for the experimental filter physical carbon content analysis, optical analyses, flowrate tests, and efficacy results in terms of log reduction values are given in Table 1.

The mean carbon content increased as the reducing atmosphere in the kiln increased. However, the variability
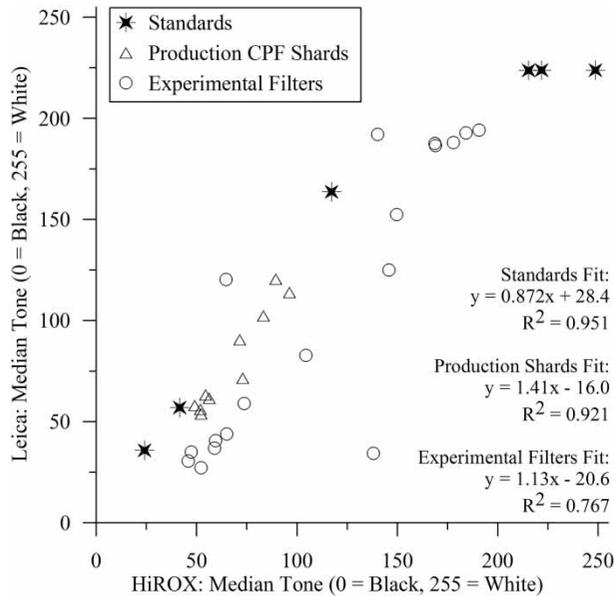

Figure 3 | Flowrate testing apparatus.

Figure 4 | Comparison of optical instruments.

of the carbon content as measured by the coefficient of variation (CV) did not directly correlate to changing atmosphere conditions, and the mid-range carbon content samples was the only data set with a CV greater than 1. The mid-range firing group was a highly varied group with a CV of 1.17, the only group with a CV greater than 1. Three of the filters in the mid-range group had lower carbon contents, and these three filters were closer to the kiln vent, which was partially opened to moderate the firing atmosphere. The individual filter positions in the kiln were not meaningful

in terms of carbon content variability for the other two sets of filters because the vent was either fully open (oxidizing) or fully closed (reducing).

Comparisons of the median tone values generated during the optical analyses and the laboratory carbon analysis results are shown in Figure 5. There is an apparent logarithmic relationship between the total carbon content and the median tone value for both instruments. The coefficient of determination between the carbon content and the median tones was less than 0.9 for both instruments, and inspection of the plots shows that the correlation between the optical analyses and the laboratory analysis is better for both the oxidizing and reducing sample sets relative to the mid-range sample sets. The poorer fit of the mid-range data may be related to the kiln position of the samples as discussed above.

The porosity was measured for one oxidizing filter and one reducing filter. The apparent oxidizing filter porosity was 44.3%, and the reducing filter porosity was relatively similar at 43.8%.

The bacterial efficacy LRV values were plotted as functions of the laboratory carbon content results and the Leica median tone values in Figure 6. The median tone values generated using the Leica instrument were selected because of the slightly higher R^2 value relative to the HiROX instrument listed in Figure 5. The oxidizing and reducing data sets plot in essentially vertical areas for both methods of characterizing carbon content because the log

Table 1 | Summary of experimental filter testing

Group	Statistic	Carbon content (%)	Visual median tone		Flowrate (mL)		LRV
			HiROX	Leica	Initial	Final	
Oxidizing	Mean	0.0284	172	190	44	29	3.66
	Std dev.	0.0064	17.7	3.2	5.8	4.9	0.48
	CV	0.224	0.103	0.017	0.131	0.167	0.131
Mid-range	Mean	0.216	110	92	49	29	4.09
	Std dev.	0.252	40.8	49.0	5.9	7.7	0.41
	CV	1.166	0.370	0.533	0.121	0.260	0.101
Reducing	Mean	1.423	57	39	63	35	2.84
	Std dev.	0.274	10.9	11.4	5.2	8.1	0.83
	CV	0.193	0.190	0.290	0.083	0.234	0.294
Overall	Mean	0.556	113	107	52	31	3.52
	Std dev.	0.667	54.1	69.9	9.6	7.1	0.79
	CV	1.200	0.479	0.652	0.185	0.228	0.225

Std dev., standard deviation; CV, coefficient of variation.
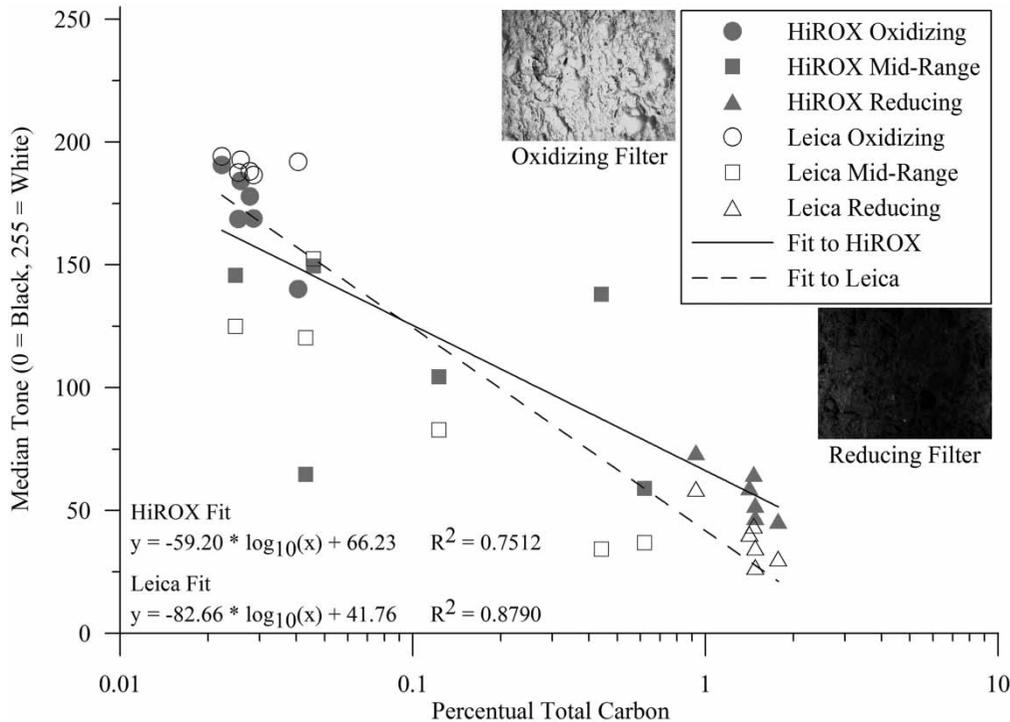
**Figure 5** | Comparison of optical and laboratory carbon content analyses.

carbon content ranges were relatively small for each atmosphere. However, the lower bound of the LRV range is smaller for the oxidizing data set. The mid-range atmosphere data points show that the range of the log carbon contents was greater relative to the data sets from the other two atmospheres. The range of LRV values was similar to the oxidizing LRV range except that the upper value of the mid-range atmosphere was above the top of the oxidizing LRV range. Figure 6 shows that the LRV values are more tightly grouped for the samples from the oxidizing atmosphere data set, but relationships between either percent carbon or median tone were not identified for any of the atmospheres or the data set as a whole.

The flowrates for each filter were measured twice, once at the beginning and once at the end of the experimentation. All 18 filters experienced a reduction in flowrate over the study period. As shown in Table 1, the CV was greater for each group at the final measurement than at the initial measurement, indicating that the use of the filters lead to a more variable flowrate. Flowrate means were similar for the oxidizing and mid-range groups but higher for the reducing group at both the initial and final measurements. Initially, the oxidizing

group flowrates showed the greatest variation as characterized by CV, and the reducing group flowrates had the smallest CV. After 4 weeks of use, the oxidizing group flowrate CV had fallen below the CVs of the other two flowrate data sets. Figure 7 shows the results of the flowrate testing. Only the laboratory carbon analyses are shown in the figure because the optical median tone plots are essentially the same in terms of general trends. The observations made about the LRV plots apply to the flowrate plots except that the magnitude of the flowrate ranges increase as reducing conditions increase. The data were inconclusive regarding the effect of any carbon core-supported biological growth on flowrate.

CONCLUSIONS

A logarithmic relationship could be identified between the median tones generated using the optical technique and the percent carbon in experimental and production filters. The results were similar for both optical instruments used in the study, but the fit of the equations was relatively poor due to variability in the carbon content of the
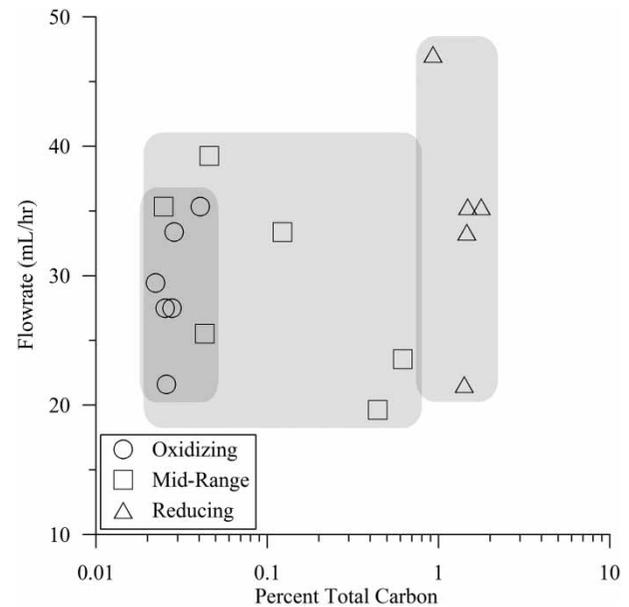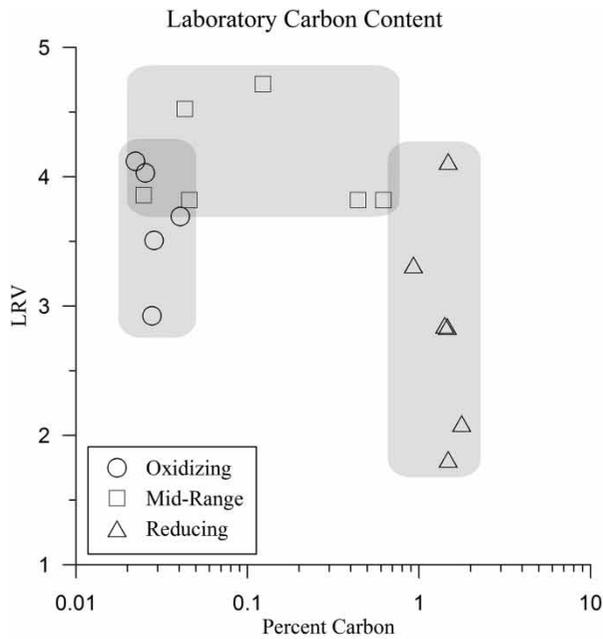
Figure 7 | Flowrate versus carbon content measured in the laboratory.
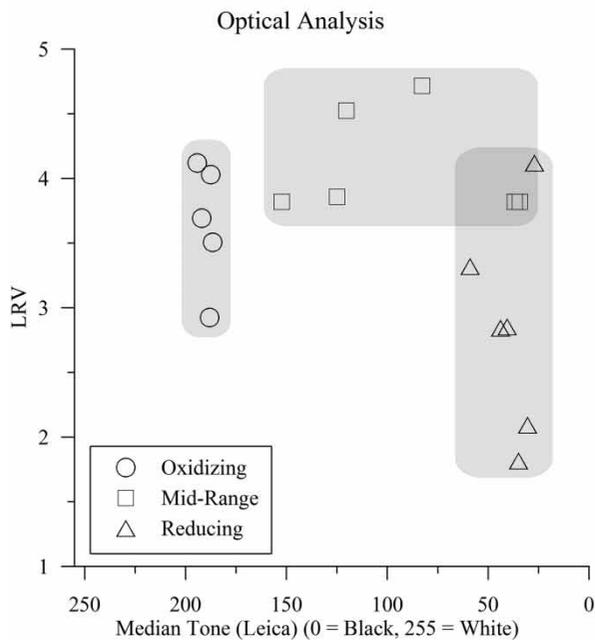
Figure 6 | LRV versus carbon content as measured in the laboratory and optically.

mid-range atmosphere samples which was largely attributed to the position of the filters in the kiln.

The variability of the mid-range atmosphere samples was also reflected in the LRV and flowrate analyses. However, the data from the initial study did not identify mathematical relationships between carbon content and

disinfection efficacy or filter flowrate. More specifically, the carbon contents of the oxidizing atmosphere filter samples were relatively similar while the corresponding LRVs and flowrates had relatively larger ranges of values. The same observations apply to the reducing atmosphere filters. While it is tempting to conclude that flowrate and LRV are insensitive to the carbon content of a filter, the sample sizes used in the preliminary study are too small for definitive characterizations.

This initial study suggests that production CPF performance may be more tightly controlled with regard to LRV and flowrate if the pore former is fully combusted (no carbon core) via manipulation of the firing sequence to create an oxidizing atmosphere in the kiln. This implies that manufacturers may be able to produce more consistently performing CPFs if they maximize pore former burnout.

Finally, it is reasonable to assume that production facilities will not be able to control the firing atmospheres to the degree that those atmospheres can be controlled in the laboratory. Given the variability of carbon content and filter performance associated with position within the kiln for the mid-range atmosphere included in this study, it is reasonable to recommend additional studies regarding kiln packing.

ACKNOWLEDGEMENTS

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