

Estimation of a Mass Transfer Coefficient for Nylon Manufacture Using Multiple Neural Networks

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Abstract

The manufacture of nylon involves the use of cyclohexane, whose successful production is strongly dependent on the kinetics and gas-liquid mass transfer of its oxidation. To properly design the cyclohexane oxidation process, both equipment and process variables must be considered; however, previous studies have been quite limited and specific only to a certain equipment setup. In this paper, a more generalized predictive model of cyclohexane oxidation is proposed that can be used to design the best production method by accurately estimating the mass transfer coefficient. Using experimental data, two neural network approaches are devised and tested. The first is a single network approach whereas the second contains multiple neural networks in a hierarchy, termed a "mixture of experts" approach. These models are assessed using cross validation, which leverages the available data for both the construction and validation of the predictive models. The capabilities of the neural network approach are compared with that of statistical nonlinear regression on a published data set.

Keywords: *Chemical Process Design, Neural Networks, Cross Validation, Mass Transfer Coefficients, Nylon Manufacture*

Introduction

Chemical processes can be very demanding to design and control for optimum production due to interaction among the raw materials and difficulty in observing important characteristics during the process. The liquid-phase oxidation of cyclohexane is such an example. This process is necessary for the production of cyclohexanol and cyclohexanone, which are required for the production of caprolactam and adipic acid, the raw materials for the nylon-6 and nylon-6,6 polymerization processes (Berezin, Denisove, and Emanuel 1966; Suresh, Sridhar, and Potter 1988). The oxidation process of cyclohexane is particularly difficult to design and control because cyclohexanol and cyclohexanone are not the final

reaction products; rather, they are intermediates in a chain of reactions. These products are highly reactive and can be easily oxidized to useless and virtually nonrecoverable by-products (Alagay, Trambouze, and Van Landeghem 1974). Therefore, the success of the process is highly dependent on the rate of oxygen feeding into the reaction mixture, which in turn is measured by the volumetric liquid-side mass transfer coefficient, $k_L a$.

The oxidation process's reaction rate, involving liquid cyclohexane and a feed gas, can be affected by kinetics and the gas-liquid mass transfer (Danckwerts 1970). These two elements are essential for the design and scale-up of the process. There are numerous studies on the kinetics of the cyclohexane oxidation process in the literature; however, study on mass transfer characteristics under actual process conditions has been limited (Tekie et al. 1997).

Tekie, Li, and Morsi (1997) used a statistical approach—a central composite experimental design—to obtain various mass transfer characteristics for two feed gases, N_2 and O_2 , in liquid cyclohexane. These mass transfer parameters were obtained for two types of plants [namely, a gas-inducing reactor (GIR) and a surface aeration reactor (SAR)]. Their experiments considered four controllable variables: x_1 , mixing speed of the liquid cyclohexane and the feed gas; x_2 , elevated pressure of the reaction mixture; x_3 , temperature of the reaction mixture; and x_4 , liquid height. Each variable was set at five levels. Tekie, Li, and Morsi's observations on the effects of mixing speed and liquid height on $k_L a$ were found to be nonlinear. Because of this observation, they introduced exponential terms of these effects into a quadratic response surface model for predicting the mass transfer coefficient. Four

separate models were developed for each combination of gas and reactor in the form of Eq. (1).

$$\ln(k_L a) = \beta_0 + \sum_{i=1}^4 \beta_i x_i + \sum_{i=1}^4 \beta_{ii} x_i^2 + \alpha_1 \exp(\gamma_1 (x_1 + \gamma_2)^2) + \alpha_2 \exp(\gamma_3 (x_1 + 3)(4 - x_4)) \quad (1)$$

where β_0 , β_i , β_{ii} , γ_1 , γ_2 , γ_3 , α_1 , and α_2 are empirical coefficients found in Tekie et al. (1997).

The purpose of this paper is to develop a more general predictive method that can be used for design of chemical processes, especially those where behavior differs significantly in different regions of the process domain. This method is illustrated on the cyclohexane oxidation process by modeling the mass transfer coefficients for a variety of plant equipment (reactors and feed gases) and process settings (temperature, pressure, etc.). This model can be used to design a cyclohexane oxidation process that is stable without the need to build pilot plants and then extrapolate their performance to a production facility. Instead of developing a complicated statistical regression model, this paper takes a more purely nonparametric approach and uses an artificial neural network that can accommodate both nonlinearities and complex variable interactions. It is shown that this approach is effective both in precision and in generalization and has promise to be used in the chemical manufacturing industry to aid in the design of such processes.

Artificial Neural Networks

Artificial neural networks began in the 1940s, with the intention of emulating the strength and processing power of the human brain. A neural network is a parallel distributed processor and it acquires knowledge through iterative "learning." This acquired knowledge is stored in its connection weights, which transcend from an initial random state to a fixed state through the learning (error feedback and correction) process.

A typical neural network consists of several layers of interconnected processing units (see Figure 1). The learning (also known as training) process involves three stages: (1) the feedforward of the input training pattern, (2) the calculation and backpropagation of the associated error, and (3) the adjustment of the weights according to an error metric. The backpropagation learning algorithm is the most well

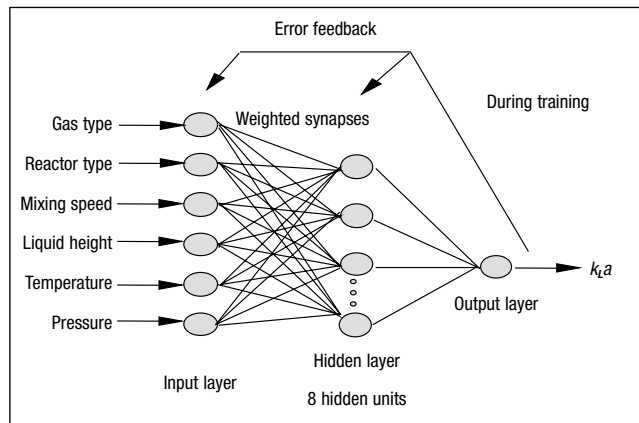


Figure 1
 Single Network Approach

known training algorithm and adjusts the connection weights according to the gradient descent method (Rumelhart, Hinton, and Williams 1986; Funahashi 1989; Hornik, Stinchcombe, and White 1989), where the squared error is minimized in the direction of greatest improvement. This method has the distinction of universal approximation, an important property for predictive modeling of manufacturing processes with nonlinear behavior.

Neural networks have been increasingly applied in many manufacturing venues. Udo (1992) presented a survey paper on the application of neural networks in manufacturing processes such as resource allocation, scheduling, process control, robotic control, and quality control. Coit, Jackson, and Smith (1998) demonstrated two applications of neural networks: a ceramic casting process and a wave soldering process. Similar to Udo, Hussain (1999) provided a review paper on the applications of neural networks in chemical process control. The author categorized control schemes into predictive, inverse model based, and adaptive control methods. te Braake, van Can, and Verbruggen (1998) presented a semi-mechanistic modeling of a first-order exothermic reaction with neural networks. They collected data on most of the parameters of the process and applied a first-order Euler approximation to calculate the reaction rate. Using the collected data along with the derived reaction rate, they trained a neural network to model the chemical relationship.

One of the ideas of this paper is to use multiple neural networks for a single prediction problem. It is applicable for the chemical process under study because the process domain can be classified into multiple distinct domains, a property of many com-

plex manufacturing processes. The multiple neural network approach is called a “mixture of experts” and was first presented by Jacobs, Jordan, and Barto (1991) and Jacobs et al. (1991). It consists of several “expert” networks and a “gating” network. Each expert learns from a subset of the available data; in other words, each expert is responsible for a local region of the space spanned by the original data set. The experts can be competitive (Jacobs, Jordan, and Barto 1991), successive, cooperative, or supervisory (Sharkey 1997). The gating network, acting as a supervisor, learns how to optimally combine outputs from the experts. There are several reasons for adopting this approach: (1) it allows easier interpretation of the models; (2) it tends to reduce model complexity; and (3) it can reduce training time and make subsequent extension of the models easier (Jacobs, Jordan, and Barto 1991; Jacobs et al. 1991; Sharkey 1997).

Development and Validation of Models

Unlike the quadratic response surface models [Eq. (1)] of Tekie, Li, and Morsi (1997), where a separate model was obtained for each set of plant equipment, the neural networks here consider plant equipment and process settings together as variables for predicting the mass transfer coefficients. This approach is advantageous because the networks can be used to optimize the complete design of the oxidation process considering variations in plants (reactor and gas) along with the usual process variables.

Data Set

Data collected from a central composite experimental design was used for building and validating the networks. The process variables include temperature, pressure, mixing speed, and liquid height whereas the observed outcome is the mass transfer coefficient. Four types of plants were considered: all possible combinations of two feed gases (O_2 and N_2) with two reactor types. Reaction temperature ranged from 330°K to 430°K , reaction pressure ranged from 7 to 35 bar, mixing speed ranged from 400 to 1200 rpm, and liquid height ranged from 0.17 to 0.27 m. The reactor had a dished bottom with an internal diameter of 0.114 m. It was equipped with four baffles spaced 90° and one tenth of the reactor diameter in width. The stirrer was a hollow shaft whose outside diameter was 0.01 m and was fitted

with a six flat-blade turbine impeller with a diameter of 0.05 m. The hollow shaft had four holes, each with a diameter of 1.5×10^{-3} m. The reactor was glass lined and could be operated in a surface aeration (SAR) or gas-inducing mode (GIR), depending on whether the holes in the hollow shaft were sealed or not. When the four holes were sealed, the reactor was operating in the surface aeration mode. There were 298 observations collected, which were pared to 296 observations after deletion of two unexplained outlier points. *Figure 2* shows the main components of the reactor setup.

Validation of the Networks

Neural networks are often referred to as “black boxes” or “opaque models” because it is very difficult, if not impossible, for a human to discern the analytic behavior of a network. Therefore, validation is at least as important as construction in neural network predictive modeling for industrial use. In this research, resampling methods, adopted from the statistical literature, were used for network validation. More specifically, the cross validation and grouped cross validation methods were used. These resampling methods leverage the available data by allowing construction of the final network using the entire data set while validating the network also using the entire data set (Geman, Bienenstock, and Doursat 1992; Twomey and Smith 1998; Wolpert 1993). The network built and validated on all data points is termed the *application network*, that is, the one that

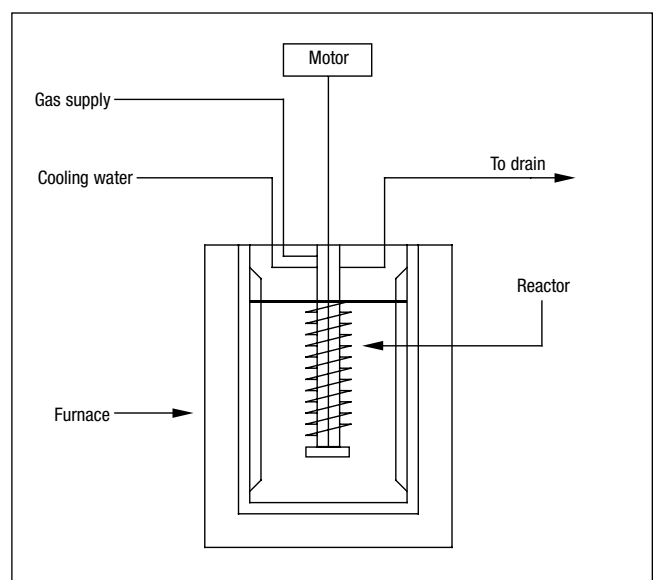


Figure 2
Schematic of Main Components of Reactor Setup

will be implemented in the manufacturing plant. Networks trained and validated on a portion of the data are *validation networks*, and they are used solely to infer performance of the application network.

The Single Network Approach

This approach uses one backpropagation network, which has six input variables—gas type, reactor type, mixing speed, pressure, temperature, and liquid height. The target (output) of the network is prediction of the mass transfer coefficient (see *Figure 1*). The network architectures, training parameters, and stopping criteria were identified through experimentation and examination of preliminary networks. The best network has six input units, one hidden layer with eight hidden units, and one output unit. The learning rate was fixed at 0.15, and training stopped at 11,000 epochs (that is, 11,000 passes through the entire training set). This network was validated using a 74-fold cross validation approach, which requires building 74 validation networks. Each validation network was constructed using 292 observations and tested on the four left-out data points. Combining all 74 test sets exhausts the original data set ($74 \times 4 = 296$). All validation networks and the final network (using all 296 data points for training) were built using the parameters and architecture specified above.

Table 1 shows the mean absolute errors (MAE), the root mean squared errors (RMSE) and the R-squared values (percentage of variance in the data explained by the neural network model) of the final network and the validation networks. The final network can explain about 98% of the variation of the mass transfer coefficients; that is, only 2% of the variability in the coefficients is not explained by the model. With a paired t-test for the application network, there is no statistical difference between the actual $k_L a$ and the predicted $k_L a$ (p value = 0.468). An F test for equal variances between the actual and

Table 1
MAE, RMSE, and R-Squared for Single Network Approach

	MAE	RMSE	R-Squared
Application network	0.000668	0.00106	0.9856
Validation networks	0.000790	0.00133	0.9773

the predicted has a p value of 0.325, which indicates that the values are statistically identical. Comparing the predictions of this single neural network with the four nonlinear regressions [(Eq. (1)] of Tekie, Li, and Morsi (1997) is shown in *Table 2*. The application consists of testing the model on the same data used to construct it (termed *resubstitution*) and the validation consists of testing the model on data independent of that used to build the model. While the resubstitution results are similar, the regression models do not generalize as well as the neural network as indicated by their reduced validation performance. Furthermore, the neural network predictions are from only a single network for all combinations of reactor and gas, while the nonlinear regression approach requires a model for each combination. *Figures 3* and *4* show the relationship between the predicted and the target mass transfer coefficients using a logarithmic scale. It can be seen that the neural network model is unbiased with uniformly good prediction. However, it can also be seen that most observations have a coefficient with log of -4.5 or less, whereas a few observations are at the higher end of the log range. This indicates the possible applicability of multiple neural networks.

Mixture of Experts Approach

This approach was motivated because the mass transfer coefficient data clusters in two unequally represented groups. The original data set of 296 was divided into two groups according to the distribution of the mass transfer coefficients, one of $k_L a$ less than 0.01 (low group) and the other of $k_L a$ greater than 0.01 (high group). There were 260 data points in the former group and 36 in the latter.

Table 2
Comparison of R-Squared of Nonlinear Regression and Single Neural Network Approaches

	Neural Network ¹	N ₂ GIR Regression ²	N ₂ SAR Regression ³	O ₂ GIR Regression ³	O ₂ SAR Regression ²
Application (<i>resubstitution</i>)	0.9856	0.9929	0.9474	0.9821	0.9634
Validation	0.9773	0.9668	0.8888	0.9625	0.7668

1. Over 296 observations.
2. 32 observations for application and 48 observations for validation.
3. 32 observations for application and 50 observations for validation.

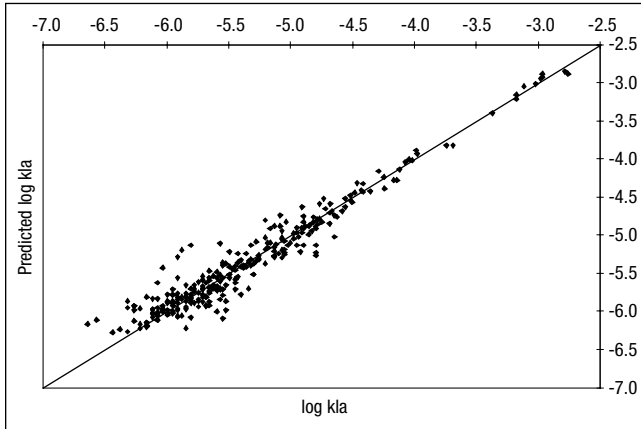


Figure 3
 Performance of Application Network for Single Network Approach

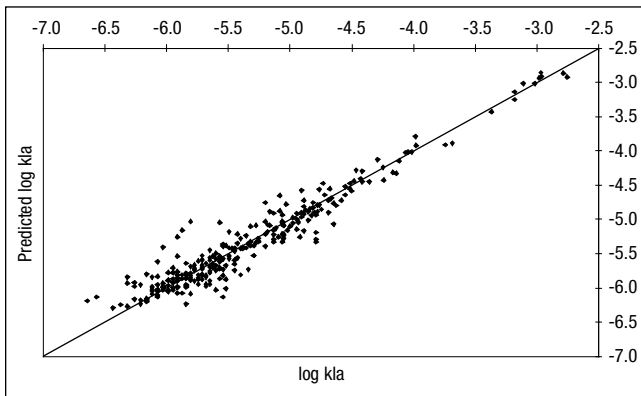


Figure 4
 Performance of Validation Networks for Single Network Approach

Then a classification (gating) network was trained using all 296 observations with 0 as targets for the observations in the low group and 1 as targets for those in the high group. The classification network was trained using the backpropagation algorithm and validated using the 74-fold cross validation method. The best classification network has six input units, one hidden layer with five hidden units,

and one output unit. A constant learning rate of 0.25 was used for training the network, and training stopped at 28,000 epochs. *Table 3* summarizes the performance of the classification network when considering different decision tolerances. For example, the 0.5 tolerance means that any output less than 0.5 is categorized as low and any other output is categorized as high. The 0.1 tolerance means that outputs between 0 and 0.1 are categorized as low and outputs between 0.9 and 1 are categorized as high. All other outputs are not classified. It can be seen that the gating network is very decisive, in that nearly all data is classified correctly even at the most stringent tolerance (0.05).

The entire data set was first classified into two groups using the gating network; then two different expert prediction networks were trained, where one of them used the 260 observations classified as low k_{La} and the other one used the data from the high k_{La} group. See *Figure 5*.

The best low expert network has six input units, seven hidden units, and one output unit. The learning rate was set at 0.10 and training terminated at 10,000 epochs. A 65-fold cross validation method was used to estimate the performance of the low expert. *Table 4* summarizes the error measures and the R-squared values of the networks. The low expert can explain about 88% of the variation of the mass transfer coefficients. Using a paired t test, the actual and predicted values of the application network yields a p value of 0.045. This indicates a statistical difference at $\alpha = 0.05$ but not at α values of 0.04 or smaller. An F test of variances indicates that the variances of the actual and predicted k_{La} are statistically identical with a p value of 0.311. *Figures 6* and *7* show the performance of the low expert and the validation networks.

Table 3
 Classification Performance of Gating Network

	% Correct Classification			
	Tolerance = 0.05	Tolerance = 0.10	Tolerance = 0.20	Tolerance = 0.50
Application network	100.00	100.00	100.00	100.00
Validation networks	97.30	98.99	98.99	99.66

Table 4
 MAE, RMSE, and R-Squared for the Low Expert

	MAE	RMSE	R-Squared
Application network	0.000424	0.000565	0.9241
Validation networks	0.000508	0.000714	0.8785

Table 5
 MAE, RMSE, and R-Squared for the High Expert

	MAE	RMSE	R-Squared
Application network	0.00063	0.00091	0.9970
Validation networks	0.00147	0.00201	0.9853

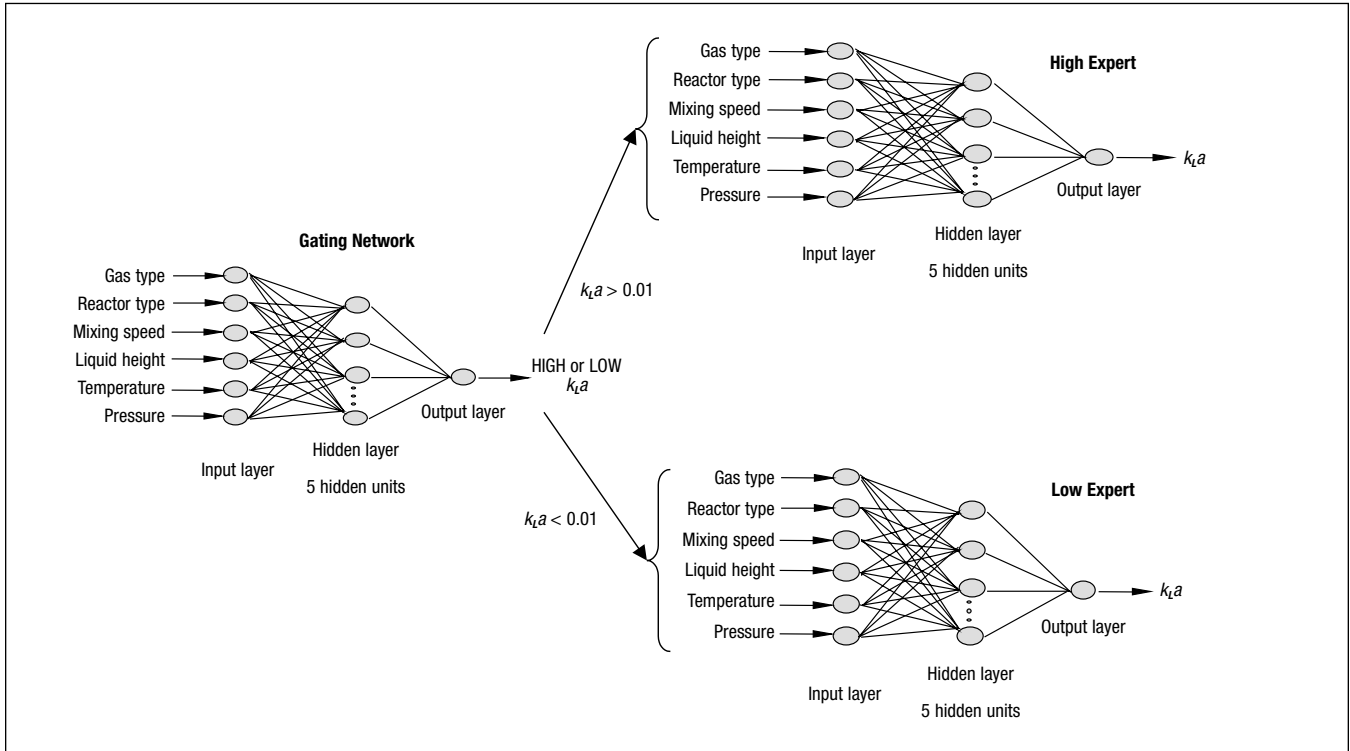


Figure 5
 Mixture of Experts Approach

The best high expert network has six input units, five hidden units, and one output unit. The learning rate was set at 0.25 and training terminated at 18,000 epochs. A leave-one-out cross validation method was used to estimate the performance of the high expert. This validation method is more computationally expensive than the grouped cross validation method. However, because there are only 36 observations in this group, the computational expense of constructing 36 validation networks can be justified. This method gives a more accurate estimate of the performance of the high expert.

Table 5 summarizes the error measures and the R-squared values of the networks. The high expert can explain about 99% of the variation of the mass transfer coefficients. A paired t test yields no significant difference between the actual and predicted $k_L a$ of the application network with a p value of 0.677. An F test of variances shows that the two are equal with a p value of 0.500. Figures 8 and 9 show the performance of the high expert and the validation networks.

Comparing the prediction versus actual figures from both network approaches shows that the mixture of experts approach is superior to the single net-

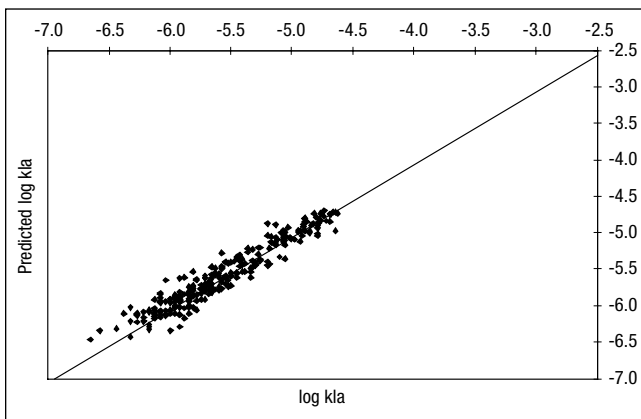


Figure 6
 Performance of Low Expert for Mixture of Experts Approach

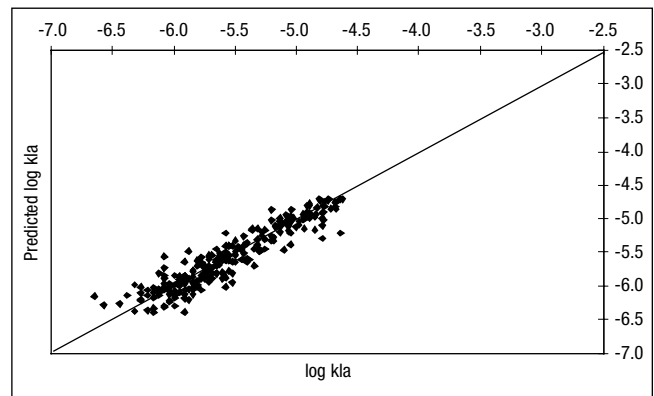


Figure 7
 Performance of Validation Networks for Low Expert

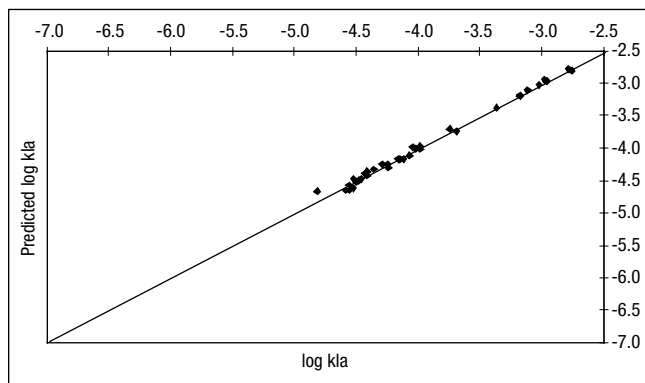


Figure 8

Performance of High Expert for Mixture of Experts Approach

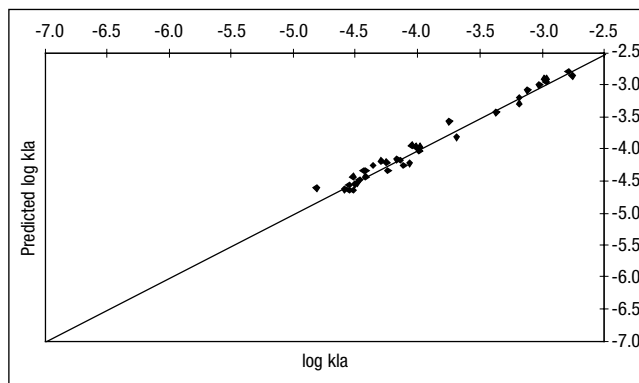


Figure 9

Performance of Validation Networks for High Expert

work approach. However, the error measures given in *Tables 4* and *5* cannot be compared directly with those in *Table 3*. *Table 6* provides a summary of the results for these two approaches and confirms the visual observations of the figures. The multiple neural network approach allows precise prediction of higher mass transfer coefficients, even though these occur relatively rarely. With a single neural network approach, precision is focused on those observations that result in lower coefficients because they are so numerous. The multiple neural network approach accommodates these two unequally represented portions of the process domain, although the hierarchical predictive model is larger and more difficult to build and validate.

Conclusions

Two neural network approaches using plant variables along process variables were investigated for developing a predictive model of mass transfer coefficients to be used in the design of a chemical oxidation process. A real data set for cyclohexane oxidation, which was previously the subject of statistical regression, was used. Unlike the regression approach, the neural network does not require the construction of separate prediction models for each equipment setup, that is, the combination of gas type and reactor type. Therefore, it can be used for design of the complete process, rather than for the process variable settings only. Furthermore, the neural network approach shows better generalization performance than the nonlinear regression indicating a more robust model.

The mixture of experts approach proves to be modestly superior to the single network approach for this application, allowing a specialized agent to be trained

Table 6

Comparison of MAE, RMSE, and R-Squared for the Two Approaches

	MAE	RMSE	R-Squared
Single network (Application)	0.000668	0.00106	0.9856
Single network (Validation)	0.000790	0.00133	0.9773
Mixture of experts (Application)	0.000449	0.00062	0.9951
Mixture of experts (Validation)	0.000624	0.00097	0.9880

on a certain region of the space spanned by the original data. This approach makes sense for the mass transfer coefficient application as there are two regions where the physical behavior of the system differs substantially and for which the data observations are unequal. Using a classification gating network followed by two expert prediction networks does entail more development work, but allows for more precise prediction of the mass flow coefficient over the entire region spanned by the data set. This approach could be used in other chemical manufacturing applications where data describing the process outcome for a variety of plant and process conditions are available, and for which behavior differs over the process domain. It is especially beneficial to use a predictive model in chemical manufacturing because of the expense of building pilot plants and the volatile dynamics of many reactions. The ability to confidently predict how a process will behave under alternative equipment setups and process variables will allow an engineer to properly design the process off-line in a cost effective and expedient manner.

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