Preprints of the 19th World Congress
The International Federation of Automatic Control
Cape Town, South Africa. August 24-29, 2014

Modelling of the ultrasonic disintegration of activated sludge

N. Lambert*, I. Smets, J. Van Impe**, R. Dewil***

**KU Leuven, Department of Chemical Engineering, Process and Environmental Technology Lab, Jan Pieter De Nayerlaan 5 – BE-2860 Sint-Katelijne-Waver - Belgium (e-mail: nico.lambert@cit.kuleuven.be).*
***KU Leuven, Department of Chemical Engineering, Chemical and Biochemical Process Technology and Control Division – W. de Croylaan 46 – BE-3001 Heverlee - Belgium (e-mail:ilse.smets@cit.kuleuven.be)*

Abstract: Ultrasonic treatment of waste activated sludge is one of the possibilities to reduce excess sludge production through the mechanism of sludge disintegration and cell lysis. In the past, several attempts have been made to model the process of solubilisation of the particulate volatile suspended solid part of the activated sludge (VSS) into soluble COD (sCOD). However, the focus of these models was predominantly on predicting an efficiency factor for the release of sCOD (Disintegration Degree, DD_{COD}) and provided no information on the release of nutrients and the instantaneous reduction of VSS. Moreover, often insufficient influential variables were included in the model equations, making the models only applicable on the training dataset of their own experimental research. This paper, therefore, seeks to build a simple model, which contains all influential input variables, that can predict not only the sCOD release but also the nutrients release (ortho-PO_4-P and soluble Kjeldahl nitrogen) and VSS reduction simultaneously. Therefore, in first instance, a Principal Component Analysis (PCA) is carried out on the input and output data matrix of obtained experimental observations that will be used as training data. In this way, certain correlated input variables and independent output variables can be removed from the model, in order to increase its simplicity and predictive nature. Then, the model is built on the basis of Partial Least Squares Regression (PLS-R) and a part of the observations is used to validate the predictive strength of the model.

Keywords: wastewater treatment modelling, ultrasonic disintegration, excess activated sludge reduction.

1. INTRODUCTION

Focused on considerably reducing the generation of excess sludge in activated sludge wastewater treatment plants, cell lysis and cryptic growth of microorganisms has been found very effective to reduce Waste Activated Sludge (WAS). Ultrasonication of WAS and RAS (Return Activated Sludge) is capable of destroying the flocs within their matrix of extracellular polymeric substances (EPS) to result first in much smaller flocs and afterwards in cell death and lysis (Xiaoxia Wang, 2010). Cell lysis is a process in which the cell disintegrates after which the organic, nitrogen and phosphorus constituents of the cell provide an autochthonous substrate that contributes to the organic loading of the wastewater. Growth on such lysis products is described as cryptic growth and results in a reduced overall biomass production (Low and Chase, 1999). In most of the scientific publications on the topic of ultrasonic sludge disintegration the efficiency of the process is assessed by the release of sCOD in the supernatant liquid of the activated sludge. It is assumed that the solubilisation of particular COD (pCOD) into soluble COD (sCOD) involves first deagglomeration of the active sludge flocs (floc disintegration), and, second, the release of the cellular material in the supernatant liquid as a result of the breakage of the cell wall (cell lysis) (Si-Kyun et al.). However, in current literature little attention is given to the release of nitrogen and phosphorous compounds in the supernatant liquid during the process of sludge disintegration although, e.g., Bougrier et al. (2005) highlight that nitrogen is mainly released as proteins and amino acids and that, at moderate specific energies, a solubilisation degree of 40% can be expected. Several previous research papers reported relevant models for single alkaline activated sludge treatment, single ultrasonic sludge disintegration and the combined treatment process (e.g., Li et al., 2010; Wang et al., 2005 and Kim et al., 2010). All these models use a different combination of independent predictive variables to determine the sCOD release in the supernatant liquid of the activated sludge. It is evident that there are still a lot of questions that remain unanswered. In the first place, it can be said that the choice of the appropriate predictor variables to describe the untreated activated sludge itself and to characterize the process conditions of the ultrasonic sludge treatment is of great importance to generate an appropriate ultrasonic sludge disintegration model. For example, it seems logical to characterize the treated sludge on the basis of both the initial (biomass quantity related) $MLSS_0$ and $MLVSS_0$ concentration. In this way it will be taken into account whether or not we are dealing with a more organic or more inorganic activated sludge or whether the sludge sample is highly thickened or not. The ultrasonic reactor design can be described by the ultrasonic density (D_S - W/cm^2) and the type of ultrasonic horn or transducer can be quantified by the ultrasonic intensity (I_S – W/mL). To have a global view on the energy that is dissipated into the sludge, the sonication time or the specific energy (E_S – kJ/kg DS) of the disintegration process can be used as independent input variable. The specific energy (E_S) is preferred over the sonication time (t) because this parameter contains more information on the overall process conditions of the disintegration process as displayed in Equation (1).

$$E_S = \frac{P_S \, . \, t_r \, . \, 1000 \, g \, . \, 1 \, kJ}{V_{sample} \, . \, c_{DS} \, . \, 1 \, kg \, . \, 1000 \, J} \qquad (1)$$

where:

E_S = specific acoustic energy [kJ.kg DS^{-1}]
t_r = residence time in ultrasonic reactor [s]
V_{sample} = sample volume [L]
C_{DS} = dry solids concentration [g.L^{-1}]

The ultrasonic specific energy is expressed as the associated amount of energy that is transferred to the activated sludge, expressed per kg of activated sludge. Equation (1) clearly indicates that the specific energy is calculated on the basis of the residence time in the ultrasonic reactor and takes into account the power of the ultrasonic generator and the sludge concentration of the activated sludge.

Because it appears from a number of publications that the pH at the start of the disintegration process has a significant influence on the disintegration efficiency, this parameter should also be part of the set of independent variables and is important to ensure an adequate predictive model.

In addition, it is also important that the lysate is not only described by the sCOD release but that also the release of nutrients and the instantaneous reduction in MLSS and MLVSS concentration, that proceeds simultaneously, is included in the development of the model. This means that there should be more than one dependent outcome variable in the ultrasonic disintegration model. Generally it can be concluded that the development of an ultrasonic sludge disintegration model based on a relevant set of independent input variables and a model that can predict more than only the sCOD release would represent an added value to the ultrasound activated sludge disintegration knowledge.

2. MATERIALS AND METHODS

2.1 Ultrasound equipment and acoustic power measurement

Two types of ultrasound equipment were used in the disintegration experiments. The plug-flow pilot reactor consists of a reactor bloc SB® with an array of 20 transducers and an ultrasound generator (LG 1001 T). The system has a fixed frequency at 25 kHz, and a variable power output with a maximum of 1000W. The stirred vessel batch reactor that is used for the execution of the experiments on a laboratory scale is a 20 kHz ultrasonic generator and horn system, type Bandelin Sonoplus® HD3200. The frequency is fixed at 25 kHz and the power output of the generator can be set up to a maximum power of 150 W by an adjustment of the amplitude.

2.2 Experimental methods

All sludge samples were taken from the sludge recycle in the full-scale municipal wastewater treatment plant of Mechelen-Noord (Belgium) at several times during a period of 4 months. The activated sludge samples had an MLSS concentration between 2.4 and 15.2 g DS/L, an average pH of 6.9 and a mean supernatant liquid soluble COD (sCOD) of 47 mg O_2/L. To assess the impact of the ultrasonic treatment on the sCOD, nitrogen and phosphorus release and instantaneous sludge reduction, ultrasonic experiments were performed on:

1. 30 L activated sludge samples in the plug-flow recycle reactor. This sludge is continuously flowing back and forth between the ultrasound device and a storage vessel at a flow rate of approximately 500 L/h. It was always ensured that the samples were taken from the sludge that flows back to the storage vessel and not of the sludge volume of the storage tank itself. Samples were taken at intermittent time intervals over a wide range of specific energy inputs (i.e., from 3000 to about 50000 kJ/kg DS).

2. 150 mL to 1000 mL activated sludge samples in the stirred vessel batch reactor. It was always endeavoured to immerse the ultrasonic horn 1 cm beneath the liquid surface level. In addition, the samples were always cooled with ice, such that temperatures above 45 ° C are avoided and a disintegration effect due to heating of the activated sludge is prohibited.

There are two main differences when comparing the ultrasonic horn system with the ultrasonic plug-flow recycle reactor in terms of the geometry and hydrodynamics of the reactors. Firstly, the ultrasonic horn is immersed directly into the solution, where the sonication takes place. In the plug-flow reactor, an array of transducers is fixed to the external surface of a tube and in this way the tube itself becomes the source of ultrasonic energy. Secondly, the ultrasonic power is more intense when compared with the plug-flow recycle reactor (Santos and Capelo, 2007). Samples were taken and sCOD, MLVSS and MLSS and soluble Kjeldahl Nitrogen (KN), NH_4^+-N and ortho-phosphate measurements were performed in accordance with the Standard Methods for the Examination of Water and Wastewater (2008). The sludge samples were centrifuged immediately after sampling in order to separate the supernatant from the active biomass, so that further biological reactions have been avoided.

2.3 Partial Least Squares regression (PLS-R)

For the prediction of the ultrasonic release of sCOD, nitrogen and phosphorus in the supernatant liquid, a model is developed based on a Partial Least-Squares approach. The aim of Partial Least Squares (PLS) is to develop a linear model that relates the input variables x_i (i = 1 ... K) (denoted as a whole by vector X) to the output variables y_j (j = 1 ... M) (denoted as a whole by vector Y), as is illustrated in Equation (2) (Appels et al., 2011). The idea of PLS regression is to create, starting from a table with N observations described by K input variables, a set of H components with H < K. In other words, PLS aims at finding uncorrelated linear transformations (latent components) of the original predictor variables, which have high covariance with the response variables. In our case we have 75 observations (= N), with 6 independent input variables (= K) and 6 dependent outcomes (= M) as described before. 60 observations are originating from the stirred vessel batch reactor with ultrasonic horn and 15 from the pilot plant plug-flow system. 16 observations were used for validation of the model.

$$y_j = b_0 + b_1x_1 + b_2x_2 + \cdots + b_Kx_K \quad for\ j = 1, \ldots, M \qquad (2)$$

PLS regression is preferred over principal component regression (PCR) and traditional multiple linear regression (MLR), given its advantage of being able to handle more than one response at once (Dahlén et al., 2000). Another advantage of PLS regression modelling is its inherent ability to detect outliers and if the number of latent variables or components is selected with care, modelling of noise will be avoided. The commercial statistical program *XL*STAT was used for the development of the PLS model and to identify the regression coefficients.

3. RESULTS AND DISCUSSION

3.1 Analysing the training data

Before moving to the development of the PLS model it is advisable to study the training data in more detail. Because PLS models assume a set of independent input data and dependent output, it is necessary to verify whether the input data (X) of the model are uncorrelated with each other, but in particular it should also be checked whether the output data (Y) are correlated with each other.

3.1.1 Correlation check of the Y-matrix

If the output data is correlated with each other than it is possible to model and analyse several Y's together, which has the advantage to give a simpler overall picture than one separate model for each Y-variable. Hence, one could evaluate the correlations in the Y-matrix by performing a Principal Component Analysis (PCA) of just the Y-matrix. By carrying out the PCA it can be examined how many components or factors, F_{PCA}, are necessary to describe the data. If this is small compared to the number of Y-variables (M), the Y's are correlated. In the best case, all the variables are described by the first component of the PCA, and a single PLS model of all Y's is warranted. If, however, the Y's cluster in strong groups, which is seen in the PCA loading plots, separate PLS models should be developed for these clustered groups (Wold et al., 2001a). To facilitate the interpretation of a loading and/or biplot, the analysis often involves a rotation of the components that were retained (Abdi, 2003). When the data follow a model stipulating (*i*) that each variable loads on only one factor and (*ii*) that there is a clear difference in intensity between the relevant factors (whose eigenvalues are clearly larger than or equal to one) and the noise (represented by factors with eigenvalues clearly smaller than one), then the rotation is likely to provide a solution that is more reliable than the original solution. The Varimax rotation, developed by Kaiser (1958), is an example of orthogonal rotation and is the most popular rotation method and is also used for the interpretation of our loading plots. For the Varimax rotation method a simple solution, that is consequently easily interpretable, means that each component has a small number of large loadings and a large number of zero (or small) loadings. This simplifies indeed the interpretation because, after a Varimax rotation, each original variable tends to be associated with one (or a small number of) component(s), and vice versa each component represents only a small number of variables.

Looking at the graphs in Figure 1, it can be concluded that a limited orthogonal rotation of the axes of the PCA improves the ease of interpretation of both first two components of the loading plot. In Figure 1 (a) the loading plot before Varimax rotation is represented and in Figure 1 (b) the loading plot after Varimax rotation is plotted. It is clear from these figures that the Varimax rotation ensures that there are high loadings for both the first and second factor, which is not the case in Figure 1 (a). The loading plot, which is represented in Figure 1 (b), reveals the relationship between the output variables in the space of the first two components. We can see that $KN/MLSS_0$, $sCOD/MLSS_0$ and ortho-P/$MLSS_0$ have positive high loadings and $MLVSS/MLVSS_0$ and $MLSS/MLSS_0$ negative high loadings for principal component 1 (D1). NH_4-N/$MLSS_0$, however, has a similar high loading for principal component 2 (D2). It can be very clearly established from the loading plot (Figure 1) that all the variables that are described by the first component of the PCA (D1) can be predicted all together by only one PLS-2-model and that the variable NH_4-N/$MLSS_0$ must be separately estimated by a different PLS-1 model. Although there is a shift in the percentage of the variability that is described by the first two separate factors due to the Varimax rotation, the total percentage of the variability that is described by the first two factors (D1+D2) is still 90.9 %.

From the above analysis it is inferred that the concentration of ammonium that is possibly released by the ultrasonic sludge disintegration is uncorrelated with the release of the other components (e.g., sCOD, TKN and ortho-PO_4). This leads to the hypothesis that the limited increase of ammonium concentration is not directly attributable to the ultrasonic sludge disintegration process itself. These observations are in high contrast to the claims formulated by Feng et al. (2009), who postulated that the ammonia nitrogen concentration of the supernatant rapidly increased with increasing energy input as a direct consequence of the ultrasonic sludge disintegration. The results are, however, confirmed by Khanal et al. (2006), Bougrier et al. (2005) and Akin (2008), who claim that the ammonium release is a secondary phenomenon due to biological hydrolysis of the released organic nitrogen. The rate of hydrolysis is dependent on the electron donor available, or rather the oxygen concentration in the activated sludge. Thus, the rate of hydrolysis is different in aerobic, anoxic and anaerobic conditions, as evidenced by the experimental examination of Henze and Mladenovski (1991). Therefore, it can be assumed that the hydrolysis during the ultrasonic treatment is very slow.

Figure 1: Loading plot, (a) before Varimax rotation and (b) after Varimax rotation.

3.1.2 Correlation check of the X-matrix

In the same way as for the Y matrix, also the X-matrix, with the input data for the development of the PLS model, should be analysed in detail. All input variables should in principle be independent to have a proper input data matrix. In the loading plots after Varimax rotation a clear picture can be sketched about the independency of the various input variables. In the case that each input variable loads high on each another principal component dimension, it can be concluded that all variables are independent. As a loading plot can only display two factors simultaneously, 3 loading plots should be prepared to evaluate the six dimensions in the principal component analysis. The three loading plots are depicted in Figure 2. In loading plot 2(a), the first dimension is determined by both the initial MLSS ($MLSS_0$) and the initial MLVSS concentration ($MLVSS_0$). The second dimension on the other hand is determined by the specific energy (E_S). From loading plot 2(b), it can be deduced that the third and fourth dimension are represented by respectively the pH and the ultrasonic density (D_S). In the last loading plot 2(c) only the fifth dimension has a high loading coming from the ultrasonic intensity (I_S). In principle, one of the two input variables, $MLSS_0$ and $MLVSS_0$, can thus be omitted from the PLS model to develop. It is deliberately chosen not to do so. The reason of maintaining both variables lies within the fact that the ratio of the MLVSS/MLSS concentration is a good measure of the organic content of the activated sludge. Since activated sludge has almost always an MLVSS/MLSS ratio between 60 and 80 percent, the two parameters are strongly connected to each other, which could be derived from the Principal Component Analysis indicating that both parameters are dependent on each other. Because it is expected that the organic content of the activated sludge can have an influence on the solubilisation efficiency, both parameters are nevertheless included as input variables of the PLS model.

3.2 Building the PLS model for ultrasonic sludge solubilisation

It is the intention to build an empirical model that fits the training data perfectly, but that will also well predict new data samples, which are not included in the training data set. The number of latent components to use in the PLS-model is a very important issue because too few components will generate an under-fitted model while too many components induce overfitting.

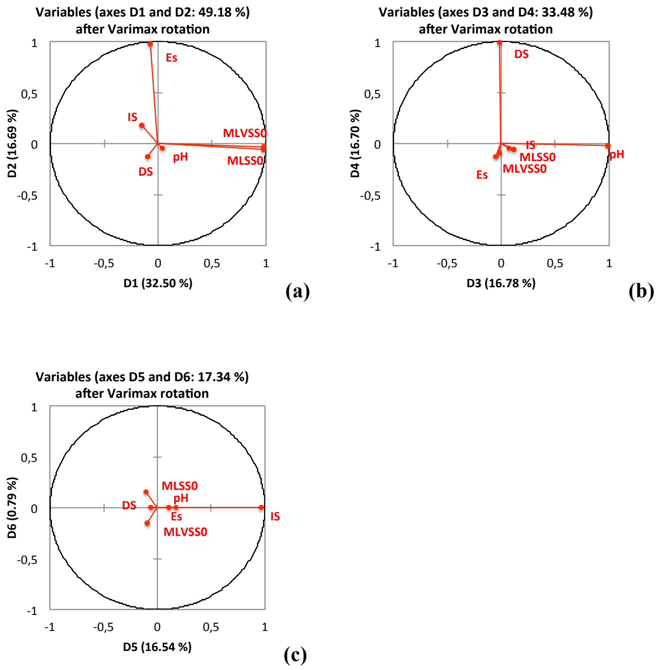


Figure 2: Loading plot after Varimax rotation, (a) for the first 2 components, (b) for the 3[rd] and 4[th] component, (c) for the 5[th] and 6[th] component.

For the response variables (M) in Y, the multiple correlation coefficient (R^2Y_{cum}) or goodness of fit is given by (Wold et al., 2001a):

$$R^2Y_{cum} = \sum R^2\,Y_a \tag{3}$$

where, R^2Y_a is the sum of squares of all the Y's explained by each extracted component a (Amaral and Ferreira, 2005). Selection of the most appropriate latent components, is, hence, the most difficult but also the most important task within PLS modelling, with the ultimate objective to filter the noise from the model.

In order to determine the optimal number of latent components used in the PLS regression model, different methods can be found in the domain-specific literature on PLS modelling (Baumann et al., 2003). Unfortunately, there is no clear directive about which method should be selected to determine the correct number of components to prevent both under-fitting and over-fitting. Many scientific publications have been devoted to study the different methods of cross-validation to yield highly predictive models and to guide the modeller in choosing the right number of latent components. It is proven that the commonly applied leave-one-out and n-fold cross-validation method has a strong tendency to over-fitting, and thus underestimating the true prediction error (Baumann et al., 2003). Therefore, it is chosen to use a combined approach, based on the above-mentioned leave-one-out principle and wherein an external validation is incorporated. All of the samples for modelling were split into a calibration set with full cross-validation (leave-one-out) (N = 75) and prediction set (n = 16). To evaluate the results, the root-mean-square error of calibration (RMSEC) and the root-mean-square error of prediction (RMSEP) were considered; the former is a measure of how well the model fits the calibration data, the latter is a measure of predictive ability of the model when the model is applied to new data (Xie et al., 2009). The RMSE is calculated by squaring individual errors of the training or validation observations, summing them, dividing the sum by the total number of observations, and then taking the square root of this quantity. Good models should have low RMSEC and RMSEP, but small differences between the RMSEP and RMSEC value. Six distinct PLS models, based on a different number of components were built. A first internal validation of the predictive nature of the different models is carried out by the assessment of the course of the R^2Y_{cum}, Q^2_{cum} and RMSEC values as a function of the number of components on which the models are built (the R^2Y_{cum} and Q^2_{cum} evolution is displayed in Figure 3(a)). The Q^2_{cum} index measures the global contribution of the H first components to the predictive quality of the model and of the sub-models if there are several dependent output variables, like in our case. The $Q^2_{cum}(H)$ index writes:

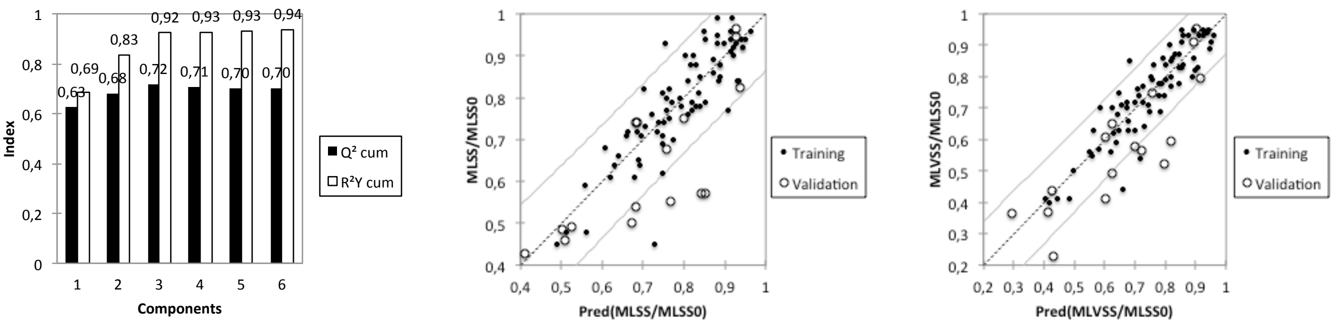
$$Q^2_{cum}(H) = 1 - \prod_{j=1}^{h} \frac{\sum_{k=1}^{q} PRESS_{kj}}{\sum_{k=1}^{q} SSE_{k(j-1)}} \tag{4}$$

The index involves the PRESS statistic (that requires a cross-validation), and the Sum of Squares of Errors (SSE) for a model with one component less. Figure 3(a) gives us a first indication that no more than three components should be used to avoid noise modelling, because the Q^2_{cum} and RMSEC stabilized at the third component. For the evaluation of the external validation, two interesting observations can be made on the basis of the RMSEP values. First and foremost, the prediction results (based on the PLS model with three components) of the validation set lead to low RMSEP values and agree well with the prediction results from the training set, as can be established from Figure 3(b-f). The RMSEP and RMSEC values are in the same order of magnitude, but the RMSEP errors are approximately two times as large as the RMSEC errors (see Figure 3(b-f)). A PLS model with a different number of components will not resolve this problem.

Eventually, the ultimate objective of this paper was reached by the determination of the regression coefficients of the PLS model with 3 components (see Table 1). With the aid of these regression coefficients it is possible to construct the equations to describe the release of sCOD, KN and ortho-PO_4-P in the supernatant liquid of the activated sludge and to predict also the related solubilisation of the VSS and this on the basis of the initial characteristics of the activated sludge ($MLSS_0$ and $MLVSS_0$) and the operational conditions of the ultrasonic treatment (E_S, pH, I_S and D_S). The model is of high quality and seems to be very useful for estimation purposes, for the characterization of the ultrasonic sludge lysis process and predicting ultrasonic waste sludge reduction, without being dependent on time-consuming chemical analyses.

Table 1: Overview of the regression coefficients of the PLS model to calculate the release of sCOD, KN and ortho-PO_4-P and reduction in ML(V)SS.

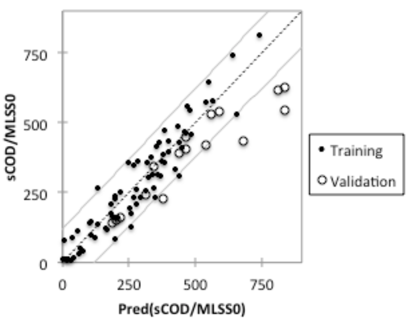
Variable	MLSS/ $MLSS_0$	MLVSS/ $MLSS_0$	sCOD/ $MLSS_0$	ortho-P/$MLSS_0$	KN/ $MLSS_0$
Intercept	1,58E+00	1,66E+00	-7,30E+02	-9,38E+00	-5,04E+01
E_s	-3,62E-06	-4,08E-06	5,25E-03	6,93E-05	3,38E-04
$MLSS_0$	-9,30E-03	-1,24E-02	4,16E+00	1,14E-01	4,69E-01
$MLVSS_0$	-8,74E-03	-1,23E-02	3,67E+00	1,04E-01	4,16E-01
I_S	-1,74E-04	-5,53E-04	1,31E+00	8,81E-03	4,55E-02
D_S	-7,82E-05	-4,10E-05	2,98E-02	1,13E-03	5,52E-03
pH	-6,55E-02	-7,45E-02	9,01E+01	1,22E+00	5,91E+00



(a)

(b)
RMSEC= 0,066
RMSEP = 0,133

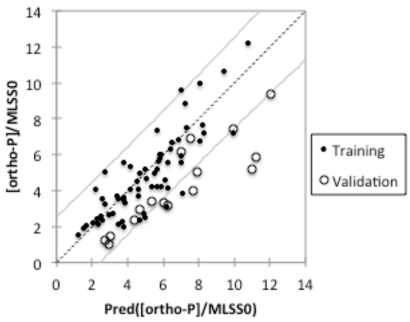
(c)
RMSEC= 0,063
RMSEP = 0,134



(d)

RMSEC= 60

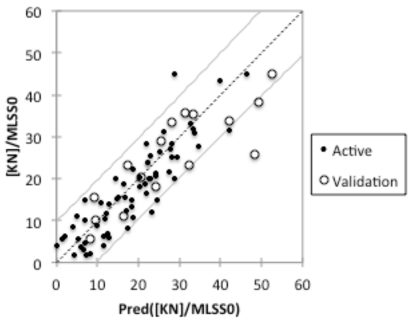
RMSEP = 133



(e)

RMSEC= 1,20

RMSEP = 2,90



(f)

RMSEC= 4,77

RMSEP = 8,08

Figure 3: (a) The evolution of R^2Y_{cum} and Q^2_{cum} with an increasing number of latent components of the PLS model and correlation statistics between the measured values and calculated values for (b) the MLSS reduction, (c) the MLVSS reduction, (d) the sCOD release, (e) the ortho-PO_4-P release, (f) the Kjeldahl-N release. The solid black symbols refer to calibration samples, and the open white symbols refer to the validation samples.

REFERENCES

Abdi, H. (2003). Multivariate analysis. In M. Lewis-Beck, A. Bryman, and T. Fut- ing (Eds), Encyclopedia for research methods for the social sciences. Thousand Oaks, CA: Sage.

Akin B. (2008). Waste Activated Sludge Disintegration in an Ultrasonic Batch Reactor. Clean 2008, Volume 36, Issue 4, Pages 360 – 365.

Amaral A.L. and Ferreira E.C. (2005). Activated sludge monitoring of a wastewater treatment plant using image analysis and partial least squares regression. Analytica Chimica Acta. Volume 544, Pages 246–253.

Appels L., Lauwers J., Gins G., Degrève J., Van Impe J., and Dewil R. (2011) Parameter Identification and Modeling of the Biochemical Methane Potential of Waste Activated Sludge. Environmental Science Technology, Volume 45, Pages 4173–4178.

Baumann K. (2003). Cross-validation as the objective function for variable-selection techniques. Trends in Analytical Chemistry, Volume 22, Issue 6, Pages 395 – 406.

Bougrier C., Carrère H., Delgenès J.P. (2005). Solubilisation of waste-activated sludge by ultrasonic treatment. Chemical Engineering Journal, Volume 106, Issue 2, Pages 163–169.

Dahlén J., Karlsson S., Bäckström M., Hagbergb J., Pettersson H. (2000). Determination of nitrate and other water quality parameters in groundwater from UV/Vis spectra employing partial least squares regression. Chemosphere, Volume 40, Pages71-77

Low E.W. and Chase H.A. (1999). Reducing production of excess biomass during wastewater treatment. Water Research, Volume 33, Issue 5, Pages 1119-1132.

Feng X., Lei H., Deng J., Yua Q, Lia H. (2009). Physical and chemical characteristics of waste activated sludge treated ultrasonically. Chemical Engineering and Processing, Volume 48, Pages 187–194.

Henze M. and Mladenovsk C. (1991). Hydrolysis of particulate substrate by activated sludge under aerobic, anoxic and anaerobic conditions. Water Research, Volume 25, Issue 1, Pages 61-64.

Kaiser H.F. (1958). The varimax criterion for analytic rotation in factor analysis. Psychometrika, Volume 23, Pages 187–200.

Khanal S. K., Grewell D. , Sung S. and van Leeuwen H. J. (2007). Ultrasound Applications in Wastewater Sludge Pretreatment: A Review. Environmental Science Technology, Volume 37, Pages 277-313.

Kim D.-H., Jeong E., Oh S.-E., Shin H.-S. (2010). Combined (alkaline+ultrasonic) pretreatment effect on sewage sludge disintegration. Water Research, Volume 44, Issue 10, Pages 3093-3100.

Li C., Liu G., Jin R., Zhou J., Wang J. (2010). Kinetics model for combined (alkaline + ultrasonic) sludge disintegration. Bioresource Technology, Volume 101, Pages 8555–8557.

Santos H.M., Capelo J.L. (2007). Trends in ultrasonic-based equipment for analytical sample treatment. Talanta, Volume 73, pages 795–802.

Standard Methods for the Examination of Water and Wastewater (2008), 18th Ed. American Public Health Association, American Water Works Association, Water Environment Federation.

Wang F., Wang Y., Ji M. (2005). Mechanisms and kinetics models for ultrasonic waste activated sludge disintegration. Journal of Hazardous Materials, Volume 123, Issues 1-3, Pages 145-150.

Wold S., Sjöström M., Eriksson L. (2001a). PLS-regression: A basic tool for chemometrics. Chemometrics and Intelligent Laboratory Systems, Volume 58, Pages 109–130.

Wold S., Trygg J., Berglund A., Antti H. (2001). Some recent developments in PLS modelling. Chemometrics and Intelligent Laboratory Systems. Volume 58, Pages 131–150.

Xiaoxia Wang, Z. Q. (2010). Characteristics of organic, nitrogen and phosphorus species released from ultrasonic treatment of waste activated sludge. Journal of Hazardous Materials, Volume 176, Issues 1-3, Pages 35-40.