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Potential strategies for process control and monitoring of stabilization of dairy wastewaters in batch aerobic treatment systems

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Abstract

This research was conducted to study the relationships between pH, dissolved oxygen (DO), and oxidation–reduction potential (ORP) during low-intensity aeration of dairy wastewaters and to determine potential strategies for monitoring and/or control of this treatment process. The results of this study ascertained that, close to the detection limit of commercially available DO probes (0.1 mg/l), DO is a poor indicator of the oxidation–reduction status of the dairy wastewater during this treatment processes. All the three parameters (ORP, DO, and pH) displayed features defining stabilization of the wastewater and hence all three can be used singly or in combination to monitor and/or to control this treatment process. The study also established strong linear relations between ORP and the log of DO; manifest in the high-correlation coefficients of 0.98 and 0.95 at the aeration rates of 0.067 and 0.034-l [air] l^{-1} [manure] min^{-1}, respectively. The latter observation confirms the higher sensitivity of ORP over DO at very low-oxygen levels; a fact which indicates the superiority of ORP in the monitoring and control of oxidation–reduction status of the wastewater close to DO detection limit. Finally, both total volatile solids (TVS) and chemical oxygen demand (COD), which are common measures of wastewater stabilization; correlated well with pH, DO, and ORP during the entire treatment process. However, because the measurements of DO are erratic close to the DO detection limit, and because ORP and pH measurements are much more consistent than the DO in the entire range of treatment, these two parameters will be more suitable for monitoring and control especially of extended aeration treatments.

Keywords: Oxidation–reduction potential; Dissolved oxygen; pH; Dairy wastewater; Stabilization; Limited aeration

1. Introduction

Oxidation–reduction potential (ORP) is a standard measure of the activity of electrons involved in oxidation reactions in aqueous environments. Numerous biological processes such as degradations of organic matter are essentially oxidation–reduction processes and past research has shown good correlations between biological activity and electrode potential [1,2]. An oxidation–reduction process is basically achieved by electron(s) transfer from the substance being oxidized (referred to as reductant) to the substance being reduced (referred to as oxidant). For all intents and purposes, a system's ORP is, therefore, indicative of the degree of oxidation–reduction process and ORP becomes the rational choice parameter for control and monitoring of such oxidation–reduction processes. In practice, however, ORP and dissolved oxygen (DO) are the two parameters widely used in the control and monitoring of aeration processes, which are also fundamentally oxidation–reduction processes [1–3,6–8,10–15]. Recently, pH has also been used successfully for online control of similar activated sludge processes mostly in combination with ORP [3–5,35–40].

In most aerobic biological systems, such as nitrification processes, oxygen is the requisite oxidant and the presence of DO at specific levels is a necessary condition for these processes to proceed unimpeded. In principle, therefore, one would be inclined to use DO as the parameter of choice to control and/or monitor these kinds of processes. However, although advanced electrochemical measurement of DO has recently tremendously improved the reliability of DO measurements, it is not always easy to implement aeration control systems based on DO measurements [6]. According to these researchers [6], choosing where to place the sensors in an aeration tank often requires trial and error and success is not guaranteed. In addition, the performances of DO probes are

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quite erratic near their detection limits and ORP measurements have been found to be better indicators of oxidation status close to the DO detection limit than the DO measurements [7]. In further support of this phenomenon, other researchers have reported linear relationships between ORP and the log of DO, which basically denote superior sensitivity of ORP over DO measurements at low-oxygen levels [8–13]. This sensitivity of ORP and the advanced development of equally reliable measurement methods of ORP thus offer a preferably more accurate alternative to determine oxidation status at low-DO levels than using DO measurements.

The ORP as a measure of the oxidative state in aqueous systems has been well demonstrated as a useful tool for indication of the biological state of systems. Use of ORP measurements in the treatment of municipal wastewaters is considered a better method than the DO measurements [1]. Charpentier et al. [6] used ORP as a control parameter to optimize the aeration of a pilot-scale plant and reported good correlations between the ORP and effluent total Kjeldahl nitrogen and nitrate. In other aerobic treatments, measurable changes in ORP have provided a good general indication of carbon oxidation and the degree to which waste stabilization has progressed [2,14,36]. The control of aeration by measuring the ORP of activated sludge has shown this type of regulation to be attractive in that it can optimize energy costs and the removal of carbonaceous and nitrogenous pollutants not to mention that this method is less expensive and easier to implement than using DO [15]. However, one drawback with ORP as a monitoring tool of oxidation status stems from its very nature of its measurement. The ORP measurements are made by determining the potential difference between an inert electrode and a reference electrode which may therefore, be affected by oxides and sulfide coatings on the electrodes [16,17]. In addition, many process products are not sufficiently electroactive to impose potentials on the inert electrode resulting in underestimation of the total potential difference [18]. These inherent problems of the ORP measurements often complicate its wider use as a control parameter in biological wastewater treatment systems. On the other hand, the pH of biological systems responds to microbial reactions, and hence, the pH status may also provide a good indication of the ongoing biological reactions [3–5,19,36,37]. For example, a decrease in pH in a nitrification process would be indicative of effective nitrification processes. Peddie et al. [12] also notes that ORP is analogous to pH in many ways and that in real-time, ORP response in different systems is empirically congruent to pH response in the systems.

Low-intensity aeration for stabilization of livestock wastewaters is a common practice to address the problem of ammonia and hydrogen sulfide stripping from these wastewaters during aerobic treatment with high-airflow rates [20–24]. In addition, when the objective of an aeration treatment of livestock wastewater is the reduction of organic loading rates into secondary treatment systems such as lagoons as opposed to complete stabilization of livestock wastewater; low-rate, low-intensity or limited aeration has been recommended for economical reduction of odor and other gaseous emissions [21–23,33,34]. In most cases, these treatment processes do not produce detectable DO levels because the little oxygen entering the reactor-system is consumed almost instantly. The objective of this study was to determine the relationships amongst pH, DO, and ORP during low-intensity batch aeration systems of dairy wastewaters and to determine suitable parameter(s) for monitoring the wastewater stabilization and/or for control of these kinds of treatment processes.

2. Methods and materials

2.1. Reactor system and instrumentation

This study was conducted in clear cylindrical Plexiglas reactors; 41 cm tall, 12.7 cm in diameter, with an operating volume of 4 l of liquid. To aerate the wastewater, a positive pressure air pump (Model DOA-P104-AA, Gast MFG Corp.) was used to introduce air into the reactor-system through a fine-bubble air diffuser with a standard oxygen transfer efficiency (OTE) of 5.0–7.6 kg O_2/kWh (Model 7″ standard FlexAir Disc Diffuser, Environmental Dynamics Inc.), located at the bottom of each reactor. Variable area flow meters (Catalogue #C-32460-40, Cole-Parmer Instrument Company) were used to regulate and maintain the flow of aeration air in each unit. Airflow rates of 0.067 and 0.034-l [air] l^{-1} [manure] min^{-1} (designated as R1 and R2, respectively in this article) were maintained throughout the study period in reactors 1 and 2, respectively.

The DO of the dairy wastewater was determined using DO-probes (Catalogue #C-53202-00, Cole-Parmer Instrument Company) and a DO-meter/controller (Catalogue #C-01972-00, Cole-Parmer Instrument Company). Both pH and ORP were measured using a combined pH-ORP meter (Orion Model 810, Orion Research Inc.). Probes to monitor both pH (Catalogue #C-05993-70, Cole-Parmer Instrument Company) and ORP (Catalogue #C-27001-62, Cole-Parmer Instrument Company) were calibrated, installed and retained in the reactors throughout the study period.

2.2. Dairy wastewater, sampling and analyses

Dairy wastewater was obtained from the equalization tank in the Knott Dairy Farm (Washington State University Dairy Research Facility). The manure collection system in this Facility is primarily flushing using supernatant water drawn from a secondary lagoon. To avoid clogging the laboratory reactor systems in our experiments, the dairy wastewater was screened through a 2.5 mm sieve. Pertinent characteristics of the dairy wastewater used in this study (see Table 1) indicate typical characteristics of wastewater from similar flush systems. Prior to filling the reactors, the wastewater was thoroughly mixed. Each reactor was then filled with 4 l of this well-mixed wastewater ready for the aeration treatment. Prior to commencing the aeration treatment, however, the wastewater in both reactors was again thoroughly stirred using a recirculation pump (Model 2E-38N, Little Giant Pump Co.) and a sample of approximately 100 ml was drawn from corresponding side ports in each reactor for laboratory analyses. Similar sampling was done every day from the same ports for the duration of the study.

Analyses were done immediately after the sampling whenever possible and if not, the samples were frozen for future analyses. Before conducting the analyses, frozen samples were allowed to thaw and equilibrate with the room

Table 1
Pertinent characteristics of the raw dairy wastewater used in this study ($n = 3$)

Total solids, TS (g/l)	11.02 ± 4.70
Total volatile solids, TVS (g/l)	6.97 ± 3.93
pH	7.78 ± 0.00
Chemical oxygen demand, COD (g/l)	13.75 ± 5.91
Total Kjeldahl nitrogen, TKN (mg/l)	1027 ± 21
Total ammoniacal nitrogen, TAN (mg/l)	651 ± 6

temperature. The chemical oxygen demand (COD) was measured using the standard ampule method [25]. The analyses of total volatile solids (TVS), total solids (TS), total Kjeldahl nitrogen (TKN), and total ammoniacal nitrogen (TAN) when and where necessary were performed according to standard methods [26]. The pH, ORP, and DO of the reactor contents in both reactors were recorded more frequently (seven times at 0, 1, 2, 5, 8, 13, and at the 24th hour) during the first day of treatment and once a day (at 11:00 a.m.) for the remainder of the study period.

3. Results and discussion

3.1. Changes in ORP, DO, and pH with time

The changes in ORP, DO, and pH with time of treatment at the two aeration rates (R1 and R2) during the treatments are presented in Fig. 1. It is evident from trends displayed in this figure that the responses of all three parameters with time were similar at the two aeration rates in every respect except perhaps in the observed absolute magnitudes of the responses. The observed ORP response with time during this treatment agrees with observations made in previous related research. Jenkins and Mavinic [27,28] observed that in a given system, the pattern of ORP in particular is reproducible even if the response varies between systems or within a system over time. In the present study, during the period the DO remained close to zero (or close to the detection limit of commercially available oxygen probes), it was impossible to discern distinctly the oxidation status of the wastewater from the measurements of DO only because of low sensitivity of the DO-measurements. This unreliability of DO measurements at low-DO levels has been previously documented by other researchers in other similar wastewater treatment systems [8,9,11–13,36]. In contrast, however, the ORP and pH measurements were comparatively more sensitive and thus more differentiated in the low-DO levels than the DO measurements. Therefore, using either the ORP or pH measurements, oxidation–reduction occurring in the wastewater can accurately be quantified and, therefore, if control or monitoring of the process are desired at these low-DO levels, either of these measurements would be more preferable.

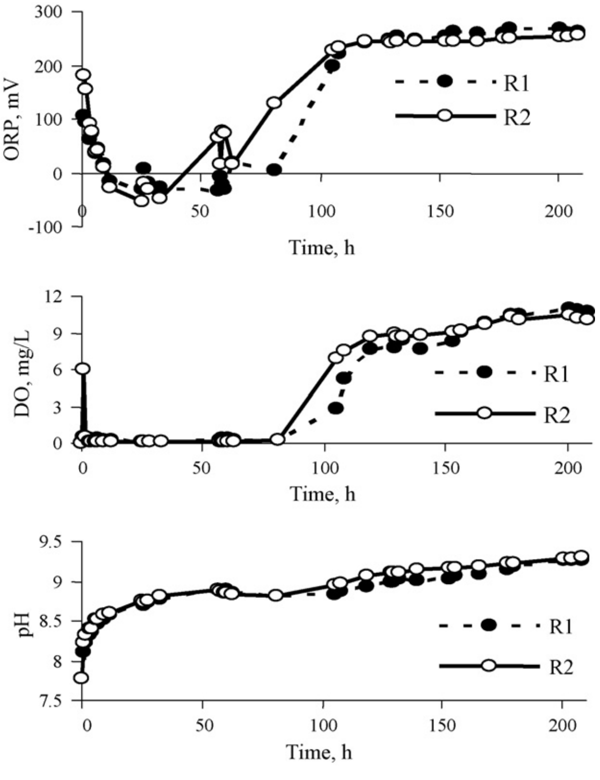

Fig. 1. Changes in ORP, DO, and pH with time during aeration treatment.

The observed decline in ORP represents the more active microbial degradation of wastewater or the consumption of the more easily degradable organic matter in the wastewater. This is sometimes referred to as the fast-rate degradation stage in the organic matter degradation kinetics [29,30,41]. The end of the fast-rate oxidation stage is indicated by a rapid rise in the ORP of the wastewater, which is also marked by a parallel step-rise in the DO. The end of fast-rate is the beginning of the slow-rate oxidation/degradation and marks the slowing down of microbial activity most probably because the more recalcitrant organic matter is the one being consumed. In this study and according to information presented in Fig. 2, it took 4 days to get to this point at which point COD degradations stood at approximately 51 and 47% of COD at the higher and lower aeration rates, respectively. Using the degradation equations obtained using linear regressions; it was projected that the ultimate COD degradations of approximately 70% would occur in approximately 9 and 10 days at the higher and lower aeration rates, respectively. In other words, the same treatment period time would be required to remove approximately 20% of the remaining degradable organic matter in the slow-rate regime compared to reduction of the initial 50% in the fast-rate regime. On the pH trend-lines, this stabilization point is marked by slight dips in pH in form of shallow 'valleys'. For practical purposes, this point then defines the critical wastewater stabilization point that can be used to control end of the treatment cycle. Treated effluent removal and filling of the reactor with a fresh batch of raw wastewater can be effected at this point in succession. Clearly therefore, these significant point changes in both ORP and DO at the end of fast-rate regime and beginning of slow-rate rate regime can accurately be used to control end and beginning of low-intensity treatment processes cycles. The slight dip (not significantly distinct) in pH at this point though theoretically usable could pose technical challenges in the use of pH in the control of this treatment process cycles.

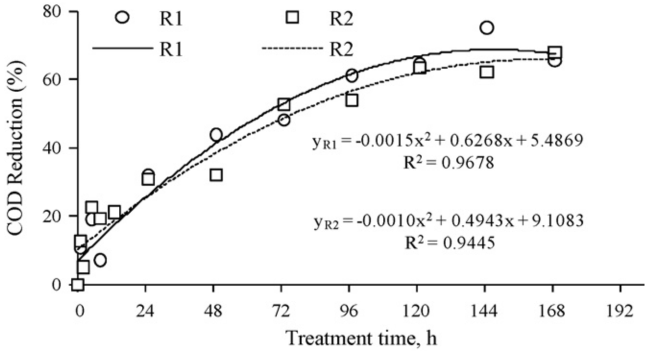

Fig. 2. Reduction of COD during 8 days of low-intensity aeration treatment.

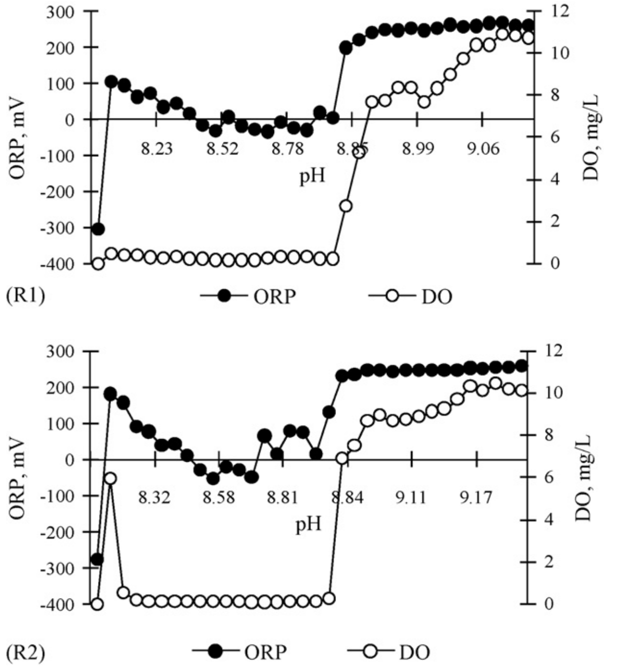

Fig. 3. Changes in ORP and DO with pH at the two aeration rates (R1 and R2).

3.2. Relationships between ORP and DO with pH

The changes in ORP and DO with pH at the two aeration rates (R1 and R2) are shown in Fig. 3. Although ORP and DO does not necessarily respond to the changes in pH the two parameters seem to respond to the pH in the same way they respond to treatment time (see Fig. 1). In other words, the trends of both ORP and DO with pH is evidently the same trends with time during the aeration. This observation has recently been noted in other similar treatment systems and has been the basis of suggested use of pH as another control parameter for oxidation–reduction treatments [3–5].

Based on the observations made in the responses of both ORP and DO with the changes in pH, regression analyses between ORP and pH and between DO and pH were further conducted individually. In addition, separate analyses were performed for the two distinct oxidation stages: fast-rate and slow-rate degradations. As noted earlier, the fast-rate regime represents the oxidation of the readily degradable organic matter while the slow-rate regime represents the oxidation of the more recalcitrant organic matter. The results of the linear regressions (in the form: $y = ax^2 + bx + c$) between ORP and pH during the treatment are presented in Fig. 4, while those of the linear regressions between DO and pH are presented later in Fig. 5. The coefficients of determination (R^2) obtained between ORP and pH ranging between 0.90 and 0.96 indicate that the ORP status could accurately be predicted from pH values during this type of treatment in both the fast-rate and slow-rate regimes. In other words, the aeration treatment resulted in measurable changes in the two variables that could be used interchangeably to parameterize the oxidation status of the dairy wastewaters during the low-intensity aeration treatments.

It is evident; however, that different relationships should be used for each of the two different regimes. In the fast-rate degradation regime, the ORP decreases with increase in pH irrespective of aeration rate (i.e. the derivative of ORP with respect to pH, d(ORP)/d(pH), is negative), while in the slow-rate degradation stage the ORP increases with increase in pH (i.e. the d(ORP)/d(pH) is positive) and eventually asymptotes. The point at which d(ORP)/d(pH) changes sign from negative to positive marked the end and beginning of the fast-rate and the slow-rate degradation regimes, respectively, thus identifying the dairy wastewater stabilization point. Since low-intensity aeration of wastewater is meant to remove the more easily degradable organic matter, this point as pointed before then marks the end of the treatment process cycle.

The relationships between DO and pH based on linear regressions presented in Fig. 5 show similar relationships to that observed earlier between ORP and pH. In general, within the fast-rate degradation regime, an increase in pH marks declining DO in the wastewater, while in the slow-rate degradation regime, an increase in pH marks an increase in DO in the wastewater. This phenomenon is very similar to the one observed previously between ORP and pH although the coefficients of determination for the linear regressions between the DO and pH were significantly lower in the fast-rate degradation regime (0.73 and 0.68 for R1 and R2, respectively) than in the slow-rate degradation regime (0.96 and 0.95 for R1 and R2, respectively). Again the point at which the derivate of DO with respect to pH, d(DO)/d(pH), changes sign from negative to positive marks the end and beginning of the fast-rate and the slow-rate degradation regimes, respectively, and consequently also identifies the wastewater stabilization point. The relatively lower coefficients of determination for the linear regressions in the lower regime are explained most probably by the erratic measurements of DO in this regime. In addition to

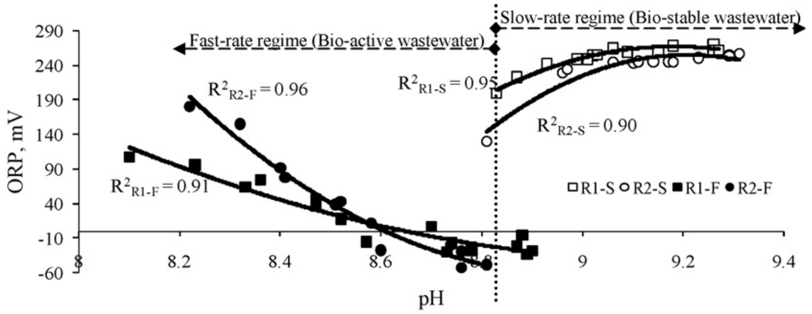

Fig. 4. Relationships between ORP and pH during fast-rate and slow-rate stabilization regimes.

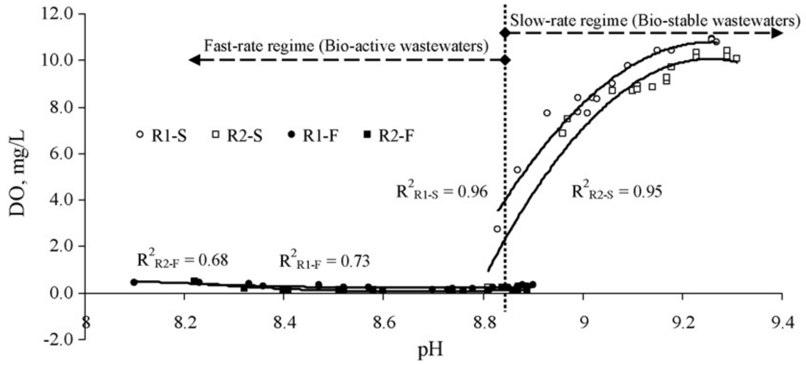

Fig. 5. Relationships between DO and pH during fast-rate and slow-rate stabilization regimes.

the significantly low values of DO in this regime, which are more susceptible to measurement errors these results further reveal the potential problem of using DO to monitor these treatment processes within the fast-rate degradation regime.

3.3. Relationships between ORP and DO

The relationships between ORP and the logarithm of DO obtained by linear regression analyses are shown in Fig. 6 at both aeration rates. These regression analyses confirm the earlier observations made about the erratic behavior of DO measurements in the neighborhood of oxygen detection limit of available commercially oxygen probes (or near zero). The relationship between ORP and DO is very strong as indicated by high-correlation coefficients of 0.98 and 0.95 in the higher and the lower aeration rates, respectively. This linear relationship between ORP and log of DO (as noted earlier) has been well documented by previous researchers [8,9,11–13], and indicates the sensitivity of ORP compared to DO measurements at low-DO levels. To be able to monitor precisely the oxidation status of the dairy wastewater during low-intensity aeration treatments especially close to DO detection limits, ORP would therefore, yet be the more preferable parameter than DO.

The slope of straight line graph defining the relationship between ORP and DO of 60 and 50 mV/Ln[DO] at the higher and the lower aeration levels is similar to previously reported values for activated sludge treatment processes [17,31,32]. Aeration treatment of dairy wastewater is thus similar to the activated sludge treatment process. This suggests that ORP can likewise be used in the control and/or monitoring of batch aeration treatment of dairy wastewater in the same way it is used in the activated sludge treatment processes.

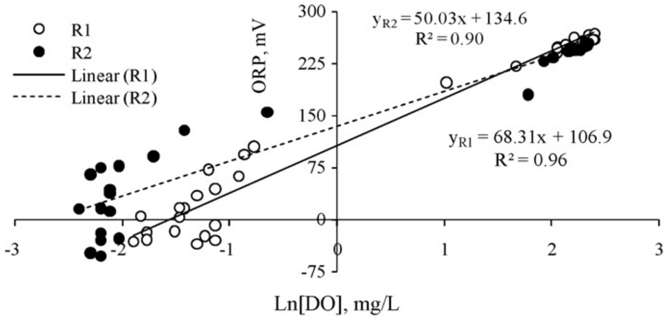

Fig. 6. Relationships between DO and ORP during aeration of the dairy wastewater at the two aeration rates (R1 and R2).

3.4. Wastewater stabilization: COD and TVS versus ORP, DO, and pH

Wastewater stabilization is commonly characterized by the chemical oxygen demand (COD), biochemical oxygen demand (BOD), or by total volatile solids (TVS) of the wastewater. These parameters are difficult to monitor in real time and, therefore, cannot be used to monitor and/or control wastewater stabilization in real time. The changes in COD and TVS as functions of ORP, DO and pH of the dairy wastewater are shown in Fig. 7. Reasonably good coefficients of determinations ranging between 0.69 and 0.93 were obtained between

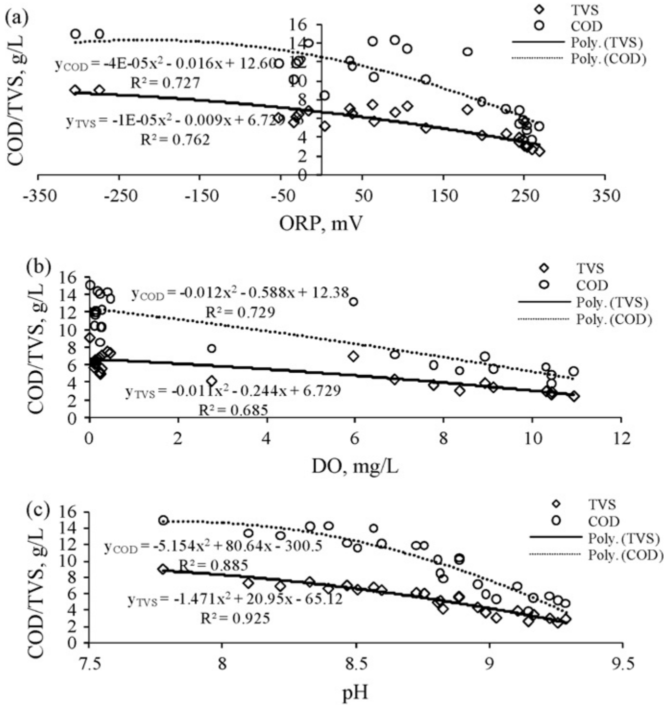

Fig. 7. (a–c) Relationships of COD and TVS of the dairy wastewater to all the three parameters: ORP, DO, and pH; during the treatment of the dairy wastewaters.

either ORP; DO; or pH with either COD or TVS. The best parameter for prediction of both COD and TVS during the entire treatment was pH (R^2 of 0.89 and 0.93 for COD and TVS, respectively); followed by ORP (R^2 of 0.73 and 0.76 for COD and TVS, respectively); DO (R^2 of 0.73 and 0.69 for COD and TVS, respectively), in that order. The erratic behavior of the DO measurements around zero is once again quite evident in these analyses as well in the data scatter plots. Li and Bishop [14] reported similar ORP and COD results from a closely related activated sludge wastewater treatment process. These researchers observed good linear relationship between ORP and COD that could be used to indicate quality of the effluent using the easier and quicker ORP measurements instead of tedious COD measurements.

It is evident from the results presented in the preceding paragraph that pH and ORP measurements are much more consistent than DO and hence may be the more suitable for monitoring and/or control of the wastewater stabilization during extended treatment processes. For extended low-intensity aeration, where additional degradation of the recalcitrant organic matter is desired, the control strategy suggested earlier may change. To optimize the hydraulic retention time to save on energy usage, a target volatile solids or COD residual levels could be identified and the corresponding ORP or pH control points computed from the regression equations of either pH or ORP. Our studies indicate that for low-intensity aeration treatments of dairy wastewater, an ultimate COD degradation of approximately 70% occur at approximately 9th and 10th days of treatment. Because of the variations in the influent characteristics, this reduction in COD could be used for control of these extended batch treatment systems for dairy wastewater using either pH or ORP. More rigorous experimentation would be needed before this number is adopted for extended treatment of dairy wastewaters.

4. Conclusions

Experiments were performed to study the relationships between pH, DO, and ORP during low-intensity aeration systems of low-strength dairy wastewaters and to determine suitable method(s) for monitoring wastewater stabilization and/or for control of these kinds of treatment processes. This study established that dissolved oxygen measurements are poor indicators of the oxidation–reduction status of the dairy wastewater during low-intensity aeration treatment processes near the detection limit of available oxygen probes.

All the three parameters (ORP, DO, and pH) display features that can be used to define stabilization of dairy wastewater during the treatment process. Both ORP and DO decreases gradually during the stabilization of the more easily degradable organic matter and increases rapidly thereafter before leveling off. On the other hand, the pH slightly dips at the point where stabilization of the wastewater occurs. All three parameters can thus be used singly or in combination to monitor stabilization of dairy wastewater and/or for control of these types of treatment processes. However, if used individually pH might pose more technical challenges than the individual use of either ORP or DO, while combinations of pH with either the ORP or the DO would offer better control strategies.

The linear relationship between ORP and the log of DO is strong as indicated by high-correlation coefficients of 0.98 and 0.95 at the aeration rates of 0.067 and 0.034-l [air] l^{-1} [manure] min^{-1}, respectively. This confirms the sensitivity of ORP measurements compared to DO measurements at very low-oxygen levels and hence ORP superiority in the monitoring and control of oxidation–reduction of dairy wastewater at these low-DO levels.

Statistically, both TVS and COD correlated well with pH, DO, and ORP measurements during the entire treatment process (both the fast-rate and slow-rate degradation regimes). However, according to the data, measurements of DO are much more erratic near the detection limit of DO. The pH and the ORP, on the other hand are much more consistent than the DO and hence either would be more suitable for monitoring the stabilization during extended low-intensity treatment processes than DO.

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