Experimental Parameters for Small-Scale Cleaning Characterization. Part II: Effect of Fluid Velocity on the Kinetics of Cleaning

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ABSTRACT
Methodologies for estimating experimental parameters for small-scale cleaning characterization are described in this series: dilution of process fluids during clean-in-place (CIP) operations was discussed in Part I (1); the effect of fluid velocity on the kinetics of cleaning is described in this part; the effect of humidity, hold time and soil load on cleanability will be discussed in Part III.

The kinetics of cleaning under worst-case conditions is modeled from first principles. The model is based on diffusion-controlled mass transfer in a laminar falling film, which typifies worst-case cleaning conditions for CIP operations. The effect of flowrate per unit width (Q/W) and fluid velocity (V) on mass transfer rate in film flow is characterized. An experimental approach for optimizing Q/W and V for identifying worst-case soils for cleaning validation is described. The model is also used to estimate fluid velocity, film thickness and Reynolds Number for a range of values of Q/W and angle of inclination (?)

The results indicate that Q/W and V have a relatively weak effect on the kinetics of cleaning. For instance, when these parameters are doubled, the mass transfer rate increases only by a factor of 8% and 12%, respectively. The results also indicate that for 5° < ? < 90° and Q/W < ~1 gpm/ft (~2 mL/s/cm), the flow would be laminar and the thickness of the film would be < ~1 mm. Further, under these worst-case conditions, V would be < ~52 cm/sec, which is substantially less than the design criterion for minimum fluid velocity in pipes and hoses – viz. 150 cm/sec (5 ft/s).

INTRODUCTION
Small-scale cleaning characterization data can be used to streamline validation requirements for multiproduct equipment – i.e. equipment that is used to manufacture or clean more than one product. This is accomplished by ranking process soils associated with a given cleaning circuit based on the relative cleanability of the soils. The hardest-to-clean or worst-case soil for the circuit is then used to validate that circuit. This approach obviates the need to validate the cleaning of every soil associated with a circuit. It also facilitates the introduction of a new product into an existing multiproduct facility. If it can be shown that the process soils associated with the new product are easier to clean than the corresponding soils of the previously validated product, the new product can be introduced into the facility without revalidating the cleaning procedures (2).

In addition to streamlining validation requirements for multiproduct equipment (3, 4), small-scale cleaning characterization studies can also be used to identify suitable cleaning chemistries (5, 6), optimize cleaning parameters and processes (7, 8) and estimate cleaning times at full scale (9, 10).

Experimental models for small-scale cleaning characterization have been described in the literature (11-14). In these studies, the rate at which the process residue is removed from the surface – i.e., the mass transfer rate – is measured under simulated cleaning conditions. A critical step in the development of these models is to identify and scale down the hardest-to-clean (worst-case) location in the equipment (15). The worst-case location is typically an area with poor circulation, such as a
shadowed or occluded area. Note that it is not necessary to simulate the entire cleaning process at small scale; instead, it is sufficient to simulate the worst-case location within the equipment. If the process residue can be adequately removed from the worst-case location, it follows that it can also be adequately removed from other locations in the equipment. Thus, with this approach, the cleaning times obtained at small scale would be indicative of those at full scale, provided that there is adequate spray coverage at all surfaces that need to be cleaned, and the worst-case location is appropriately identified and simulated at small scale.

The scalability of small-scale cleaning characterization data depends on the accuracy with which relevant experimental parameters are estimated. Experimental parameters for simulating the worst-case location fall into two distinct categories (1):

- Parameters that can be readily determined from equipment and process data, such as:
  - Material of construction and surface characteristics such as roughness and curvature of coupons or parts used to simulate large-scale equipment;
  - Post-soiling parameters such as hold time, and ambient temperature and humidity; and,
  - Cleaning parameters such as rinse or wash time, temperature of rinse solvent, and temperature and concentration of cleaning solution.

- Parameters that typically need to be determined from first principles, such as:
  - Dilution of the process fluid during cleaning (i.e. soil to rinse solvent or cleaning solution ratio).
  - Velocity of rinse solvent or cleaning solution at the worst-case location – the subject of this paper.
  - Soil load.

**FLOWRATE AND FLUID VELOCITY AT WORST-CASE LOCATION**

Engineering standards provide guidelines for setting the minimum average velocity or minimum volumetric flow rate of rinse solvent or cleaning solution in various sections of a CIP circuit. For instance, it is recommended that for pipes and hoses the minimum fluid velocity \( V \) be \( \geq 5 \text{ ft/s} \) (1.5 m/s), and for film flows in vessels the minimum flow rate per unit width \( (Q/W)_{MIN} \) be \( \geq 2.5 \text{ gpm per ft of vessel circumference} \) (31 L/min/m) (16). These criteria are designed to ensure turbulent flow during CIP; the 5 ft/s criterion for \( V \) is also designed to ensure flooding in pipes and hoses.

For pipes and hoses, the 5 ft/s criterion for \( V \) can be readily met at all locations and is therefore relatively straightforward to simulate at small scale. For film flows, however, the flow rate per unit width at the worst-case location \( (Q/W)_{WCL} \) – such as the underside of an impeller blade or a magnetically coupled bottom-mounted impeller – is likely to be substantially less than \( (Q/W)_{MIN} \). Design and operational variables that contribute to \( (Q/W)_{WCL} \) being less than \( (Q/W)_{MIN} \) are summarized in Table 1.

<table>
<thead>
<tr>
<th>Design or operational variable</th>
<th>Sources of variation in ( Q/W ) at worst-case location</th>
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</thead>
<tbody>
<tr>
<td>Non-uniform distribution of flow through sprayball</td>
<td>Partial clogging of sprayball and/or incorrect installation of sprayball</td>
</tr>
<tr>
<td>Non-uniform distribution of surface area</td>
<td>Surfaces to be cleaned are not distributed uniformly along spatial coordinates</td>
</tr>
<tr>
<td>Non-uniform distribution of spray within vessel</td>
<td>Sprayball design, fabrication and/or location, proximity of worst-case location to sprayball, etc.</td>
</tr>
<tr>
<td>Indirect impingement and/or lack of impingement at worst-case location</td>
<td>Shadowed or occluded areas, angle of inclination of surface being cleaned, etc.</td>
</tr>
</tbody>
</table>

Table 1. Design and operational variables that contribute to the flow rate per unit width at the worst-case location \( (Q/W)_{WCL} \) being substantially less than the recommended minimum operating value \( (Q/W)_{MIN} \). For vessels, the recommended value for \( (Q/W)_{MIN} \) is 2.5 gpm per foot of vessel circumference.
The mass transfer rate from a stationary surface into a laminar falling film of a Newtonian fluid was investigated by Kramers and Kreyger for a flat rectangular layer (17, 18) and by Blount for viscous drops (19, 20). Their results indicate that the mass or molar flux \( N \) of the solute (A) into the fluid (X) is a function of the solubility \( S \) and diffusivity \( D \) of the solute in the fluid; the density \( \rho \), dynamic viscosity \( \mu \) and flowrate per unit width \( Q/W \) of the fluid; and the length \( L \) of the surface along the direction of the flow (Figure 1):

\[
N = k \cdot S \cdot (D L)^{2/3} \cdot (Q/W)^{1/9} \cdot (\rho^{-1})^{2/9}
\]  

(1a)

where \( \rho^{-1} \) is \( \mu/\rho \), the kinematic viscosity; and \( k \) is a constant that includes the acceleration due to gravity \( g \), the angle of inclination \( \theta \), and the width \( W \).

For a surface of given length, and a given fluid viscosity and angle of inclination,

\[
N \approx (SAX DAX^{2/3}) \cdot (Q/W)^{1/9}
\]  

(1b)

Further, for laminar falling films,

\[
Q/W \approx V^{3/2}
\]

where \( V \) is the average fluid velocity (21); thus,

\[
N \approx (SAX DAX^{2/3}) \cdot V^{1/6}
\]  

(1c)

The flux \( N \) of the solute into the fluid is the rate at which the solute is removed from the surface. Thus, \( N \) is effectively a measure of the kinetics of cleaning. Note that the effect of \( Q/W \) and \( V \) on the mass transfer rate – and therefore the kinetics of cleaning – is relatively weak. For example, when \( Q/W \) or \( V \) is doubled, the mass transfer rate increases only by a factor of 2 \( 1/9 \) (8%), and \( 21/6 \) (12%), respectively.

Equation 1 is valid when (1) the Reynolds Number \( Re = 4(Q/W)/\rho \) < 1500, the criterion for laminar flow in a falling film; (2) the velocity profile in the falling film is fully developed, a condition that holds when \( L >> \delta \), the thickness of the film; and (3) the distance over which the solute diffuses into the film \( d \) is << \( \delta \), and as a result, the velocity profile for \( 0 < y < d \) can be
approximated as a linear function of distance from the surface being cleaned (y) (Figure 1). In terms of the parameters in Equation 1, the third condition is satisfied when the solubility (S_A) and/or diffusivity (D_X) of the solute in the fluid are low enough for the mass transfer to be diffusion controlled.

The above conditions would be satisfied at the worst-case location in the equipment for a process soil that is difficult to clean, as this represents a worst-case scenario from the standpoint of cleaning – viz. diffusion-controlled mass transfer in a laminar falling film. Thus, Equation 1 can be used to characterize the effect of process parameters such as Q/W and V on the kinetics of cleaning under worst-case conditions and for design purposes. It should also be noted that since Equation 1 is derived for mass transfer of a single component (A) into a pure solvent (X), its applicability to complex multi-component process soils or solvents containing formulated cleaning agents would require the use of an effective solubility and diffusivity in the cleaning solution. Consequently, Equation 1 cannot be readily used to predict the cleanability of multicomponent soils; nonetheless, it can be used to characterize the effect of process parameters on the kinetics of cleaning, and to thereby identify and establish meaningful operating ranges for critical process parameters. Further, this equation can also be used to develop experimental models for cleaning (2).

An experimental approach for optimizing flowrate and fluid velocity for evaluating relative cleanability at small scale is described in the next section.

FLOW RATE AND FLUID VELOCITY FOR EVALUATING RELATIVE CLEANABILITY

Consider a system that is validated to manufacture and clean product A. A new product (B) needs to be manufactured and cleaned in the same equipment. A small-scale study is performed to evaluate the cleanability of A relative to that of B. If A is harder to clean than B, the new product could be introduced without revalidation. The objective is to determine the optimum flowrate per unit width (Q/W) and fluid velocity (V) for the small-scale study.

For products A and B, Equation 1b can be written as follows:

\[
N_A = (S_A D_A)^{2/3} \cdot (Q/W)^{1/9}
\]

\[
N_B = (S_B D_B)^{2/3} \cdot (Q/W)^{1/9}
\]

Thus,

\[
\frac{N_A}{N_B} = \frac{(S_A D_A)^{2/3}}{(S_B D_B)^{2/3}} \quad (2c)
\]

Since the cleaning time (t) is inversely proportional to the mass transfer rate (N), the cleanability of B (t_B) relative to that of A (t_A) can be expressed as

\[
\frac{t_B}{t_A} = \frac{N_A}{N_B} = \frac{(S_A D_A)^{2/3}}{(S_B D_B)^{2/3}} \quad (2d)
\]

Equation 2d indicates that relative cleanability (t_B/t_A) depends on the physical properties of A and B (viz. the solubility and diffusivity of A and B in the fluid). Further, relative cleanability is independent of Q/W, and therefore of V. Thus, if the objective of the study is to rank A and B based on cleanability, Q/W and V can be set to any reasonable value, provided that the resulting flow is laminar – in this case Re = 4(Q/W)/? < ~1000. In practice, however, a lower value of Q/W is preferable because the absolute difference between t_A and t_B (|t_A - t_B|) is amplified, which in turn makes it commensurately easier to differentiate between the two soils based on the larger magnitude of |t_A - t_B|. If necessary, |t_A - t_B| can be amplified by reducing Q/W up to the point where the film is still intact and uniform, i.e. it does not disintegrate into slower-moving unsteady drops.

An equation for estimating fluid velocity of a laminar falling film from Q/W and ? is derived in the next section.

ESTIMATION OF FLUID VELOCITY IN A LAMINAR FALLING FILM
A laminar falling film of a Newtonian fluid flowing primarily under the influence of gravity is shown in Figure 1. The flow is delineated as a thin sheet of liquid flowing down an inclined flat plate of length L and width W. As the liquid flows down the plate, it forms a film of thickness δ and develops a parabolic velocity profile, with the maximum velocity at the film surface. For this type of flow, the average fluid velocity (V) and film thickness (δ) can be expressed as follows (21):

\[ V = \frac{\rho g \delta^3 \sin \alpha}{3 \mu} \quad (3) \]
\[ \delta = \frac{3 \mu \omega}{\sqrt{\rho g W \sin \alpha}} \quad (4) \]

Where \( \rho \) is the density of the liquid, \( g \) is acceleration due to gravity, \( \alpha \) is the angle of inclination, \( \mu \) is the dynamic viscosity of the fluid, \( \omega \) is the mass flow rate, and \( W \) is the width of the film.

Equations 3 and 4 can be combined to eliminate \( \omega \) and express \( V \) in terms of measurable parameters:

\[ V = \sqrt{\frac{g \sin \alpha (Q/W)^2}{3 \nu}} \quad (5) \]

Where \( Q \) is \( \omega / \rho \), the volumetric flow rate, and \( \nu = \mu / \rho \), the kinematic viscosity of the fluid.

Equation 5 is valid under the following conditions: (1) when edge effects are negligible, a condition that is valid when L and W are \( >> \delta \); and (2) when viscous forces are large enough to prevent continued acceleration of the liquid along the length of the plate – i.e., at a low Reynolds Number, when the flow is laminar. Under these conditions, \( V \) is independent of the distance traversed along the incline (L). Note that at the worst-case location the above conditions would be satisfied because (a) the surfaces being cleaned are relatively large, and thus L and W would be \( >> \delta \); and (b) the flow would be laminar.

The Reynolds number (Re) is used to classify a falling film into three flow regimes: (a) laminar flow with negligible rippling (Re < 20); (b) laminar flow with pronounced rippling (20 < Re < 1500); and (c) turbulent flow (Re > 1500). When Re is less than 20, the ripples are very long and grow slowly down the surface of the film. As Re increases above 20, the ripple growth increases rapidly. Because of the assumptions made in developing the above model (21), the error in using Equation 5 to estimate velocity increases with ripple growth and Re. The velocities estimated using Equation 5 have been shown to be in good agreement with experimentally observed velocities when Re is less than 1000 (22, 23).

The average velocity of the cleaning solution is estimated from the flowrate per unit width (Q/W) and the angle of inclination (\( \alpha \)) using Equation 5. The estimates are based on the kinematic viscosity (\( \nu \)) of water at 20°C (0.01 cm²/s), acceleration due to gravity (g) of 981 cm/s², and a range of values of Q/W and \( \alpha \). The results, summarized in Table 2, indicate that for \( 5^\circ < \alpha < 90^\circ \) and Q/W < \( \sim 1 \) gpm/ft (\( \sim 2 \) mL/s/cm), the flow would be laminar and the thickness of the film would be \( < \sim 1 \) mm, if the film were stable (i.e., if it did not disintegrate into unsteady drops). Further, under these conditions, the average velocity of the fluid would be \( < \sim 52 \) cm/sec, which is substantially less than the design criterion for VMIN in pipes and hoses – viz. 150 cm/sec (5 ft/s).
Table 2. Average velocity, film thickness, Reynolds Number and relative mass transfer rate in a laminar falling film for a range of values of flowrate and angle of inclination.

<table>
<thead>
<tr>
<th>Angle of inclination (α)</th>
<th>Volumetric flowrate per unit width of film (Q/W)</th>
<th>Film thickness (δ)</th>
<th>Average velocity (V)</th>
<th>Reynolds Number $(4Q/W)^{1/2}$</th>
<th>Relative mass transfer rate (N/N°)</th>
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<tr>
<td></td>
<td>gpm/ft</td>
<td>mL/s/cm</td>
<td>mm</td>
<td>cm/s</td>
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<tr>
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<td>0.40</td>
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<td>828</td>
</tr>
<tr>
<td></td>
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<td>0.36</td>
<td>42.9</td>
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<td></td>
<td>0.50</td>
<td>1.03</td>
<td>0.32</td>
<td>32.7</td>
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</tr>
<tr>
<td></td>
<td>0.25</td>
<td>0.52</td>
<td>0.25</td>
<td>20.6</td>
<td>207</td>
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<td>60°</td>
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</table>

CONCLUSION

Small-scale experimental models are used to determine worst-case soils for cleaning validation, and estimate cleaning times and other performance parameters. A critical step in the development of these models is to identify and scale down the hardest-to-clean or worst-case location in the equipment. For cleaning operations, the worst-case location is typically an area within the equipment with poor circulation, such as a shadowed or occluded area. Examples of such locations include the underside of a probe or an impeller blade where there is no direct or indirect impingement of the cleaning solution during CIP operations. Instead, the flow of the fluid at the worst-case location is in the form of a laminar falling film.

A mathematical model for diffusion-controlled mass transfer in a laminar falling film was used to characterize the effect of flowrate per unit width (Q/W) and fluid velocity (V) on the kinetics of cleaning under worst-case conditions. The results indicate that the effect of these parameters on the rate of mass transfer — and therefore the kinetics of cleaning — is relatively weak. The mass transfer rate increases only by a factor of 8% and 12%, respectively, when Q/W or V is doubled.

An experimental approach for optimizing Q/W and V for evaluating relative cleanability at small scale was described. Relative cleanability was shown to depend on the solubility and diffusivity of the soils being compared. Further, for diffusion-controlled mass transfer — which typifies worst-case cleaning conditions for CIP operations — relative cleanability was found to be independent of Q/W and V. Thus, if the objective of the study is to rank coils based on cleanability, Q/W and V can be set to any reasonable value, provided that the resulting flow is laminar. In practice, however, a lower value of Q/W is preferable because the absolute difference between the cleaning times of the soils ($\Delta t$) is amplified, and as a result, the ability to differentiate between the soils based on the larger magnitude of $\Delta t$ is enhanced commensurately. If necessary, $\Delta t$ can be amplified by reducing Q/W up to the point where the film is still intact and uniform — i.e. it does not disintegrate into slower-moving unsteady drops.

The laminar falling film model was also used to estimate fluid velocity (V), film thickness (δ) and Reynolds Number (Re) from
Q/W and the angle of inclination (\(?\)). The results indicate that for \(5^\circ < ? < 90^\circ\) and \(Q/W < ~1\) gpm/ft (~2 mL/s/cm), the flow would be laminar and would be < ~1 mm, if the flow was stable. Further, under these conditions, \(V\) would be < ~52 cm/sec, which is substantially less than the design criterion for \(\text{VMIN}\) in pipes and hoses – viz. 150 cm/sec (5 ft/s). The calculated values of \(V\) have been shown to be in good agreement with experimentally observed velocities when \(\text{Re}\) is less than 1000.

**SYMBOLS AND ACRONYMS**

CIP  Clean-in-place  
\(?D\)  Diffusivity 
\(g\)  Acceleration due to gravity \(\text{g}\) 
\(L\)  Length of object being cleaned 
\(N\)  Mass or molar flux 
\(Q\)  Volumetric flow rate 
\(\text{Re}\)  Reynolds number 
\(S\)  Solubility 
\(t\)  Time 
\(V\)  Average velocity of cleaning solution 
\(W\)  Width of laminar falling film 
\(\?\)  Angle of inclination (slope) 
\(\?\)  Film thickness 
\(\mu\)  Dynamic Viscosity 
\(\nu\)  Kinematic viscosity 
\(\?\)  Density 
\(\?\)  Mass flow rate

**SUBSCRIPTS**

\(A\)  Product A  
\(B\)  Product B 
\(\text{MIN}\)  Minimum 
\(\text{WCL}\)  Worst-case location 
\(X\)  Fluid X

**REFERENCES**


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