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Investigation on the synthesis conditions at the interpore distance of nanoporous anodic aluminum oxide: A comparison of experimental study, artificial neural network, and multiple linear regression

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ABSTRACT

Using nanoporous anodic aluminum oxide thin layer becomes more popular in recent years due to its capability to be a membrane in some engineering applications. The main purpose of this paper is to investigate the synthesis conditions at the interpore distance of nanoporous anodic aluminum oxide through an experimental study, an artificial neural network (ANN), and a multiple linear regression (MLR) model. A total of 33 experimental data used to establish both models. The models have three inputs including the concentration of electrolyte, temperature, and applied voltage. The interpore distance of nanoporous anodic aluminum oxide is considered as output in the models. The results of the models are compared with the results of experimental study and an empirical formula proposed by Nielsch. The results reveal that the proposed models have good prediction capability with acceptable errors. However, in this research, the proposed ANN model is accurate than the MLR analysis and both of them are better than empirical formula. The proposed models can also predict the results of experimental study successfully.

1. Introduction

Nanoporous anodic aluminum oxide (AAO) had attracted intensive interest due to its potential to use as a membrane in some applications such as the gas separation [1–4], drug delivery [5,6], and bone fixation [7]. Anodizing aluminum in an acidic electrolyte resulted in a thin layer of compact aluminum oxide, following by an ordered array of nanopores [8–10]. The synthesis process led to mechanically robust and thermally durable [3] and inert, wide and variety of pore size distribution, which showed unique capability for biomolecule separation process [11] and hemodialysis [12–15].

The geometry of aluminum oxide layer executed important role for a separation process which could lead to maximize permeation and flux across nanoporous anodic aluminum oxide membrane. For example, as a hemodialysis membrane, a pore size was desirable which had capability to clear urea, creatinine, vancomycin and inulin as a waste product with small and middle of molecular weight while maintaining large molecular weight solutes (albumin) [16–21].

The geometry of nanoporous anodic aluminum oxide had grown through anodization was represented schematically in Fig. 1.

Many equations were suggested for correlation between the geometry of aluminum oxide layer and conditions of anodized aluminum [22–25]. According to Nielsch et al. [22], the interpore distance (D_c) in nanometer (nm) was linearly proportional to the applied voltage (U) in volts (V) of the steady-state growth of oxide layer as follows:

$$D_c = \lambda_c U \quad (\lambda_c \approx 2.5\ \mathrm{nm\,V^{-1}}) \tag{1}$$

where λ_c is a proportional constant in nm V^{-1}.

It was assumed that the pore diameter was a function of applied voltage. The other efforts showed that anodizing temperature and concentration of electrolyte cause changing the interpore distance [23–25]. In this regard, three important effecting parameters on the interpore distance of nanoporous aluminum oxide were identified as following:

- Concentration of electrolyte (C) in mol/dm^3.
- Anodizing temperature (T) in K.
- Applied voltage (U) in V.

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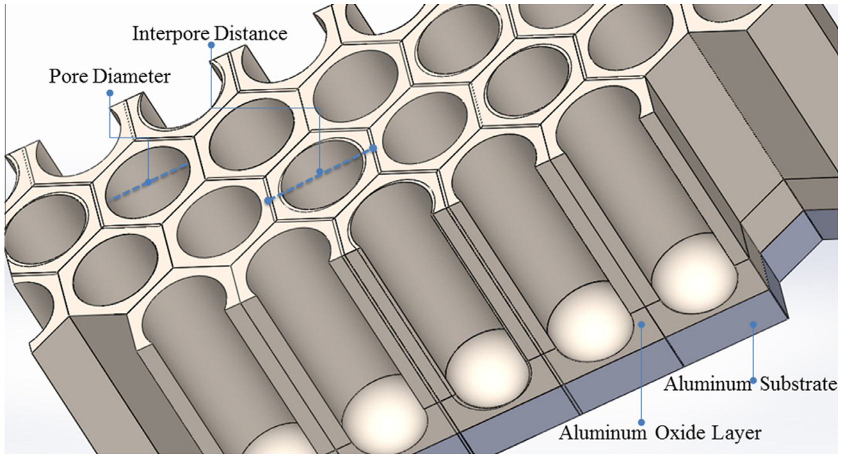

Fig. 1. The schematic geometry of nanoporous anodic aluminum oxide layer and aluminum substrate.

In general, these parameters had a considerable effect on the interpore distance of nanoporous aluminum oxide, but the contribution of each parameter separately was studied a little. Nowadays, computer-based methods such as artificial neural networks (ANNs) and multiple linear regression analysis have attracted some attentions to be replaced with high-cost experimental studies. The above-mentioned techniques were used in different fields of engineering applications such as civil engineering [26–29], chemical engineering [30,31] and material science [32–35].

The artificial neural networks (ANNs) are known as the important and successful simulating tools of input–output datasets. The most fundamental part of such networks is the training process. The ANN model is trained with some relevant experimental results and then can predict the output with an accepted error.

This paper presents an artificial neural network (ANN) and a multiple linear regression (MLR) model accompanished with an experimental study to investigate the synthesis conditions at the interpore distance of nanoporous anodic aluminum oxide. In this regard, a total of 33 experimental records are used with three factors each. The results reveal that output in the models have an agreement with experimental records. In addition, all results are compared with output of formula proposed by Nielsch [22]. It is clear that ANN is more accurate than formula proposed by Nielsch [22] and MLR model.

2. Experimental study

A high purity aluminum foil (99.99% Alfa Aesar) was used as the primary material. At first, the aluminum substrate was degreased by Acetone, and then the native oxide layer upon the aluminum foil was removed through sinking the sample in a caustic solution (3 mol/dm^3 (M)) for 30 s. A self-organized two-step anodization was used to fabricate the thin film layer of anodic aluminum oxide. The simple electrochemical cell was equipped with a magnetic stirrer under the constant temperature by the powerful circulator system (Thermo Haake, DC 10-K15) and a platinum electrode was employed as a counter electrode. The distance between the anode and cathode was around 3 cm. The first step of anodization was conducted on an oxalic acid electrolyte (0.3 M) at a constant potential of 40 V. This process lasted 2 hours (h) while anodizing temperature was constant at 27 °C. The chemical etching procedure was used to remove thin anodic aluminum oxide with the presence of 6 and 1.8 wt% of H_3PO_4 and $H_2Cr_2O_4$ at 75 °C for 3 h, respectively. After then, the second anodization was performed in the same experimental condition and this step lasted 3 h. All solutions were prepared from reagent grade chemicals and deionized water. Prior to each step, the aluminum sample was ultrasonically cleaned using Acetone for 15 minute (min) following with a 5 min-ultrasonic cleaning by deionized water.

The geometrical characterization of nanoporous anodic aluminum oxide was done by a Field Emission Electron Scanning Microscope (FE-SEM, Hitachi SE-4160) after sputtering a 15-nm thickness of Au layer.

3. Data collection

The objective of this research is to develop an artificial neural network (ANN) and a multiple linear regression analysis (MLR) for predicting the interpore distance of nanoporous anodic aluminum oxide. The first step is data gathering for the training, validating, and testing process of the ANN model. All data are collected from the literature [22,36–48] and self-carried out experimental study.

In total, 33 records are collected from the literature. Of these; 19, 7, and 7 records are used for training, validating, and testing of the model, respectively. The selection process is done randomly to reduce hand-selection errors. Table 1 summarizes input and output ranges for the ANN model.

4. Artificial neural network

Artificial neural network modeling as a computer-based methodology is developed in an attempt to imitate the obtained knowledge and skills of the human brain. It offers considerable support in terms of organizing, classifying, and summarizing data. It also helps to distinguish among input data and gives a high degree of prediction accuracy because of requiring a few assumptions. These characteristics make neural network approach an important tool for forecasting some complex problems. Here, a brief description of ANN is presented to help readers [49].

Table 1
The range of parameters in collected database.

Variables	Minimum	Mean	Maximum	Standard deviation
C (M)	0.1	0.5309	2.4	0.4669
T (K)	273	280.576	300	8.4317
U (V)	12.5	45.5303	195	43.6791