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WASTE MINIMIZATION IN BATCH VESSEL CLEANING

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Although the cleaning of processing vessels has always been a significant source of waste, only recently have fundamental studies been undertaken to understand the cleaning process. The study described here began with a review of the literature on industrial cleaning practices. The literature was found to be mainly empirical. In order to provide a practical direction for research into the cleaning process, observations were made of a commercial batch processing facility. Based on these observations, short-term process improvements are suggested, and a new area for fundamental research is identified.
KEYWORDS Waste minimization Reflux cleaning Reactor Batch vessel.

INTRODUCTION

The cleaning of process facilities depends on the chemical dynamics at the surfaces of the equipment. Empirical practices have been developed to achieve contaminant removal to a degree found suitable for long-term process operations. However, many general practice cleaning techniques only involve parts of the equipment, with accumulation of contaminants allowed to occur elsewhere in reactors, columns, headers, etc. The conditions of fluid flow used during cleaning are frequently based on considerations other than effective contaminant removal. For example, the vessel design and agitation rate that is best for mixing the contents of a reactor are not necessarily optimum for cleaning the reactor walls. Various organic liquids and/or aqueous solutions are utilized to clean process equipment, and these liquids generate waste. Even if recycle is used, some significant amount of loss and waste results, usually far exceeding the actual mass of contamination removed. Thus, the complexities of industrial cleaning practices must be included in research which aims to understand the chemical dynamics at contaminated surfaces.

One research project currently underway is aimed at understanding the cleaning techniques and associated waste generation of batch processing vessels. As a starting point, two approaches were undertaken: the study of a commercial

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batch processing facility, and a literature search on the subject of vessel cleaning. At the plant it was found that most cleaning is accomplished by a technique known as reflux cleaning. Reflux cleaning involves partially filling a reactor with solvent, with subsequent heating of the solvent (with agitation) to its boiling point. The vapors from the boiling solvent then condense on the cold walls of the vessel, and the falling film of condensate rinses the walls.

Because of the wide use of reflux cleaning, an extensive search for literature on this technique was undertaken. Unfortunately, no literature was found on reflux cleaning (or any other vessel cleaning technique) in the chemical industry *per se*. Some literature on vessel and piping cleaning using liquids was found in the food industry literature, especially relating to milk processing. Although generally of an empirical nature, literature from the food industry was found to address several issues relevant to reflux cleaning, such as soil deposition and removal by falling films of liquid. For example, Tissier *et al.* (1988) used liquid holdup equations presented by Van Rossum (1958) as a check on the amount of residue left on a plate by falling films of model solutions and dairy products, and then measured rinsing kinetics as the residue was removed by a falling film of water. Tissier *et al.* (1981) have also studied the rinsing kinetics of milk storage tanks. The fouling of heat exchange surfaces by milk was studied by Lalande and Corrieu (1981), Tissier *et al.* (1985), and Tissier and LaLande (1986). The removal of milk deposits from heat exchange surfaces was addressed by Gallot-Lavallee *et al.* (1981), and removal of tomato paste residue from heat exchangers was studied by Jackson (1984).

Plett (1984, 1985b) hypothesized that the different phases of a rinsing curve in food processing piping are based on different mass transfer mechanisms. A rapid depletion phase attributed to axial dispersion is followed by a slower phase which Plett attributes to diffusion from dead spaces (e.g. pipe tees or valve junctions). A final, very slow decay period is attributed to soil desorption from the walls.

The subject of falling liquid films is germane to both the residue deposition process as a tank is drained, and residue removal by condensing vapors. The former depends primarily on the falling film hydrodynamics, while the latter additionally involves simultaneous heat and mass transfer. There is a considerable amount of literature on the subject of falling films dating back to the work of Nusselt (1916, 1933). Falling film hydrodynamics have been studied by Dukler and Bergelin (1952), Jackson (1955), Dukler (1959), and Ito and Tomura (1979), among others. The specific hydrodynamic problem of liquid holdup on a surface as it is withdrawn from a bath (or equivalently as the liquid is drained) was addressed by Van Rossum (1958), and later reviewed by Tallmadge and Gutfinger (1967). The effect of stainless steel surface finish on liquid film holdup was studied by Milledge (1981).

In the metalworking and electronics industries, a technique known as "solvent vapor degreasing" is widely used to clean metalworking fluids and other residues from parts and circuit boards. The technique is very similar to reflux cleaning in that condensing vapors are used to remove unwanted residues. Solvent vapor degreasing has been discussed by Meserve (1982), Johnson and Wedmore (1983a, 1983b), and Johnson (1990a). However, these works are primarily practical guides. Johnson (1990b) confirmed that there is a lack of fundamental understanding of the cleaning process in general, and of cleaning with condensing vapors in particular.

Surface cleaning using falling films not created by condensing vapors has also been addressed. Patel and Jordan (1970) studied the cleaning of a stainless steel plate with a falling film of liquid as a function of inclination angle, surfactant solution flow rate, and temperature. It was found that removal increased with flow rate and temperature, and that the maximum cleaning was achieved with a vertical plate. Chilukuri and Middleman (1983) developed equations to describe circulation, diffusion, and reaction within a liquid residue trapped in the microscopic cavities of a solid surface as another fluid flows over it. The theoretical results were tested experimentally by Tighe and Middleman (1985), and found to be in good agreement.

Several studies of mass transfer from solid surfaces into falling films are relevant to the fundamental residue removal process in reflux cleaning. Kramers and Kreyger (1956) developed an equation and conducted experiments to correlate the overall mass transfer coefficient between a dissolving wall and the laminar sublayer of a fully-developed, turbulent falling film. Oliver and Atherinos (1968) later showed that Kramers and Kreigers' correlation for the laminar sublayer also applied to laminar films with ripples. Kramers and Kreigers' data was also used by Iribarne *et al.* (1967) in conjunction with their own data to demonstrate the usefulness of their dimensionless variable system for correlating liquid–solid mass transfer in falling films. The dimensionless variables developed by Iribarne *et al.* were also used by Wragg *et al.* (1968) to correlate another set of data. More recently, Andersson (1987) relaxed the assumption of fully-developed flow, and presented a similarity solution to predict the solid/liquid mass transfer coefficient in an accelerating falling liquid film. His results include asymptotic solutions for high and low Schmidt number cases, and a numerical solution for use in the intermediate Sc range.

When considering contaminant removal, the cleaning of a pipe by a flowing liquid is physically similar to the cleaning of a vessel wall by a falling liquid film. Several studies have been published on the cleaning of pipes, again emanating from the food processing industry. Timperly (1981) showed that the cleaning achieved in a pipe could be related to the shear stress at the laminar boundary layer for turbulent flow. He reported that the shear stress and therefore cleaning efficiency in pipes of different diameters scales with linear velocity rather than Reynolds number. Nassauer and Kessler (1981) used a conductivity method to study the removal of brine film from pipes by rinsing with fresh water. Soil removal rate and turbulent shear stress in pipes were simultaneously measured by Paulsson and Tragardh (1985) using a constant-temperature anemometer.

In addition to the residue removal studies described above, several works have focused on the mechanism by which residues are bound to solid surfaces. Bourne and Jennings (1961) studied the adsorption behavior of proteins, examining the relative influence of electrostatic and van der Waals forces. They present evidence that two kinds of bonding exist in soil systems. The first is a soil–soil cohesion bond, which is relatively weak compared to the second, soil-surface adhesion bond. These results have been supported by the studies of Plett (1984, 1985b) discussed above, which show two or more different regions in the rinsing curve. Nassauer and Kessler (1985) concluded that globular proteins with hydrophilic groups were bound to a surface primarily by electrostatic forces, whereas denatured whey proteins where primarily bound by van der Waals forces between the surface and hydrophobic groups.

There are several general reviews of cleaning in the food industry which are also pertinent to a study of reflux cleaning. Plett (1985a) reviewed the cleaning of fouled surfaces in food processing equipment, focusing on equipment design, operating parameters, and surfactant solution characteristics. Another review, by Kulkarni *et al.* (1975), provides a comprehensive summary of cleaning in the food (especially dairy) industry. Methods of determining cleanliness are described, as are various factors effecting cleaning efficiency. Corrieu (1981) discusses methods of evaluating cleanliness, cleaning mechanisms and kinetics, and parameters such as detergent nature and concentration, temperature, mechanical action, surface finish, type of soil. His conclusion is that a systematic, fundamental approach to understanding cleaning is needed.

PLANT DESCRIPTION

Industrial-scale organic synthesis is often conducted in facilities with large numbers of batch reactor vessels and separation units. Several products are produced in each piece of equipment in campaigns from one to several batches in length. Typical processes include synthesis in 3.8 to 11.4 m^3 batch reactors and separation of product in 2 to 3 m diameter rotary vacuum dryers. The reactors and dryers are either stainless steel or glass-lined. In the stainless steel reactors, heat exchange is via internal coils. Dryers and glass-lined reactors are usually fully jacketed for temperature control.

Equipment operating procedures provide written guidelines for cleaning of the vessels. However, the exact method of cleaning a reactor with a particular chemical residue is frequently determined by the operator, based on experience with previous cleanings. Typically, the reactors are cleaned by partially filling with a solvent and refluxing for 30 minutes to several hours, after which a visual inspection is performed. If the vessel does not appear clean, the procedures are repeated with the same or a different solvent. Dryers are cleaned by filling with solvent and rotating with heat to within 15°C of the solvent boiling point. The dryers are visually inspected to determine the degree of cleanliness.

Cleaning of reactors or dryers usually generates from 1 to 10 m^3 or more of waste solvent, which is recovered in a dedicated solvent recovery system. However, since solvent recovery is not 100% efficient, every kilogram of solvent sent to recovery results in some amount of waste which is then incinerated or sent to biological treatment. In addition to recoverable solvent waste, cleaning also produces vent gases which typically are scrubbed to remove solvent vapors and then vented to atmosphere.

RESULTS

Three different methods were used to better understand industrial batch reactor cleaning: personnel interviews, construction of databases from production records, and generation of cleaning curves from vessel cleaning solvent samples. The goals of the investigation were to better quantify the cleaning procedures, to put boundaries on plant conditions which would help define critical basic research, and to establish preliminary conclusions for possible waste reduction.

Personnel Interviews

Twenty four of the operators, shift supervisors, and technical staff were asked to describe: (1) how reactors and dryers are cleaned, (2) how cleanliness is evaluated, (3) how much waste is generated during cleaning processes, (4) if any substances are particularly difficult or easy to clean, (5) if improper cleaning is a common cause of products which do not meet specifications, and (6) if they had any suggestions to improve cleaning. The responses to the first two questions have already been incorporated into the plant description given above. The personnel interviewed said that although a written record is kept of each cleaning, the amount of solvent used varies widely depending on the chemicals which are being processed and the vessels used.

Based on the interviews it was concluded that some chemicals are more difficult to clean from the vessels than others. However, the authors observed that it was difficult for personnel to identify a vessel-chemical system that was particularly difficult to clean due to the large variety of chemicals produced. There did seem to be some agreement that glass-lined vessels, which are used for acidic mixtures, are more difficult to clean than the stainless steel vessels. This perception may be attributable to: (1) the types of chemicals in these mixtures, (2) the effect of acidic conditions on the glass surface, (3) properties of the glass that is used, (4) the fact that in the glass-lined vessels the walls also act as heat-transfer surfaces and vessels are not completely filled during cleaning, or (5) simply a perception that arises because chemical residues are easier to see on the dark-blue glass than on stainless steel. This question of the different cleaning properties of glass and stainless steel may have an impact on cleaning waste minimization, and is worthy of further investigation.

The personnel did not feel that improper cleaning prior to a product switch-over was a major cause of poor-quality product. In fact, because of ever-rising product quality standards, it was admitted that many vessels are probably over-cleaned to prevent product cross-contamination.

In general, the personnel interviews revealed that the established cleaning practices do produce consistently high-quality product. They also indicated, however, that there is a lack of understanding of the vessel cleaning process. Given the current level of understanding, any further optimization of vessel cleaning for the purpose of waste minimization would be difficult.

Production Records

To further explore the relationship between cleaning and product quality, a database was constructed from the production and product quality records for one group of 26 batch reactors during a one-year period. The database was used to determine in which unit each off-quality batch was processed, and what material was processed in the unit immediately beforehand.

A somewhat surprising result emerged from the analysis. An off-quality batch was preceded by an on-quality batch of the same material 96% of the time; in the other 4% a different chemical had been made in the reactor. When a different chemical is manufactured in the previous batch, the possibility exists for cross-contamination due to inadequate cleaning, and hence off-quality product. However, many other factors could also contribute to the rejection of a batch on the basis of specifications. With the same chemical preceding an off-specification batch, the issue of cleaning may have very little impact on the objective of high percentage product quality. With this distribution (96%/4%), it appears that vessel cleaning can be better studied through the direct observation of plant operations rather than the analysis of data on poor product quality. Thus, the database produced no substantial evidence that insufficient cleaning was causing a significant fraction of poor-quality product. It could even be postulated that the vessels may be over-cleaned.

In an attempt to quantify the amount of solvent used and the time consumed in reactor cleaning, a database was constructed from the cleaning records for a one month period. For the 43 reactor cleanings in the period studied, information was gathered on the material produced in the vessel prior to cleaning, the solvent used for cleaning, the time spent at reflux, and the volume of solvent used for each cleaning step. The analysis produced the results shown in Table I.

A histogram indicating the distribution of total reflux times for each unit cleaning is shown in Figure 1. The reflux times have a wide distribution, with cumulative reflux times ranging from 0 to 64 hours. Although the average total reflux cleaning time is 10 hours, the median is only 5 hours.

TABLE I

Summary of 3.8–11.4 m^3 reactor cleaning records

	Average	Range
Amount of solvent used in a vessel for a single cleaning step	2.5 m^3	1.1–8.3 m^3
Percentage of total reactor volume introduced in a single cleaning step	32%	7–100%
Total number of hours spent at reflux per cleaning	10 hours	0–37 hours
Total amount of solvent used per vessel cleaning	5.0 m^3	0.76–28.2 m^3
Number of cleaning "batches" required per vessel	2.0 batches	1.0–7.0 batches

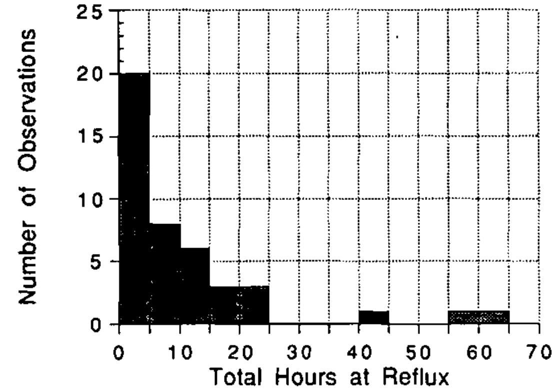

FIGURE 1 Histogram of reflux cleaning times.

Vessel Cleaning Data

Cleaning dynamics were observed by withdrawing solvent samples from several reactors at various times during cleaning. The samples were later analyzed for residue content by gas chromatography. Knowing the amount of solvent charged to the reactor allowed the total amount of residue removed from the vessel walls to be calculated. This was then plotted as a function of time to produce a "cleaning curve".

Figure 2 shows a cleaning curve from a stainless steel reactor which was soiled with an *n*-carboxylic acid residue. The vessel was cleaned by refluxing with denatured ethanol. The most striking feature of this plot is that the amount of residue in the solvent appears to have reached a maximum after the first two hours of cleaning. Since the reactors typically take about 30 minutes to reach reflux, this implies that the bulk of the cleaning occurred within approximately the first 1.5 hours of refluxing.

Figure 3 shows data from the cleaning of a dryer containing residue from a high-molecular-weight photographic chemical, using acetone as the cleaning

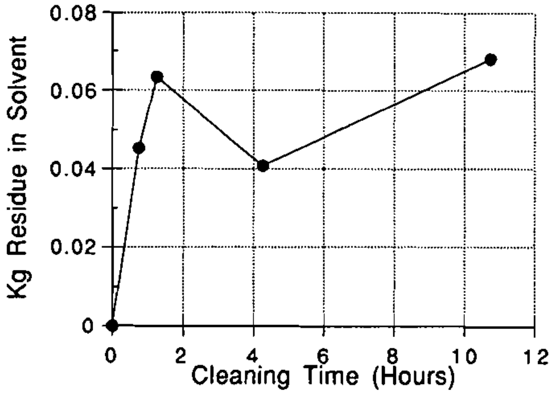

FIGURE 2 Cleaning of *n*-carboxylic acid from SS reactor using ethanol.

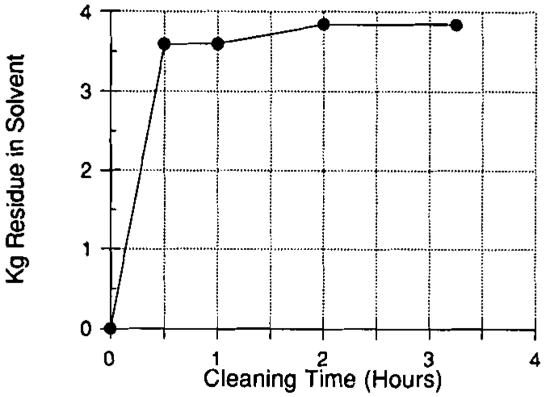

FIGURE 3 Cleaning of photographic coupler from SS dryer using acetone.

solvent. It should be noted that dryers take longer to heat than reactors, because they use tempered water rather than steam as the heat source. As noted above, dryers are not heated to reflux. In Figure 3, the first three samples were taken at essentially ambient temperature (25°C), as heating was not started until 30 minutes into the cleaning. The fourth point was taken at approximatley 30°C, and the fifth point at approximately 35°C. The same trend in the cleaning curve is seen for the dryer as was seen with the reactor, except that in the dryer it appears that essentially all of the cleaning occurred in the first 30 minutes.

CONCLUSIONS

The interactions with the operating personnel supported the main finding from the literature survey. That is, although empirical practices have been used successfully for many years, there is a lack of fundamental understanding of cleaning which prevents further optimization with respect to waste minimization. Because the exact requirements to produce a sufficiently clean vessel are not understood, an over-compensation is required to assure product quality. This over-compensation produces an amount of waste which could be reduced if the cleaning process were better understood. This study is a first step toward increased understanding of the cleaning process, and some possible improvements have been identified.

The two aspects of the reflux cleaning process which could be readily changed to minimize the amount of waste generated are the reflux time and the amount of solvent used. The time spent at reflux directly influences the condenser vent losses and the available reactor time for production. The steam consumed by unnecessary refluxing is also a source of waste, as steam generation requires the burning of fossil fuels. The amount of solvent used has a direct impact on both the energy required for solvent recovery and the amount of liquid capacity needed in solvent recovery.

The database developed in this study showed little direct correlation between off-specification product rejections and cleaning practices. From this it is inferred that the vessels are cleaned adequately, and perhaps more intensely than necessary. Information gathered in the cleaning sampling experiments suggests that the median of 5 hours at reflux exceeds the time required for the residue to desorb or reach a steady-state concentration. These observations indicate that in some cases reflux time could be shortened. As noted above, this would reduce the amount of cleaning solvent vent losses and steam usage, and increase the available time for production.

Reducing the amount of solvent used for cleaning could be accomplished in two different ways. The first would be to investigate alternate cleaning methods which require less solvent per reactor. For instance, a reactor might be cleaned with a spray nozzle using significantly less solvent. The second way to reduce the solvent usage is to reduce the number of times a reactor is filled during each cleaning. This could be accomplished through improved understanding of residue removal followed by inclusion of specific, effective cleaning procedures tailored for each product rather than relying on general cleaning procedures and overcompensation. These cleaning procedures should originate with the product development engineer or scientist who is familiar with the chemistry of the material.

CURRENT WORK

The research described above represents a new approach to the study of waste minimization because a graduate student was employed by an industrial concern solely to allow the observation of actual industrial cleaning practices. A research project was developed interactively with the company to provide pragmatic direction to the fundamental research. The study is focused on reflux cleaning dynamics, and seeks to determine how vapor condensation influences residue removal by a flowing liquid. The ultimate goal is to determine if there is an advantage, based on the fundamentals of heat and mass transfer, to reflux cleaning over other methods (e.g. spraying, filling-and-draining, circulating liquid) which do not expose the residue to condensing vapors. The results of this study will be presented in future papers.

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