Cleaning Trials with Permanent Ink on Frosted Glass

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Introduction

The porous frosted glass areas on standard microscope slides are difficult to clean. Soiling them with a dark soil such as permanent marker ink allows the cleanliness to be quantified photographically. Many common permanent inks are difficult to remove because they contain dark pigments (i.e. carbon black, soot-like particles) with a polymeric binder. A difficult-to-remove soil on a porous surface allows us to answer many relevant cleaning process questions, such as:

1. Which individual component of a cleaning formulation is doing the most work?
2. What is the best temperature to use for cleaning?
3. What ultrasonic frequency is best for this situation?
4. Which cleaning solution or concentration is best?

Of course, a study like this will produce results that are most applicable to cleaning permanent ink on frosted glass. But the technique is universal and can be adapted to other substrates and soils that are relevant to the reader's situation.

Experimental Details

For this experiment, two standard permanent markers (Standard and Industrial Sharpie®) were used to soil the frosted portions of microscope slides. These soiled slides were cleaned in various cleaning solutions in a 45 kHz ultrasonic bath set at 50 °C and maximum power.

Experimental Procedure

1) The frosted portions of six glass microscope slides were completely colored using black markers.
2) Photos were taken of the soiled slides and several blank slides (as controls) in a photo box with white LED illumination.
3) Using image analysis software (ImageJ, https://imagej.nih.gov/ij/), regions were selected inside the black areas of the soiled slides and of the frosted area on the blank slides to measure the mean brightness values (0 to 255) for each slide.
4) The brightness values of the soiled slides were divided by the brightness values of the control slides to produce %-clean values. One minus these values produces a %-dirty value (% opaque).
5) The six soiled slides were cleaned with six different cleaning solutions in 100-mL beakers suspended in the ultrasonic bath.
6) Every 10 seconds, the slides were removed and analyzed using steps 1-5.
7) The locations of the six beakers were rotated to new positions after every 10-second cleaning interval.

Results

The %-dirty values were plotted vs time for each of the cleaning solutions (Fig. 1) showing what appears to be first-order exponential decay of the %-dirty curves. To test the first-order nature of these curves, the natural log of the %-dirty values were plotted vs time, and linear trend lines were computed (Fig. 2 and Table 1).

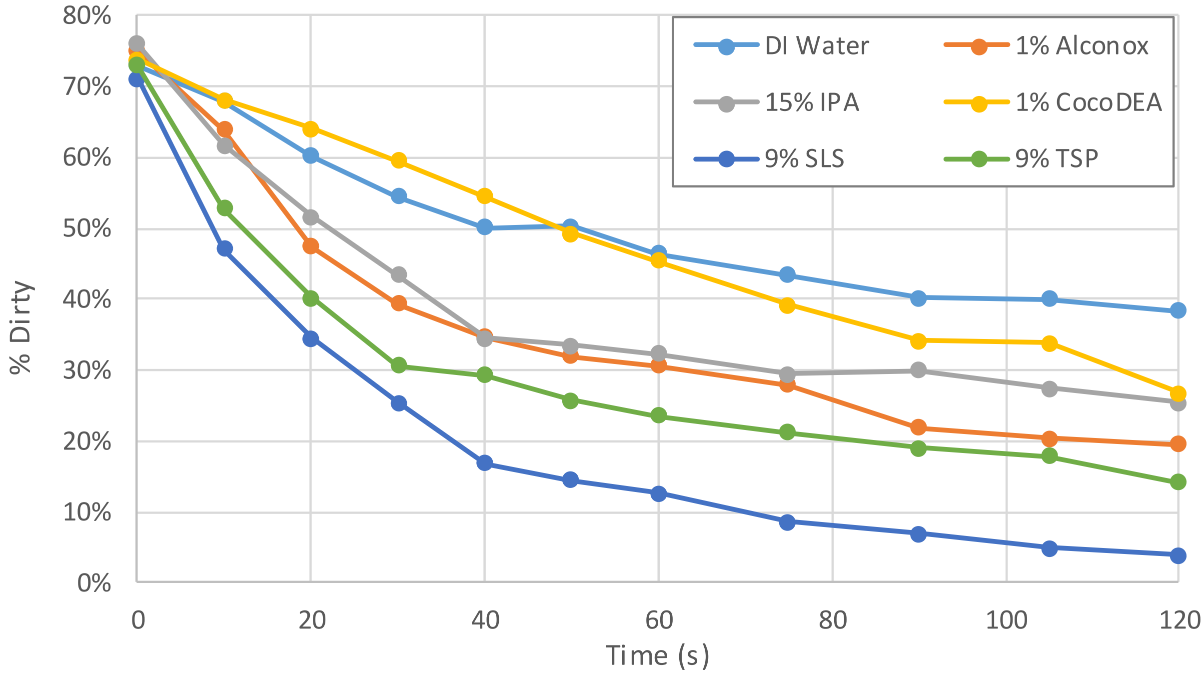

Figure 1: The comparison of six cleaning solutions against permanent ink on frosted glass with 45 kHz ultrasonics at 50 °C.

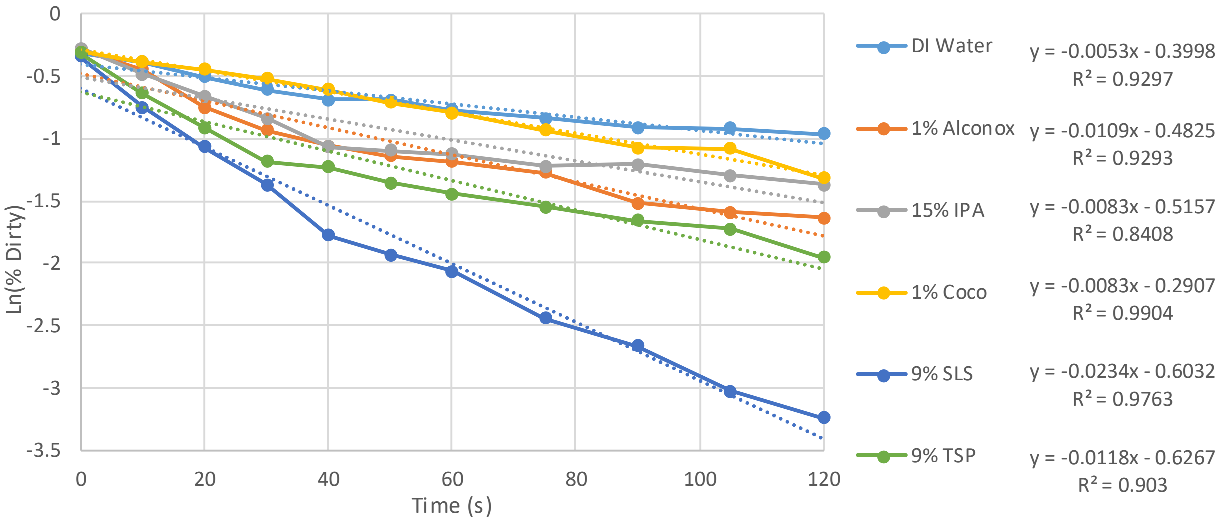

Figure 2: The natural log of the %-dirty curves showing the first-order-kinetic cleaning action as evidenced by the high R^2 values. The slope of each trend line is equal to the negative of the cleaning rate constant (-k).

Table 1: Comparisons of various cleaning formulation components using first-order kinetic analysis. The uncertainty estimates are ± 1 standard deviation.

Cleaning Formulation Component	**Cleaning Rate Constant k / s^{-1}**	**Cleaning Half-life $t_{1/2}$ / s**	**Ten Half-lives $10t_{1/2}$ / min**
DI Water	0.0053 ± 0.0005	130 ± 12	22 ± 2
1% Alconox	0.0109 ± 0.0010	64 ± 6	11 ± 1
15% Isopropyl Alcohol (IPA)	0.0083 ± 0.0012	84 ± 12	14 ± 2
1 % Cocamide DEA	0.0083 ± 0.0003	84 ± 3	13.9 ± 0.5
9 % Sodium Lauryl Sulfate (SLS)	0.0234 ± 0.0012	30 ± 2	4.9 ± 0.3
9 % Trisodium Polyphosphate (TSP)	0.0118 ± 0.0013	59 ± 6	10 ± 1

This same procedure was used to analyze five products from Alconox, two of which were received through their free test sample request program (Detergent 8 and Detonox). The soil in this case was an "industrial strength" permanent ink (Industrial Sharpie®). The cleaning curves for these five cleaners are shown in Figure 3.

Notice that there appear to be two rates of cleaning – a steep initial rate followed by a more gradual rate. The gradual rate will be the limiting rate of cleaning, so the later parts of these curves were analyzed with first order kinetics (Fig. 4).

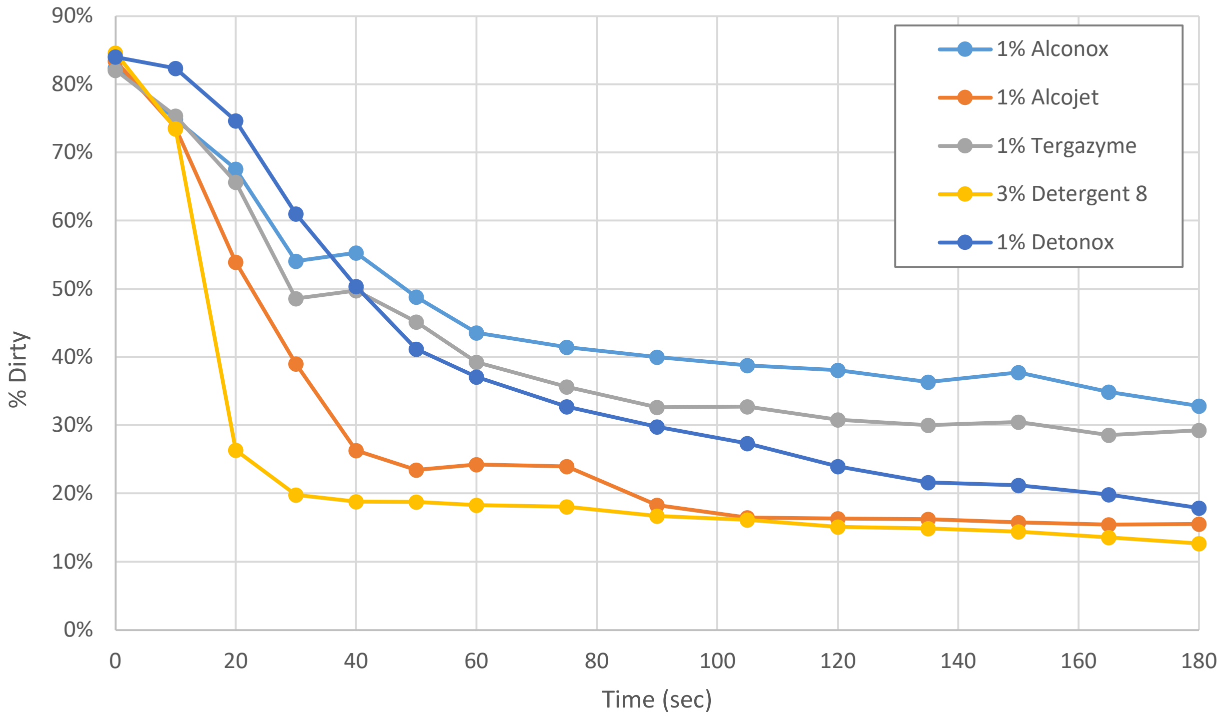

Figure 3: Comparison of five Alconox products against industrial permanent ink on frosted glass using 45 kHz ultrasonics at 50 °C

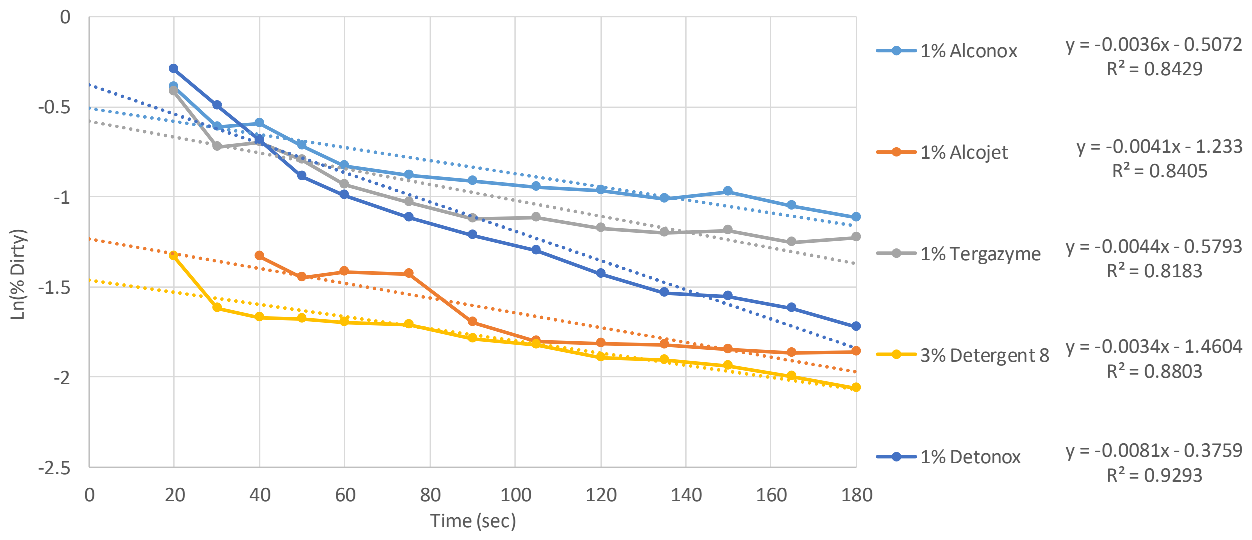

Figure 4: First-order kinetic analysis of the cleaning curves of five Alconox cleaners against industrial permanent ink on frosted glass cleaned with 45 kHz ultrasonics at 50 °C

Table 2: First-order kinetic analysis of five Alconox cleaners against industrial permanent ink on frosted glass cleaned with 45 kHz ultrasonics at 50 °C. The uncertainty estimates are ± 1 standard deviation.

Alconox Cleaner	**Cleaning Rate Constant k / s^{-1}**	**Cleaning Half-life $t_{1/2}$ / s**	**Ten Half-lives $10t_{1/2}$ / min**
1% Alconox	0.0036 ± 0.0005	191 ± 25	32 ± 4
1% Alcojet	0.0041 ± 0.0006	170 ± 25	28 ± 4
1% Tergazyme	0.0044 ± 0.0006	158 ± 22	26 ± 4
3% Detergent 8	0.0034 ± 0.0004	204 ± 23	34 ± 4
1% Detonox	0.0081 ± 0.0007	85 ± 7	14 ± 1

Discussion

The cleaning curves of the common cleaner components show that cleaning in deionized water was the least effective for this type of soil and substrate. The mechanical action of ultrasonic cavitation was able to remove some of the ink with a cleaning half-life of 131 seconds (Tab. 1). This long half-life predicts a completely-clean time of 22 minutes, assuming that the cleaning mechanism that worked on the first half of the soil will be valid for the remaining half of the soil. It is common for this assumption to fail for the last small amount of soil that is trapped in surface pores.

Figure 1 shows that the high detergency of sodium lauryl sulfate, aided by its higher concentration, was most effective in cleaning the ink from the frosted glass samples. This formulation component had the shortest cleaning half-life of 30 seconds with a predicted completely-clean time of 5 minutes as seen in Table 1.

A 1% by mass solution of Alconox performed in the middle of the group in the first cleaning trial with a cleaning half-life of 64 seconds and a predicted completely-clean time of 11 minutes. Trisodium polyphosphate (TSP) is a major component of the Alconox cleaner formulation. At 9% by mass, TSP produced a similar cleaning half-life of 59 seconds with a completely-clean estimate of 10 minutes.

Cocamade DEA and isopropyl alcohol (IPA) performed similarly in this trial with cleaning half-lives of 84 seconds and completely-clean estimates of 14 minutes. The IPA solvent produced a faster initial rate of cleaning and had the least linear first-order kinetics model ($R^2 = 0.84$). This reveals perhaps multiple cleaning mechanisms. The most logical mechanisms are rapid initial solvation of the polymeric binder followed by the mechanical action of the ultrasonic cavitation.

The industrial grade permanent ink was more difficult to remove as seen in Figure 3 and Table 2. The completely-clean estimate for 1% Alconox increased from the previous value of 11 minutes against the standard ink to 32 minutes against the industrial ink.

From Figure 3 it appears that there is some rapid solvency cleaning of the ink by the Alcojet and Detergent 8 cleaners. However, these cleaners slow down dramatically after the initial dissolution is complete. The mechanical action of the ultrasonic cavitation continues to show modest results over time with these two cleaners against this soil. The slow cleaning mechanism will limit cleaning the most. Therefore, only the slow portions of the cleaning curves were examined with first-order kinetic models (Fig. 4) producing the cleaning results in Table 2.

The best cleaner for the industrial ink seems to be Detonox even though it does not show the quick initial solvency. It maintained a consistent cleaning curve and is predicted to continue cleaning with a half-life of 85 seconds and a completely-clean estimate of 14 minutes.

Conclusions

Two cleaning trials were reported showing the results of various Alconox cleaners and cleaner components against two permanent marker inks on frosted glass. The detergency of sodium lauryl sulfate was shown to be most effective against these inks, and this detergency performance was confirmed by the performance of the high-detergency Detonox cleaner against industrial-grade permanent ink.

One thing should not be missed in this report. Whereas, the results are applicable to ink on frosted glass, the techniques shown here are universal. Standard sample coupons (like microscope slides), standard soils (like inks, greases, films, and polymers), and standard imaging techniques combine to provide the cleaning professional with a powerful tool for process improvement.

You can receive hands-on experience with these techniques at the Product Quality Cleaning Workshop (www.shsu.edu/pqcw), led by the Cleaning Research Group at Sam Houston State University.