



Development and comparison of non-parameter regression methods for prediction of cell voltage and current efficiency in a lab scale chlor-alkali membrane cell

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ABSTRACT

This article presents the development & comparison of Non-parameter Regression Methods such as Artificial neural network (ANN), Genetic algorithm optimization (GA) and Support vector machine (SVM) models for the prediction of cell voltage and caustic current efficiency (CCE) versus different operating parameters in a lab scale chlor-alkali membrane cell. In order to validate the model predictions, the effects of various operating parameters on the cell voltage and CCE of the membrane cell were experimentally investigated. Each of six process parameters including anolyte pH (2–5), operating temperature (25–90°C), electrolyte velocity (1.3–5.9 cm/s), brine concentration (200–300 g/L), current density (1–4 kA/m²), and run time (up to 150 min) were thoroughly studied.

The new models yielded the accurate prediction of experimental data with the lowest standard deviation error (SD). It was found that the developed models are not only capable to predict the voltage and CCE but also to reflect the impacts of process parameters on the same functions. According to the obtained results, SVM model is suitable for the prediction of CCE with an average deviation of 1.53% while GA & ANN models are more accurate than SVM model for predicting the voltage with an AD of 1.21% & 1.27%, respectively.

Keywords: Chlor-Alkali; Membrane cell; Electrolysis; Artificial neural network; Genetic algorithm; Support vector machine

1. Introduction

Chlor-alkali (CA) production is the largest industrial scale electro synthesis. One of the major issues confronting the chlor-alkali industry is the high power consumption, i.e. about 1.5×10^8 MWh of electricity per year [1]. It was estimated that the power consumption accounts for over 50% of the operating costs [2,3]. Improvement of the electrolytic process in this respect, i.e. reduction in cell voltage, would be beneficial from both economical and environmental point of views.

Cell voltage and current efficiency are two most important process parameters proportional to the power consumption of a CA plant. Therefore, the process evaluation is important from industrial point of view in order to quantify the impact of process variables on these two parameters. At the same time, prediction of the cell voltage and current efficiency can facilitate achieving the optimum conditions which will further reduce the intercalary costs of trial and error experiments.

Various methods have been employed to predict and quantify the process parameters, i.e. statistical

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methods [4], analytical formulations [5] and non-parameter regression methods like artificial neural network (ANN) [6], Genetic Algorithm (GA) and support vector machine (SVM) [7]. On the other hand, the effects of operating parameters (five factors) on the performance of a CA membrane cell using Taguchi and ANOVA techniques were recently studied by Jalali et al. [4] but the effect of electrolysis duration was neglected in that study.

Statistical methods are used to analyze the results of the experiments and models on response and also they can be used to determine the contribution of each influencing factor. However, the main concern with statistical methods is the difficulties in fulfilling many rigid assumptions that are essential for justifying their applications such as those of sample size, linearity, and continuity. One alternative approach for system predicting is non-parameter regression methods. In nonparametric regression a priori knowledge of the functional relationship between the dependent variable Y and independent variables, X_1, X_2, \dots, X_m , is not required. In fact, one of the main results of non-parametric regression is determination of the actual form of this relationship.

The objective of this article is to develop general models based on these techniques including ANN, Genetic Algorithm Optimization (GA) and SVM that relate the cell voltage and caustic current efficiency (CCE) to operation parameters and quantify the impact of process variables on these two parameters. Implementation of such methods would enable the operators of chlor-alkali membrane cell units to define their optimum process conditions and benefit from the savings in time and energy.

On the other hand, the CA model achieved by these methods can be employed to examine the effects of various operating parameters as well as to compare the model predictions with the experimental values. With the developed models, one can further study the variations of dependent parameters versus independent parameters. The main difference of non-parameter regression techniques from statistical methods is attributed to its relinquishment in terms of strict conditions for data samples and associated assumptions. This is applicable to the existing situation of data availability for the cell voltage and CCE factors, which are not good enough for either statistical or numerical modeling. At the same time, analytical models are better than these models in terms of their touching the detailed mechanisms of interactions among various impact factors. Nevertheless, such methods' limitations are also from their attempts to specify the complicated processes by detailed mathematical formulations, since many doubtful, interactive, and dynamic system

components can barely be expressed as precise analytical formulations. Under such conditions, non-parameter regression methods become one of the usable means for analyzing the related effects and interactions; it can be used without disturbing either a number of prerequisites associated with statistical models or being forced to assume unrealistic or oversimplified system conditions that are needed for analytical simulation [6,7].

So, the main aim of this study was thus to investigate the impacts of operating parameters on the cell performance indicators, i.e. cell voltage and CCE, and to predict the same by ANN, GA & SVM techniques. Process parameters that have been experimentally studied at four levels include anolyte pH (2–5), cell temperature (25–90°C), electrolyte velocity (1.3–5.9 cm/s), brine concentration (200–300 g/L), current density (1–4 kA/m²) and run time (up to 150 min).

2. Theoretical background

One alternative approach for system predicting is the non-parameter regression methods like ANN based on the theory of artificial intelligence, genetic algorithm (GA) based on the idea of "survival of the fittest" and "natural selection" and SVM based on the structural risk minimization (SRM) principles.

Meanwhile, with the developed models, one can further study the variations of dependent parameters versus independent ones.

Artificial neural networks have several attractive properties for the modeling of complex production systems, i.e. capability of universal function approximation, resistance to noisy or missing data, accommodation of multiple non-linear variables with unknown interactions, and good generalization ability [8]. For manufacturing processes where either no satisfactory analytical model exists or a low-order empirical polynomial model is inappropriate, neural networks are a good alternative approach.

When artificial neural networks are used for prediction and forecasting, the underlying philosophy is similar to that used in traditional statistical approaches. Therefore, ANNs and statistical models are closely related. Consequently, the principles that are considered good practice in the development of statistical models need to be given careful consideration. The major areas that should be addressed include data pre-processing, choice of adequate model inputs, choice of an appropriate network geometry, parameter estimation, and model validation. At each stage, a number of alternatives are available to modellers. This offers great flexibility, but can also create difficulties as

there are no clear guidelines to indicate under what circumstances particular approaches should be adopted. Therefore, the performance is very much dependent on the network architecture. Hence, an optimum or near optimum network structure is of utmost importance. This can be done by means of GAs as a powerful optimization tool. So, the design of neural networks using GA principles can be very helpful in terms of two main issues [9]:

- It automates the design of the network which will otherwise have to be done by hand using trial and error.
- The process of design can be analogous to a biological process in which the neural network blueprints encoded in chromosomes develop through an evolutionary process.

Based on SVM principle, this method achieves an optimum network structure by striking a right balance between the quality of the approximation of the given data and the complexity of the approximating function. The SVM reveals the underlying statistical relationships among variables corrupted by random error. This SVM algorithm presented by Vapnik [10], as other similar non-parametric statistical regression methods is intended to alleviate the main drawback of parametric regression, i.e., the mismatch of assumed model structure and the actual data. Based on this principle, SVM achieves an optimum network structure by striking a right balance between the quality of the approximation of the given data and the complexity of the approximating function. Therefore, the over-fitting phenomenon in the general ANN can be avoided and excellent generalization performance can be obtained. Furthermore, in SVM, support vectors corresponding to the hidden units of the general ANN are automatically determined after the SVM training. This implies that the difficult task of determining the network structure in the general ANN can be prevented.

- Compared with traditional neural networks, SVM possesses prominent advantages:
- Strong theoretical background provides SVM with high generalization capability and can avoid local minima.
- SVM always has a solution, which can be quickly obtained by a standard algorithm (quadratic programming).
- SVM need not determine network topology in advance, which can be automatically obtained when training process ends.
- SVM builds a result based on a sparse subset of training samples, which reduce the workload.

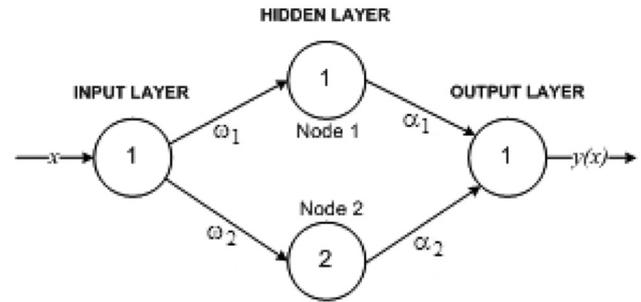


Fig. 1. A simple ANN scheme with one input node, two hidden nodes, and one output node.

2.1. Artificial neural network

The enormous interconnections in the ANN framework create a great number of degrees of freedom, or fitting parameters, and thus may permit it to reflect the system's complexity more effectively than conventional statistical techniques.

Artificial neural networks are massively parallel interconnected networks of simple elements and their hierarchical organizations which are intended to interact with the objects of the real world in the same way as biological nervous do, or simply a system of interconnected computational units, or nodes [11]. A simple neural network is shown in Fig. 1. It consists of an input layer, a hidden layer, and an output layer.

In turn, these layers have a certain number of nodes or neurons, so that the nodes are also called input nodes, hidden nodes, and output nodes. Fig. 1 shows a network with one node in the input layer, two nodes in the hidden layer, and one node in the output layer. The output, $y(x)$, is a function of the input, x , and a set of parameters. Input nodes receive data from sources external to the network and send them to the hidden nodes, in turn the hidden nodes send and receive data only from other nodes in the network, and output nodes receive and produce data generated by the network which goes out of the system. In general, the number of input nodes may be greater than one. Likewise, the number of hidden layers can be greater than one, but a network with a single hidden layer is simpler and useful for many applications. Furthermore, the output layer can have several nodes. In general, a typical network has n input nodes, one hidden layer with h nodes, and m output nodes. A typical problem is then to estimate the output as a function of input. This function is unknown but may be approximated by a superposition of certain activation functions such as hyperbolic tangents, sigmoids, polynomials, and sinusoids in a neural network fashion.

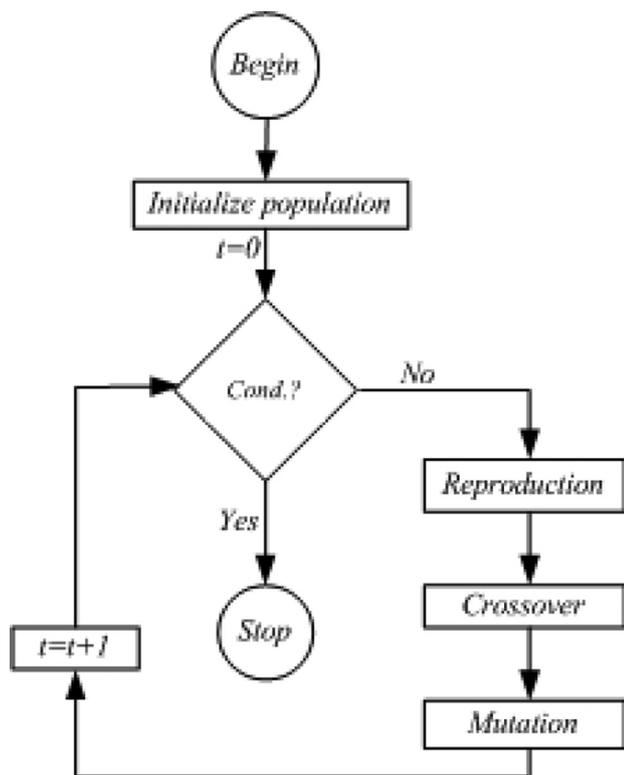


Fig. 2. A flowchart of working principle of genetic algorithm [12].

2.2. GA

Based on the idea of “survival of the fittest” and “natural selection”, GA is a class of parallel iterative algorithm with a certain learning ability that repeats evaluation, selection, crossover and mutation after initialization until the stopping condition is satisfied [12]. GA is naturally parallel and exhibits implicit parallelism, which does not evaluate and improve a single solution, but analyses and modifies a set of solutions simultaneously. Even if the selection operator can select some “good” solutions as seeds with random initialization, the crossover operator can generate new solutions, hope-fully retaining good features from parents, and the mutation operator can enhance diversity and provide a chance to escape from the local optima [9].

A major issue in the genetic based design of a neural network is that of representation (encoding). The encoding should be capable of capturing all of the important aspects of the problem. Therefore in GA, the representation scheme should be capable of allowing new, meaningful and valid network architecture to be produced by the genetic operators, (like crossover or mutation). GA is applied to the neural network in two different ways:

- They either employ a fixed network structure with a connection under evolutionary control.

- They are used in designing the structure of the network.

Therefore the evolution that has been introduced to neural networks can be divided roughly into different levels: (a) connection weights; (b) architecture; (c) learning rules. Fig. 2 shows the working principles of GA [12].

2.3. SVM

SVM is a relatively novel powerful machine learning method based on statistical learning theory (SLT), which is a small-sample statistical theory introduced by Vapnik [10]. SVM is powerful for the problems characterized by small samples, nonlinearity, high dimension and local minima. Currently, SVM is an active field in the artificial intelligent technology, and has been applied to the pattern recognition and function estimation [13]. The empirical risk minimization (ERM) principle is generally employed in the classical methods such as the least-square methods, the maximum likelihood methods and traditional ANN. In SVM, the ERM is replaced by the SRM principle, which seeks to minimize an upper bound of the generalization error rather than minimize the training error [13,14].

In addition, the basic concept of the SVM regression is to map the input data into a feature space via a non-linear map. In the feature space, a linear decision function is constructed. The SRM principle is employed in constructing optimum decision function. Then SVM nonlinearly maps the inner product of the feature space to the original space via kernels [15].

3. Experimental

3.1. Materials

The electrolyte was prepared from analytical grade NaCl and NaOH from Merck Inc. (Germany) using double distilled water. All other chemicals used for analysis were also of analytical grade.

3.2. Apparatus

The cell performance test was carried out in a CA set-up similar to a scaled-down industrial brine electrolysis unit. Fig. 3 shows a simplified flow diagram of the set-up used in this study. The cell was a divided filter-press type (*Electrocell AB, Sweden*) with Flemion[®] 892 as the separator, a standard DSA Cl₂ as the anode and a Ni plate as the cathode. The electrode membrane gap was 2 mm (See Fig. 4). The feed tanks were heated by means of jacketed heaters and their temperatures

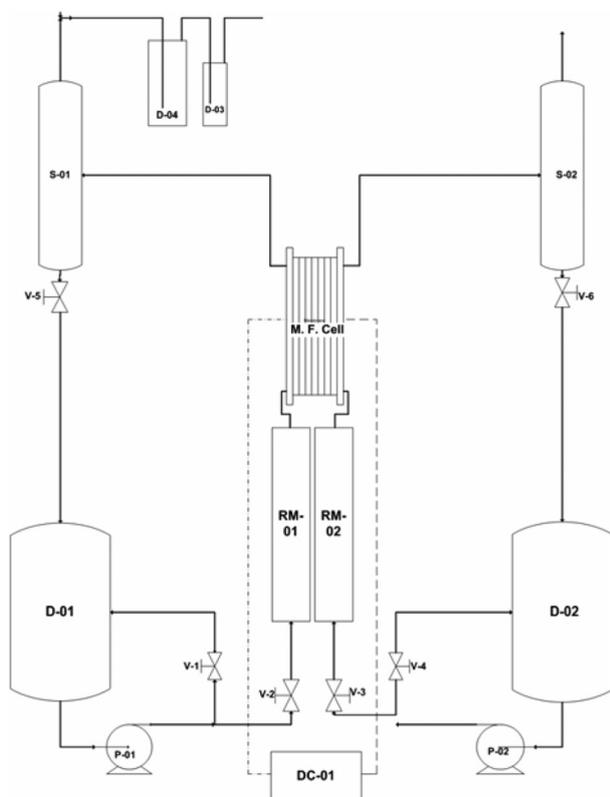


Fig. 3. Process flow diagram of the chlor-alkali set-up utilized: 1. Membrane flow cell (M.F. cell), 2. Electrolyte tank (D-01, D-02), 3. Magnetic pumps (p-01, p-02), 4. Gas separator (S-01, S-02), 5. Rotameter (RM-01, RM-02), 6. DC power supply & 7. Two feed tank consist of NaOH for neutralization produced chlorine (D-03, D-04).

were monitored by digital thermometers. Galvanostatic operation was employed using a DC power



Fig. 4. Side view of the membrane cell used in this study, Cell body (Teflon), EPDM gasket, Standard DSA/Cl₂ anode, Flemion 892 membrane, Nickel cathode, Flow frame (Teflon), Electrolyte inlet, Electrolyte outlet.

supply. Anolyte pH was measured by a pH-meter inserted in the anolyte feed tank. The membrane was immersed in a NaOH solution for a day to reach equilibrium before each experiment.

3.3. Experimental procedure

The anolyte and catholyte were circulated in separate hydraulic circuits during the experiment by two magnetic pumps according to Fig. 3. The overflows from the anolyte and catholyte compartments of the cell were fed into distinct separators. The bubble free electrolytes were returned to the appropriate feed tanks for further recirculation. During electrolysis, Cl₂ gas produced was absorbed by 2M NaOH solution in D-03 and then D-04. Constant currents were applied to the cell and the cell voltages were measured. After each test, the set-up was washed thoroughly with distilled water, drained and dried. The electrolysis run times were 30, 60, 90, 120 and 150 min.

3.4. Chemical analysis

In order to measure the amount of caustic produced, catholyte samples were collected and titrated against 0.1 N HCl. These data were then used for calculation of the CCE.

4. Results and discussions

4.1. Data collection

According to our experiences as well as those of previous works [16–18] the important parameters affecting the CA cell performance along with the levels of these parameters are as follows: (1) anolyte pH: 2, 3, 4 and 5, (2) cell temperature (°C): 25, 50, 70 and 90 (3) flow velocity (cm/s): 1.3, 2.2, 3.7 and 5.9, (4) brine concentration (g/L): 200, 235, 270 and 300, (5) current density (kA/m²): 1, 2, 3 and 4 and (6) run time: from 30 to 150 min. The latter are summarized in Table 1.

By running experiments under these conditions using the procedure described elsewhere, cell voltage and CCE data at various operating conditions were obtained.

4.2. Calibration & models development

The cell voltage and CCE data were divided into two data sets, consisting of training and validation test data. In the training phase, a larger part of the data, i.e. 75–80%, was used to train the models and the remaining data, 20–25%, were used in the validation phase.

Table 1
Levels of process parameters

	pH	Cell temperature (°C)	Flow velocity (cm/s)	Brine concentration (g/L)	Current density (kA/m ²)	Run time (min)
Level 1	2	25	1.3	200	1	30
Level 2	3	50	2.2	235	2	60
Level 3	4	70	3.7	270	3	120
Level 4	5	90	5.9	300	4	150

The scatter plots in Figs. 5–8 (a & b) provide comparisons of the measured cell voltage and CCE levels with the ANN & GA-derived test and train ones respectively, while Figs. 9 and 10 (a & b) shows comparisons of the measured cell voltage and CCE levels with those of the SVM-derived.

Different scenarios on the type of kernel and kernel parameters for SVM & ANN structure were analyzed to obtain the best fit to the given data in previous works [6,7].

Also, in order to get performance variation information for GA model, a total of five runs for cell voltage & CCE are performed that the variation information is shown in Tables 2 and 3, respectively.

According to these results, all data sets provide a low average deviation (AD) among experimental data, ANN, GA and SVM model predictions. Also Tables 4 and 5 show the results of error analysis for prediction outputs of cell voltage & CCE from a developed ANN, GA & SVM models, respectively.

There are indicated that outputs from the GA & ANN models are more accurate than SVM model for cell voltage prediction with an AD of 1.21% & 1.27%, respectively. On the other hand, SVM model is suitable for the prediction of current efficiency with an AD (for test validation data) of 1.53%.

4.3. Effect of operating parameters on the cell performance indicators

Based on ANN, GA & SVM outputs, the effect of controllable factors on mean responses for the CA cell voltage is displayed in Fig. 11.

According to these results, cell voltage is enhanced dramatically with current density while it is altered slightly with the anolyte pH. On the other hand, the cell voltage is significantly decreased with increasing the cell temperature while only slightly with those of flow rate and brine concentration. The cell voltage is also decreased with run time.

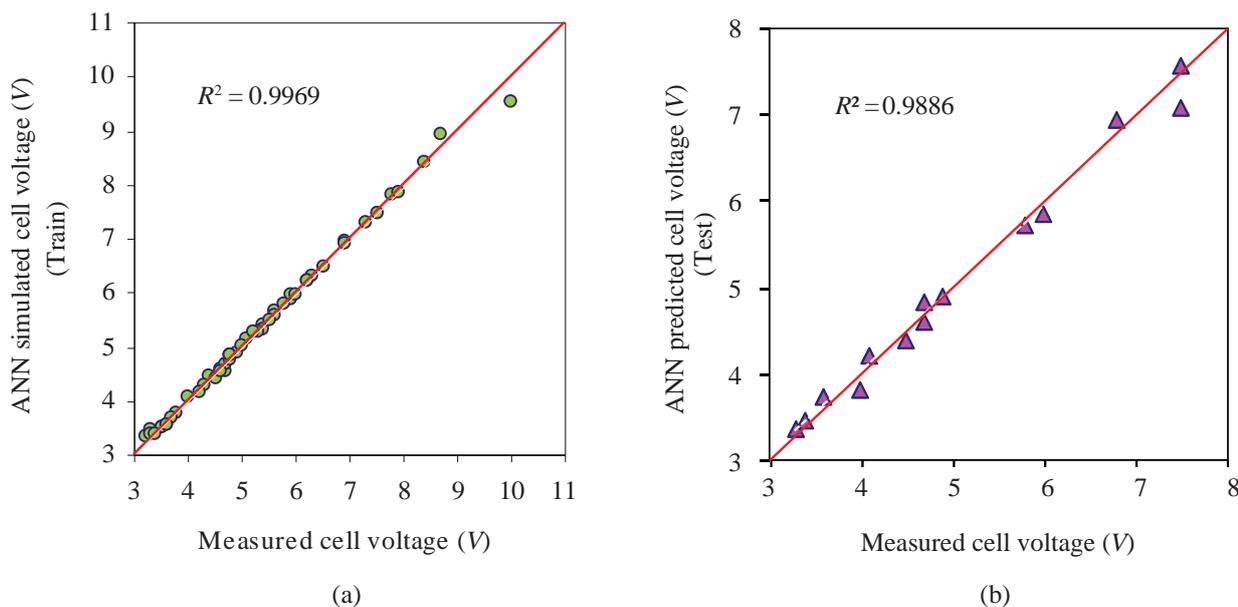


Fig. 5. The measured versus ANN-simulated for Cell Voltage (a) Train and (b) Test values.

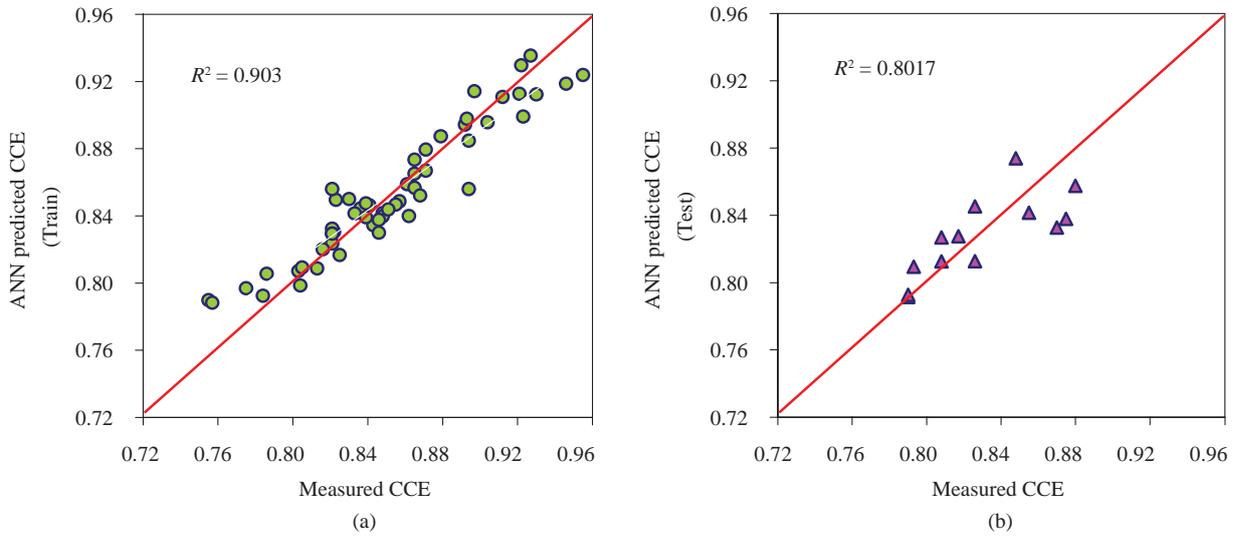


Fig. 6. The measured versus ANN-simulated for CCE (a) Train and (b) Test values.

The operating conditions to obtain a minimum value for the cell voltage simulated by ANN & SVM models are as follows: pH (2), T (90°C), Flow (5.9 cm/s), C_{brine} (300 g/L), i_p (1 kA/m^2) and run time (150 min).

Another valuable response which is directly proportional to the total energy consumed by an electrolysis cell is the current efficiency. The CCE was thus measured according to the procedures described in the experimental section and was calculated based on the following

equation [19]:

$$\eta_{\text{NaOH}} = \frac{m(t) - m(t=0)}{(It/nF) \times MW_{\text{NaOH}}} = \frac{m(t) - m(t=0)}{(It/nF) \times 40} \quad (1)$$

The impacts of various operating parameters on the CCE, based on ANN & SVM results, are shown in Fig. 12.

According to Figs. 11 and 12, the current efficiency decreases with pH due to the production of by products such as hypochlorite and chlorate in the analyte

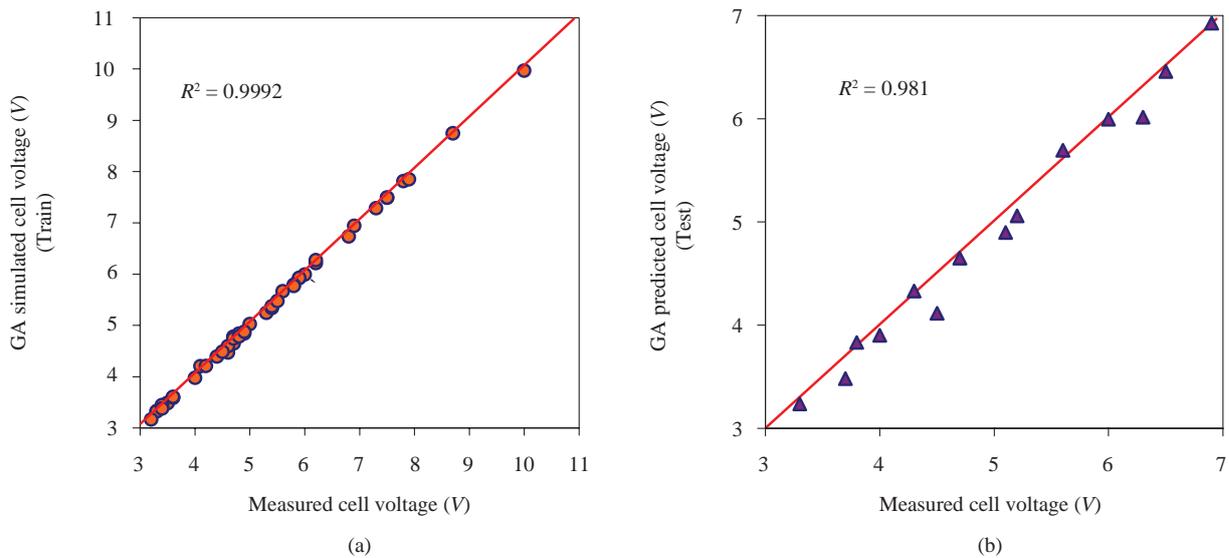


Fig. 7. The measured versus GA-simulated for Cell voltage (a) Train and (b) Test values.

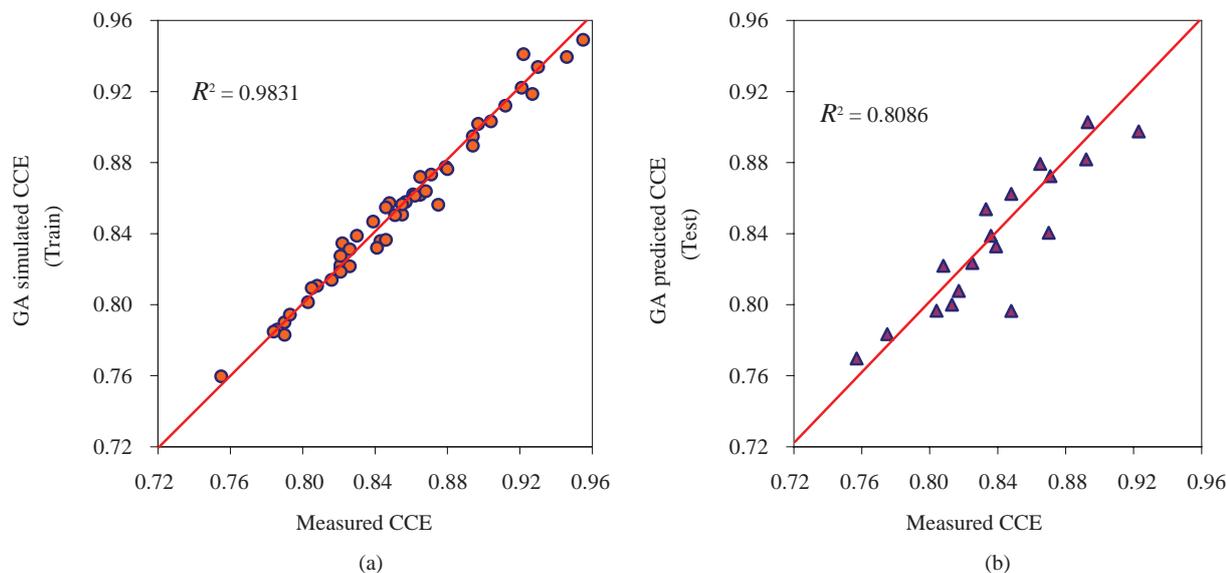


Fig. 8. The measured versus GA-simulated for CCE (a) Train and (b) Test values.

solution at higher pH while pH does not have a sensible impact on the CA cell voltage.

As it is seen, CCE improves with cell temperature due to depressing of the side reactions while the overall cell voltage decreases with temperature because of a decrease in the voltage components of the cell such as decomposition potential, IR drops and the overpotentials.

By increasing electrolyte velocity, a slight decrease in the cell voltage can be observed as shown in Fig. 11. This may be caused by a reduction in the amount of attached H_2 and Cl_2 bubbles on both sides of the membrane and the bubbles remained within the catholyte

and anolyte [20,21]. The presence of the bubbles decreases the actual conductivity of the electrolyte and thus increases the cell voltage.

The cell voltage is also decreases slightly with brine concentration within the brine concentration range studied, but the effect of brine concentration on current efficiency is pronounced, as seen in Fig. 12. This is likely to be due to suppressing the oxygen evolution as a major side reaction at low brine concentrations.

According to the data of these Figures, the current density was discovered to be the most remarkable parameter influencing the cell voltage and current

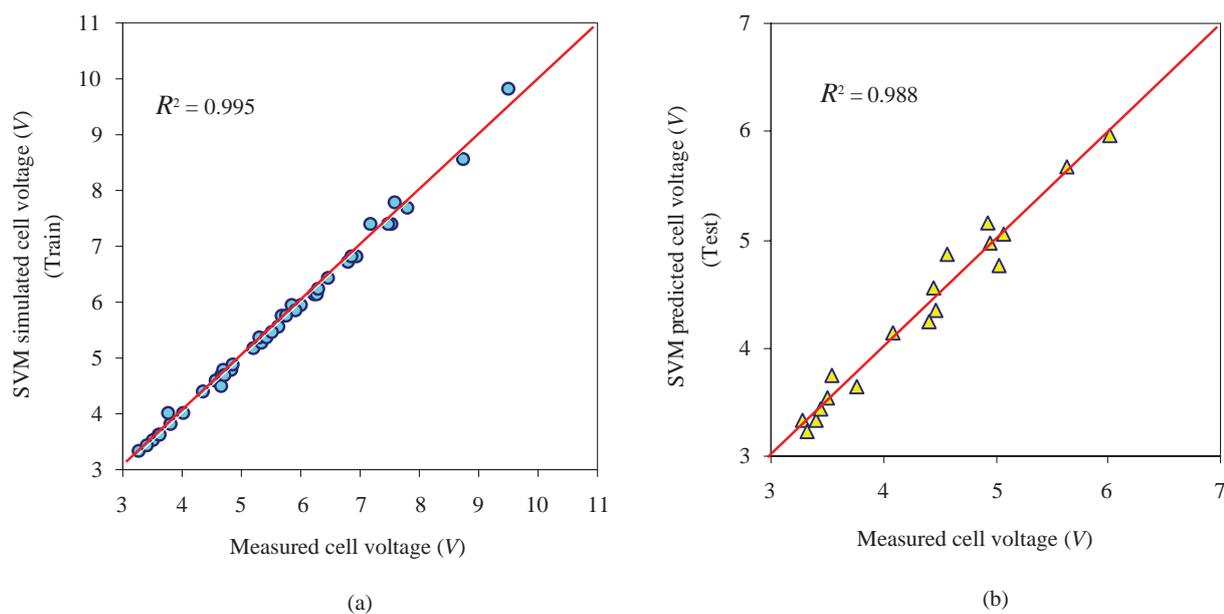


Fig. 9. The measured versus SVM-simulated for cell voltage (a) Train and (b) Test values.

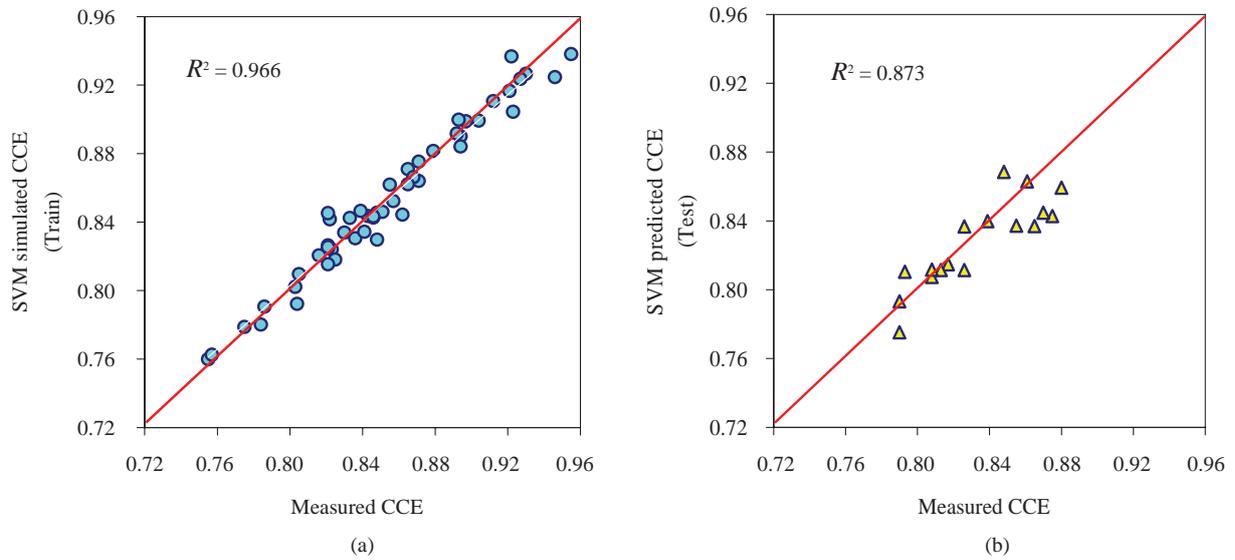


Fig. 10. The measured versus SVM-simulated for CCE (a) Train and (b) Test values.

Table 2
Performance variation information of GA for different runs of cell voltage

Run	Structure profile	Train error	Test error	Train performance	Test performance
1	RBF 4:4-16-1:1	0.099	0.194	0.155	0.300
2	Linear 5:5-1:1	0.070	0.068	0.311	0.324
3	Linear 6:6-1:1	0.070	0.066	0.310	0.315
4	MLP 5:5-5-1:1	0.022	0.052	0.122	0.297
5	MLP 6:6-10-7-1:1	0.006	0.034	0.028	0.138

efficiency. These facts were expected to consider the ohms law for the cell voltage ($\Delta V_{Cell} = I \times R_{Cell}$); and Eq. (1) describing the inverse relationship between current density and CCE.

Both functions have a downtrend with increasing run time. The lower CCE achieved at higher run times. In other words, the higher caustic concentration may be due to the OH^- back migration toward anolyte at higher run times. The catholyte conductivity enhances with NaOH concentration within the caustic

concentration range studied resulting in a decrease in the cell voltage.

5. Conclusions

Cell voltage and CCE are widely employed for improving or enhancing the performance of chlor-alkali membrane cells. Therefore, accurate prediction of CCE and cell voltage is of utmost importance. In this study novel non-parameter regression methods for the prediction of cell voltage and CCE were developed.

Table 3
Performance variation information of GA for different runs of CCE

Run	Structure profile	Train error	Test error	Train performance	Test performance
1	RBF 6:6-3-1:1	16.367	16.008	0.795	0.908
2	RBF 6:6-7-1:1	13.877	12.958	0.674	0.686
3	Linear 6:6-1:1	0.156	0.157	0.681	0.767
4	MLP 6:6-8-1:1	0.029	0.073	0.157	0.465
5	MLP 6:6-10-6-1:1	0.024	0.074	0.130	0.465

Table 4
Error analysis for prediction outputs of cell voltage

Method	Error analysis	Test validation data	Train data	Overall data
ANN [6]	AD%	2.46	0.94	1.27
	R ²	0.989	0.997	0.995
SVM [7]	AD%	2.52	1.29	1.59
	R ²	0.988	0.995	0.993
GA	AD%	2.82	0.67	1.21
	R ²	0.981	0.999	0.994

Table 5
Error analysis for prediction outputs of CCE

Method	Error analysis	Test validation data	Train data	Overall data
ANN [17]	AD%	3.50	2.27	3.14
	R ²	0.802	0.903	0.879
SVM [18]	AD%	1.53	0.79	0.98
	R ²	0.873	0.966	0.943
GA	AD%	1.77	0.52	0.87
	R ²	0.809	0.983	0.941

Based on the results obtained in this study, the following conclusions are drawn:

- Non-parameter regression methods can be used to predict the process variables & impacts of operating parameters including pH, temperature, flow rate, brine concentration, current density and run time in experimental systems such as membrane cell of chlor-alkali.
- This technology has been shown to be a useful tool not only to approximate but also to predict cell voltage and CCE versus process parameters in membrane chlor-alkali cell.
- ANN, SVM & GA models were able to predict the cell voltage values with an AD of 1.27%, 1.59% and 1.21%, respectively.
- Prediction of ANN, SVM & GA models for CCE values in test validation data have an AD of 3.50%, 1.53% and 1.77%, respectively.
- The current density and cell temperature have the highest effect values on the cell voltage.
- Based on simulated outputs, the following operating conditions are proposed to maximize the current efficiency and minimize the cell voltage: pH = 2; Temperature = 90°C; flow rate = 5.9 cm/s; brine concentration = 300 g/L; and current density = 1 kA/m².

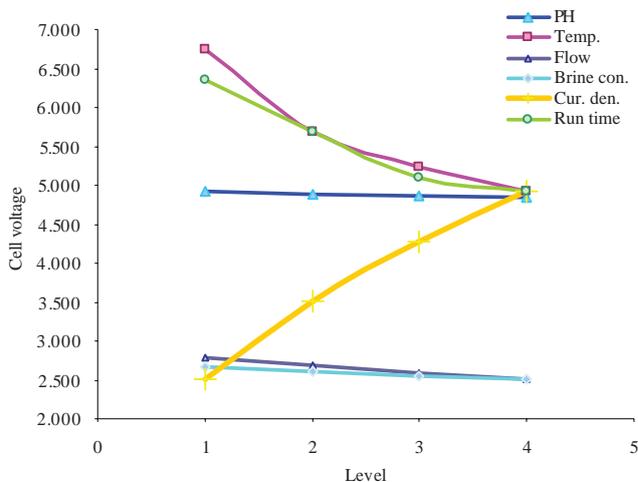


Fig. 11. Effect of various operating parameters on the cell voltage resulted from the models.

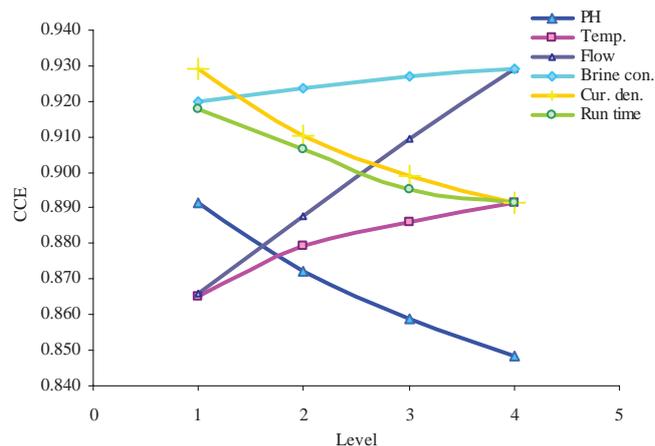


Fig. 12. Effect of various operating parameters on the CCE resulted from the ANN & SVM based models.

- The comparison between GA model in this work and ANN & SVM modes from previous works [17,18], is shown that developed GA & ANN models can forecast the cell voltage, and the developed SVM model can predict CCE values, more accurately.

Acknowledgements

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Symbols

- AD Average deviation = $\frac{1}{n} \sum_{i=1}^n \left(\frac{y_{\text{exp.}} - y_{\text{cal.}}}{y_{\text{exp.}}} \right) \times 100$
- C_{brine} Brine concentration, g/L

F	Faraday's constant (96,458 C/mol)
F	Electrolyte velocity, cm/s
I	Current, kA
$m(t = 0)$	Mass of initial caustic soda, 5 g
$m(t)$	Mass of produced caustic at time t , g
MwNaOH	Caustic molecular weight, 40 g/mol
N	Number of exchange mole electron
R^2	R-squared value
t	Run time, s
X, X_1, \dots	Independent or predictor variables
X_p	
$y(x)$	Artificial neural network output
η_{NaOH}	Caustic current efficiency

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