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Mathematical modelling of real-time control system for industrial wastewater management

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ABSTRACT

The composition of wastewater produced in industrial processes can vary dynamically and often randomly. Immediate intervention is necessary to preserve the water quality before any parameter of the wastewater exceeds the threshold limit; therefore, the parameters of the wastewater must be determined in real time. Direct laboratory methods have long lead times; indirect methods (refraction, pH, conductivity, turbidity, etc.) would be suitable as they measure indirect values, so correlations must be found between the measured values and the component(s) for analysis. These correlations are often stochastic in nature having a standard deviation of the same magnitude as the measured values. This paper deals with pharmaceutical wastewater with a high sucrose content, the online measurement of its organic material content characterised by chemical oxygen demand (COD_{cr}), refraction and resistance. It finds a correlation between the COD_{cr} and resistance-corrected refraction (r^*). To reduce the standard deviation, the concept of cumulative COD_{cr} is introduced. Concentration limit values could be determined to compare with the cumulative COD_{cr} values calculated continuously during the fill up of the wastewater tank. This means that the treatment with the wastewater (into the drain, dilution and transfer) can be decided. The validity of the model is checked by trial calculations based on a high number of measurements.

Keywords: Industrial wastewater; Mathematical model; Refraction; Chemical oxygen demand

1. Introduction

Unlike slow-changing communal wastewater, the composition of wastewater produced in industrial processes can vary dynamically and often randomly. Intervention is necessary (generally immediately) to preserve the water quality before any parameter of the wastewater exceeds the threshold limit [1–4]. To achieve this, the parameters of the wastewater must be determined in real time [5]. Despite being able to provide correct results, traditional laboratory methods are not suitable due to their long lead times. Online analysers (total organic carbon, phosphate, etc.) also produce direct results, but they are expensive and require significant maintenance, and the time needed for detection can be longer than desired. Operating them requires special knowledge [6,7]. The remaining methods are the indirect ones (such as

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refraction, pH, conductivity, turbidity, etc.) [8–10]. Their advantages include immediate measurement and evaluation, relatively low cost, no particularly special knowledge is required, and they are easy to maintain and calibrate.

Besides their advantages, however, they have numerous disadvantages. The most important is that they measure indirect values, so correlations must be found between the measured value(s) and the analysed component(s). These correlations are often stochastic in nature, as the results from online measurement can be influenced by other parameters [11–16]. Applying them does not mean laboratory measurements can be totally left out, but these do not have to be real time (they generally have to be carried out during calibrations and official checks).

Due to the characteristics of wastewater, many measuring methods can be applied. In this paper, we deal with a rather special, but important case, a pharmaceutical company's wastewater with high sucrose content.

2. Measurements

The main part of the dissolved or solubilised compounds of the water comes from washing the pharmaceutical instruments of the factory when changing manufactured medicines. This is a diluted coating solution for pills, and mainly contains sucrose, as well as citric acid or its salts, surfactants and talcum powder. The base conductivity originates from the salinity of the water system.

The basic principle of our method is that the concentration of a sucrose solution can be measured by refractometrics in real time, so we can expect a direct relation between the refraction (the sugar content of an aqueous solution, BRIX) and the chemical oxygen demand (COD$_{cr}$) values [3,4,8,10–16]. Since the refractometric measurement can be executed online, this measurement method can be the basis for this control system.

The first step was to calculate the relation between the BRIX and COD$_{cr}$ values for different sucrose solutions with precise laboratory measurements. For the pure sugar solution, there is a direct relation between COD$_{cr}$ (k) and BRIX (r refraction), which can be expressed with the following equation (Fig. 1):

$$k = \alpha \cdot r \tag{1}$$

where α = 12,500 mg/L·BRIX and R^2 = 0.9950.

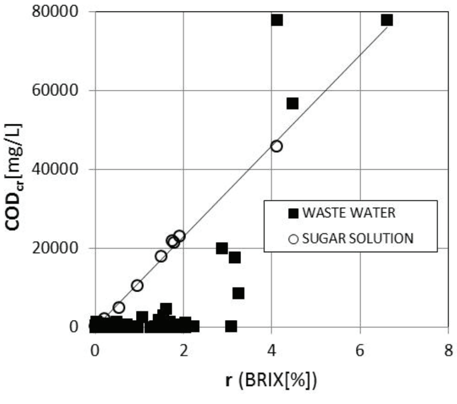

Fig. 1. Chemical oxygen demand (COD$_{CR}$) as a function of refraction (r) for industrial wastewater containing sugar and pure sugar solution.

A high number of real-time, online measurements were taken to calculate the sucrose equivalent r (BRIX [%]), the conductivity Ω (mS/cm), pH and temperature T (°C) values.

The optical lenses of the online instrument must be cleaned regularly (washing). This was done using citric acidic water. After washing, the equilibrium set in some 60 s later (as shown by the restoration of pH values), so the data obtained within 60 s of the washing was deleted. Similarly, the samples with obvious measurement problems and identical, consecutively measured data were also deleted.

In some 150 samples, the COD$_{cr}$ was determined under laboratory conditions. From the data – and taking the above into consideration – 120 samples were selected with the associated online data mentioned above. These measurements can be regarded as independent.

For the wastewater, we used Eq. (1) correlation directly, α = 5,663 mg/L·BRIX and R^2 = 0.4315. However, there is no correlation between COD$_{cr}$ and conductivity (R^2 = 0.0011); a higher correlation coefficient can be given (R^2 = 0.6183) with the introduction of conductivity, that is:

$$k = \alpha \cdot r + \beta\Omega = \alpha r^*, \tag{2}$$

where $r^* = r + \frac{\beta}{\alpha}\Omega$ is the corrected refraction value (a corrected BRIX value is indicated with BRIX*); β is the questionable coefficient (β = –0.1 mg·cm/L·mS). Unfortunately, the correlation coefficient was lower than the previous value, $R^2 \cong 0.8$ ($R \cong 0.9$), and only slightly exceeds the value where the correlation is still acceptable, $R^2 \cong 0.36$ ($R \cong 0.6$).

3. Model

3.1. Probability model

Since wastewater can contain other components besides sugar having an effect on the BRIX values associated with several COD$_{cr}$–s, the latter can be regarded as a random variable, κ where:

$$M(\kappa) = k \quad \text{and} \quad D^2(\kappa) = M\left(\left[\kappa - k\right]^2\right) = \sigma^2 \tag{3}$$

are (multitude) mean value and standard deviation, respectively. In this case, the relation between COD$_{cr}$ and BRIX will be stochastic.

The correlation may be increased by applying a logistic model. The mean value of k using this approximation will be a polynomial of degree q as follows:

$$\ln\frac{k}{k_\infty - k} = a_0 + a_1 r^* + \ldots + a_q r^{*q} + \ldots, \tag{4}$$

where a_q–s are the mean values of polynomial coefficients as random variables; k_∞ is the asymptote of k ($k \to k_\infty$ if $r^* \to \infty$). The standard deviation as the function of r^* similar to Eq. (4) will be as follows:

$$\ln\frac{\sigma}{\sigma_\infty - \sigma} = b_0 + b_1 r^* + \ldots + b_q r^{*q} + \ldots. \tag{5}$$

where b_q–s are the standard deviations of polynomial coefficients, and σ_∞ is the asymptote of σ as above.

3.2. Mathematical method

The values of polynomial coefficients are calculated by variations of parameters [8]. Using the mean square error (MSE) [17–19], the function of parameters needs to be constructed for the mean value of Eq. (4), as can be seen below:

$$\mathrm{MSE} = \sum_{i=1}^{n} [\ln \frac{x_i}{k_\infty - x_i} - \sum_{q=0}^{\infty} a_q r_i^{*q}]^2 \rightarrow \mathrm{MIN} \tag{6}$$

where x_i and r_i^* are the measured values of $\mathrm{COD_{cr}}$ and corrected refraction value, r^*, from Eq. (2) at the ith sample.

The measured (calculated) values of the (square) standard deviation:

$$d_i^2 = [x_i - k_i]^2 \tag{7}$$

is the square of the difference of the measured (x_i) and calculated mean value (k_i) from Eq. (4) at the ith sample. The standard deviation parameters can be given similarly to Eq. (6):

$$\mathrm{MSE} = \sum_{i=1}^{n} [\ln \frac{d_i}{\sigma_\infty - d_i} - \sum_{q=0}^{\infty} b_l r^{*q}]^2 \rightarrow \mathrm{MIN} \tag{8}$$

For the mean value of Eq. (6), the correlation coefficients were also calculated by the degree of the polynomial $q = 1$, $R^2 = 0.8138$ and $q = 2$, $R^2 = 0.9481$. This latter exceeds the "good" correlation coefficient ($R^2 > 0.8$). For the higher polynomial degree, the R^2 value was enhanced further, but the σ_∞ approximated 0 because there were only a few measurements in the higher BRIX range, so there was no way to increase the degree of the polynomial. The mean values of polynomial coefficients are as follows: $a_0 = -6.05$, $a_1 = 1.09$/BRIX, $a_2 = 0.209$/BRIX^2 and $k_\infty = 72{,}000$ mg/L, and the standard deviation of those will be $b_0 = -4.42$, $b_1 = 1.38$/BRIX, $b_2 = 0.16$/BRIX^2 and $\sigma_\infty = 12{,}500$ mg/L.

3.3. Calibrating curves

Rearranging Eqs. (4) and (5), the calibrating curves that are dependences of k and σ on the corrected refraction of Eq. (2) will be as follows (Fig. 2):

$$k = k_\infty \frac{\exp(a_0 + a_1 r^* + a_2 r^{*2})}{1 + \exp(a_0 + a_1 r^* + a_2 r^{*2})} \tag{9}$$

$$\sigma = \sigma_\infty \frac{\exp(b_0 + b_1 r^* + r^{*2})}{1 + \exp(b_0 + b_1 r^* + b_2 r^{*2})} \tag{10}$$

The standard deviation σ is the same order of the mean value k. The $k \pm 2\sigma$ is the interval where the random variable κ has a probability of 95% in $P(k - 2\sigma < \kappa < k + 2\sigma) = 0.95$.

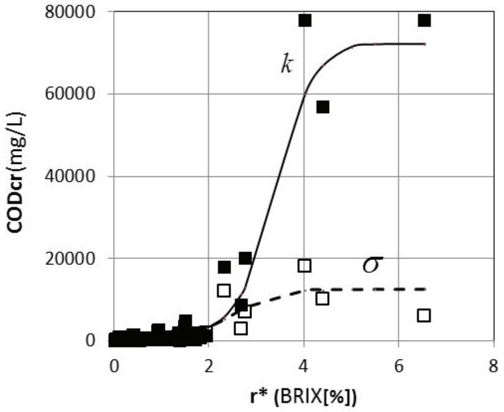

Fig. 2. The calibrating curves, the mean value (k) and standard deviation (σ) of $\mathrm{COD_{cr}}$ as the function of the corrected value of refraction r^*. The symbols are the measured values; the curves are from Eqs. (9) and (10).

The lower value is practically 0; therefore, the upper (calculated) value must be given. This upper limit (95% one-sided probability level) is as follows:

$$k^+ = k + 1.64\sigma \tag{11}$$

Each sample ($i = 1, 2, \ldots n$) has an equal amount of wastewater. Take a tank and start to fill it in until sample j ($i \le j$). The j/n means the fullness of the tank, the tank level ratio.

3.4. Cumulative $\mathrm{COD_{cr}}$

The $\mathrm{COD_{cr}}$ threshold for the wastewater that has to be run out into the drain is k_{LIMIT} (1,000 mg/L) that is $k^+ \le k_{\mathrm{LIMIT}}$. Since $k \cong \sigma$ in the most important range ($0 < r^* < 2$), k must be much smaller in Eq. (8) than k_{LIMIT} ($k < 400$ mg/L) so the $\mathrm{COD_{cr}}$ value that goes into the drain is much lower than permitted. The solution may be to decrease the standard deviation.

One way to do so is introducing the cumulative $\mathrm{COD_{cr}}$. The cumulative random variable to the jth measured value will be as follows:

$$K_j = \frac{1}{n} \sum_{i=1}^{j} k_i \tag{12}$$

where $i = 1, 2, \ldots j$, and n is the number of measurements belonging to the full tank.

In the case of independent samples, the mean value and standard deviation of cumulative $\mathrm{COD_{cr}}$ have been defined as follows:

$$K_j = \frac{1}{n} \sum_{i=1}^{j} k_i \tag{13}$$

$$\Sigma_j^2 = \frac{1}{n^2} \sum_{i=1}^{j} \sigma_i^2 \tag{14}$$

The upper limit of Eq. (11) will now be as follows:

$$K_j^+ = K_j + 1.64\Sigma_j \tag{15}$$

An additional limit can be defined by:

$$K_{j,LIMIT} = \frac{j}{n} k_{LIMIT} \tag{16}$$

If $K_j^+ < K_{j,LIMIT}$, then the average COD$_{cr}$ in the tank is less than the threshold k_{LIMIT} = 1,000 mg/L. In this case, the wastewater can be run out into the drain at any time (Fig. 3). Furthermore, there is another threshold limit for the cumulative COD$_{cr}$ from Eq. (16) ($j = n$ full tank), namely:

$$K_{MAX} = k_{LIMIT} \tag{17}$$

If $K_j^+ > K_{j,LIMIT}$ but $K_j^+ < K_{MAX}$, then the waste can be run out by dilution (watering).

If $K_j^+ > K_{MAX}$, it must be transferred (Fig. 3).

4. Results

4.1. Virtual tanks

Twelve virtual tanks were prepared from the data with 20 elements ($n = 20$). The two criteria were: firstly, the average COD would be around the critical 1,000 mg/L mark ($0 < r < 3$), and secondly, every piece of data would appear only once (independent data). This latter criterion is needed because independent measurements can also lead to the same data. Therefore, some 80 independent data items remained, from which 20 were chosen with different randomisation techniques (i = 1, 2, ... 20). The fullness of the virtual tank was marked j (j = 0, 1, 2, ... n), and the ratio j/n essentially meant the tank level in a volume proportion.

In a virtual tank, the measured cumulative values of COD$_{cr}$ corresponding to Eq. (13) will be as follows:

$$X_j = \frac{1}{n}\sum_i^j x_i \tag{18}$$

In Fig. 4, we see the calculated (K_j) and measured (X_j) values of cumulative COD$_{cr}$ and the upper limit of that K_j^+ at the number 1 tank (Table 1). In this case, K_j^+ has reached the K_{MAX} limit at value j_W so a decision must be made whether to let the wastewater into the drain diluted (watering), or to fill up the tank and transport it. The decision is partly economical, and partly legal and technical, considering that it means three run offs and two tanks of water are needed for the watering (Fig. 4).

In the table, the characteristic COD$_{cr}$ values of full tanks can be seen, namely the calculated and the measured cumulative COD$_{cr}$ values ($j = n$) K and X, furthermore the standard deviation and the upper limit of that Σ and K^+ complementing the rate X/K^+, the efficient estimation of which is 60%–70% on average. The model chi-square test has

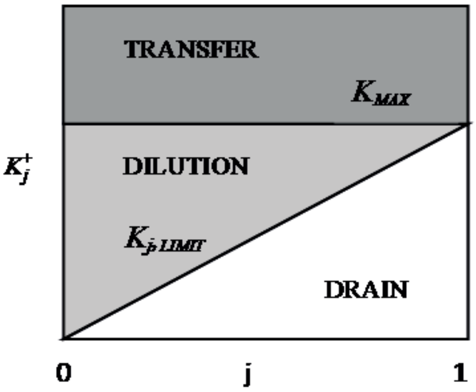

Fig. 3. Limit values of cumulative COD$_{cr}$ as the function of sample serial number (j) and scheme of operations connecting with them.

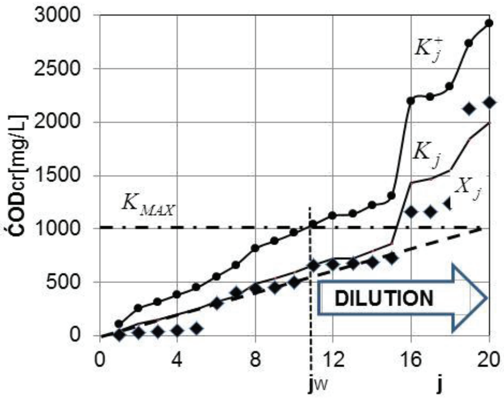

Fig. 4. Changes of the cumulative COD$_{cr}$ (K_j) and upper limit of that (K_j^+) plotted against the function of sample serial number (j) for tank 1. The symbols are measured values (X_j), the lines are calculated from the model.

Table 1
Cumulative COD$_{cr}$ characteristics of full tanks as the function of the serial numbers of virtual tanks

COD$_{CR}$ (mg/L)	1	2	3	4	5	6	7	8	9	10	11	12
X	2,118	236	1,125	845	1,511	622	683	912	1,146	825	442	1,965
K	1,996	244	1,012	1,005	1,370	503	605	886	915	953	412	2,148
Σ	695	106	385	390	486	245	274	362	412	365	135	731
K^+	3,136	418	1,643	1,645	2,167	905	1,054	1,480	1,591	1,552	633	3,347
X/K^+	0.68	0.57	0.69	0.51	0.70	0.69	0.65	0.62	0.72	0.53	0.70	0.59

been controlled [20] using data in the table. The number of cells is 5; the degrees of freedom is 4; the chi-square is 0.533 and the *p* value is 0.9702. The test has given a good fit between the measured data to the calculated values by the model.

4.2. Determination of optimum measurement number

In practice, we take n samples during the tank filling, in this case $j = n$ and $K_j = K$. As K is different from tank to tank, it can therefore also be regarded as a random variable K. The mean value of K for m tanks is:

$$M(K) = \frac{1}{m}\sum_{l=1}^{m} K^{(l)} = \bar{K} \tag{19}$$

where

$$K^{(l)} = \frac{1}{n}\sum_{i=1}^{n} k_i^{(l)} \tag{20}$$

is the mean value of $\mathrm{COD_{cr}}$ of lth full tank based on the Eq. (13); $k_i^{(l)}$ is the mean value of $\mathrm{COD_{cr}}$ of ith measurement in the lth tank (assuming the sample number is the same, n for all samples). As Eq. (20) is an integral sum, the mean value does not (really) depend on n ($n >> 1$).

The standard deviation of κ will similarly be as follows:

$$\bar{\Sigma}^2 = \frac{1}{m}\sum_{l=1}^{m} (\Sigma^{(l)})^2 = \frac{\mu^2}{n} \tag{21}$$

where, using Eq. (14), the sign in Eq. (21):

$$\mu^2 = \frac{1}{mn}\sum_{l=1}^{m}\sum_{i=1}^{n} (\sigma_i^{(l)})^2 \tag{22}$$

is a (double) integral sum, and it does not depend on n either. By enhancing the number of samples, $n \to \infty$, the standard deviation, $\bar{\Sigma}$, can be decreased for as long as the samples are independent. The question is: what is the optimum number of samples?

Let $x_i^{(l)}$ be the measured value of $\mathrm{COD_{cr}}$ at the ith sample in the lth tank. In this case, the measured cumulative values of that corresponding to Eq. (20) will be as follows:

$$X^{(l)} = \frac{1}{n}\sum_{i}^{n} x_i^{(l)} \tag{23}$$

X_l, equivalent to K_l, is an integral sum and therefore does not depend on n.

The measured square standard deviation equivalent to Eq. (7) can be written as follows:

$$s^2 = \frac{1}{m}\sum_{l=1}^{m} (X^{(l)} - K^{(l)})^2 \tag{24}$$

where both $X^{(l)}$ and $X^{(l)}$ do not depend on n, so it approximates the real (multitude) standard deviation and is independent from the number of samples.

The optimal number of samples n_{OPT} is the number of samples ($n = n_{\mathrm{OPT}}$) where Eq. (22) and Eq. (24) would be equal. An *F*-probe was used to check the equality of the two standard deviations. As we apply an upper approximation for the standard deviation ($\bar{\Sigma} > s$), the *F*-probe can be written as follows:

$$F = \frac{\bar{\Sigma}^2}{s^2} = \frac{\mu^2}{n_{OPT}}\frac{1}{s^2} \le F_{m-1,m-1}(1-\varepsilon) \tag{25}$$

where $F_{m-1,m-1}(1-\varepsilon)$ is the critical value for the m–1 degrees of freedom and the 95% probability level ($1 - \varepsilon = 0.95$). ($m \to \infty$ $F_{m-1,m-1}(1-\varepsilon) \to 1$). From this, the optimal number of samples is:

$$n_{OPT} = \frac{1}{F_{m-1,m-1}(1-\varepsilon)}\frac{\mu^2}{s^2} \tag{26}$$

4.3. Technical arrangements

Different technical arrangements can be produced based on the model [18,19,21] and an intelligent computer program, which controls the parameters and makes decisions both for the type of outlet and for the time. It is advisable to display the values on the computer screen with different markers, warnings and instructions on an easy to understand page.

5. Discussion

For the refractometric online analysis of the organic material content of industrial wastewater containing sucrose, the biggest problem is the high standard deviation of the samples. As the laboratory measurement of $\mathrm{COD_{cr}}$ content (k) is fairly accurate, the causes of the high standard deviation can be found in the refraction measurement (r) errors, namely:

- the measurement error of the refractometer;
- the effects of second compounds (e.g. citric acid etc.);
- the standard deviation due to inhomogeneity derived from samples not from the same place (sample was taken at the same time, but not from exactly the same place); and
- the measured BRIX values are higher than in reality because of the sucrose adhesion on the instrument.

The first three causes would result in symmetric standard deviations compared with the refraction of a pure sugar solution (r_{SUGAR}, Fig. 1). As the standard deviation is asymmetric to that (around 90%, $r > r_{\mathrm{SUGAR}}$), we assume that of the four effects, the adhesion may contribute most to the standard deviation.

With the introduction of cumulative $\mathrm{COD_{cr}}$, the deviation can be reduced, as in the case of independent samples, the square standard deviations are summed up. The standard deviation is thus inversely related to the square root of the number of samples. From the table, it can be seen that the difference between the estimated and measured values of $\mathrm{COD_{cr}}$ is much smaller (roughly a half, a third) than it was in the case of the real-time measurements, meaning a much better estimation of the upper limit.

The standard deviation can be decreased by raising the number of samples to the number of independent samples, which can be determined by a model. From the table data, μ = 5,630 mg/L, s = 416 mg/L, $F_{11,11}$ (0.95) = 2.82, from which the independent samples $n_{OPT} \cong 65$ nearly equal the data number (80), i.e., they can be regarded independent, as expected. Increasing the m value, the accuracy of approximation can be improved, n_{OPT} can be a bit higher.

The deviation is mainly caused by the adhesions. If we assume these happen randomly between two washings, the samples taken in each washing can be considered independent. This applied to the current samples.

6. Conclusions

The measurement of organic content (COD_{cr}) can be taken by combining refractometry and conductivity. The advantage of the method over direct methods is that it is much cheaper and much faster, and most importantly it makes immediate intervention possible. Its disadvantage is the inaccuracy, but this is important only around the critical value (1,000 mg/L). It means that the COD_{cr} content of the water discharge to the drain is some 35% lower than the permitted limit.

This value can be significantly decreased with a better measurement layout (more practical placing of refractometer), more effective washing but first and foremost by increasing the number of samples up to the independent samples. These can result in at least a 50% improvement, which means the 35% mentioned above can be lowered to 10%–20%. For the outlet COD_{cr}, it would mean 800–900 mg/L instead of 1,000 mg/L, which could be satisfactory in practice.

The model and the control system shown in this article offer a great, economical and environmentally friendly opportunity, but an additional checking system is needed to be able to monitor the correct functioning of the online instrument continuously.

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