ANN approach in the creation of thin gas-sensitive films based on modified polyacrylonitrile

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Abstract. In this study, the dependence gas sensitivity coefficient of silver-containing polyacrylonitrile (PAN) films on the technological parameters using artificial neural networks (ANN) is studied. Modelling is performed based on 38 datasets at different conditions. Initially, the experimental data on the fabrication of the films with gas-sensing properties have been collected, which constitutes the simulation space. At the next stage, the analysis of the measurement results has been carried out, self-tuning and training of the neural network have been done to provide the required output signal. Using the neural network model, the optimal technological parameters for creating efficient gas sensors for nitrogen dioxide have been established. Verification of the model was performed: the data convergence was 8-13%.

1. Introduction
In the direction of developing criteria for creating gas-sensitive layers of gas sensors with predetermined properties, intensive research is being carried out. The forecast of properties of materials of a sensitive layer allows reducing considerably expenses of time and means at the creation of sensors. In connection with this, it is proposed to use an approach based on the principles of modelling gas sensitivity characteristics from data on technological regimes of material formation. Since the method of obtaining a nanostructure has a strong influence on the evolution of the properties of materials [1,2].

Criteria for creating sensors with specified properties have not defined yet. Forecasting the properties of substances can significantly reduce the time and expense of creating substances with certain properties. In this regard, it is proposed to use an approach based on the principles of modelling gas-sensitive characteristics by technological data. Based on the parameters of the process, such as temperature, annealing time, etc., the gas sensitivity coefficient is modelled.

In this study to simulate the gas-sensitive properties of metal-containing polyacrylonitrile (PAN), artificial neural networks (ANN) are used, which allow obtaining rather complicated control laws to show the influence of technological parameters of forming the PAN films on their electrophysical and gas-sensitive properties. The advantage of sensors based on electrically conductive polyconjugated organic polymers is their functioning under normal conditions, which makes it possible to create unheated gas sensors [1].

2. Experiment
One of the simplest ways to predict properties is the multiple linear regression method, which presents the results of observations with a polynomial of the form:

\[ y = b_0 + b_1 \cdot x_1 + b_2 \cdot x_2 + b_3 \cdot x_3 + b_4 \cdot x_4. \] (1)
The Method of Least Squares was used to calculate the coefficients of the linear regression equation
\( b_0, b_1, b_2, b_3, b_4 \), which provides the minimum sum of squared deviations of the experimental data from
the values calculated from the regression equation. The main goal of multiple regression is to build a
model with a large number of factors that determine the influence of each of them individually, as well
as the totality of their influence on the model indicator.

Descriptors for modelling are the technological parameters for fabricating films (temperature and
time of the first and second IR annealing stages, weight concentration of metal in the composition of
PAN films). To select descriptors, we considered the values of the parameters obtained experimentally,
as well as the parameters representing functions of the form \( X_i = g(x_i) \), where \( g \) are the elementary
functions \( (e^x, \sin(x), \cos(x), \ln(x), 1/x) \).

The Method of Least Squares was used to process the experimental data to establish the relationship
between the resistance and the technological parameters of the formation of the material based on the
films of metal-containing PAN. The Least Squares calculation was implemented using the Maple 12
software package.

As a result, the following equation is obtained:

\[
S = -0.29m + 0.0002T_1 + 0.019 t_1 - 0.0016 T_2 + 0.013 t_2 + c,
\]

where \( S \) is the gas sensitivity of the sample, defined as the ratio of the film resistivity before and after
the introduction of the detectable gas \( R/\overline{R} \); technological parameters: \( m \) – weight concentration of silver (%), \( T_1, t_1 \) - temperature and time of the first IR annealing stage, \( T_2, t_2 \) - temperature and time of the
second IR annealing stage; \( c \) is a free term.

Statistics results: coefficient of determination \( r^2 = 0.26 \); Fisher's criterion \( F = 2.46 \); the explained
variance is \( \nu = 0.15 \); the standard deviation \( s = 0.22 \).

Prediction of properties can also be carried out with the help of ANN, which is a simplified
mathematical model of information processing by the human brain. In prediction tasks, time series
representing the values of controlled variables over a certain time interval are used as input signals. The
output signal is a set of variables that is a subset of the variables of the input signal.

According to the general rules for constructing neural network models, the experimental data used
in the training of ANN do not undergo any preliminary processing, in contrast to the regression analysis.
The prediction of the properties of objects using structural data using the neural network approach has
been successfully applied in various fields of physical research [3-17]. At the same time, the necessary
amount of data required for learning a neural network can be substantially less than for constructing
a regression dependence. For example, in [4] modelling is performed based on 90 datasets at different
operating conditions. Two types of ANN [10], multilayer perceptron (MLP) and generalized regression
neural network (GRNN) were used to test the granulation process in a fluidized bed using a small number
of data sets (53 trials).

In [18] provides a mathematical justification for the possibility of approximation by networks of the
"multilayer perceptron" type. Taking into account the ability of a multilayer perceptron to generalize
and filter noise, we can talk about the possibility of its use in regression analysis problems. Multilayer
networks are distinguished by the fact that between the input and output data there are several so-called
hidden layers of neurons adding more nonlinear bonds to the model. Thus, the hidden layers further
transform the information and add nonlinearities to the model.

One of the problems with using neural networks is the impossibility of preliminary determining
the optimal number of hidden layers and neurons in them. If the number of neurons is too small, then it is
equivalent to the loss of some nonlinear connections in the model, if there are a lot of neurons, this can
lead to a "retraining" of the network, that is, it simply "learns" the data, and does not recognize their
structure. After choosing a specific topology, it is necessary to select the parameters for training the
neural network. From the correct choice of parameters depends not only how quickly the network
answers will converge to the correct answers. For example, choosing a low learning rate will increase
the convergence time, but sometimes avoid the paralysis of the network. During the learning process,
the network in a certain order scans the training sample, choosing the optimal values of the weights. The
activation function limits the amplitude of the output signal of the neuron. To simulate the dependence
of the gas sensitivity factor on technological parameters, it is proposed to use a neural network with direct signal propagation (multilayer perceptron).

The use of a neural network approach for predicting the properties of objects in different areas of research is presented in Table 1.

### Table 1. Using a neural network approach for predicting the properties of objects

| ANN                        | Object                                      | Data used as input variables                                                                                                                                                                                                 | Validation                              | Ref.  |
|---------------------------|---------------------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------------------------|-------|
| Multilayer Perceptron     | to predict the solar irradiance             | solar irradiance and air temperature data                                                                                                                                                                               | K-fold cross-validation                | [3]   |
| MLP-model                 |                                             | the values of chemical reagents dosage, froth height, air and wash water flow rates, gas holdup and Cu, Mo grades in the rougher feed and flotation column feed, column tail and final concentrate streams | cross-validation technique              |       |
| Multivariate Non-Linear   |                                             | to estimate the metallurgical performance of Cu and Mo respectively in a flotation column                                                                                                                                    |                                         |       |
| Regression (MNLNR),       |                                             |                                                                                                                                                                |                                         |       |
| multi-layer perceptron    |                                             |                                                                                                                                                                |                                         |       |
| (MLP) model, sigmoid      |                                             |                                                                                                                                                                |                                         |       |
| activation function and   |                                             |                                                                                                                                                                |                                         |       |
| Levenberg-Marquardt       |                                             |                                                                                                                                                                |                                         |       |
| learning rule             |                                             |                                                                                                                                                                |                                         |       |
| Multi-Layer Perceptron    |                                             | The variations of the ratio of binder solution to feed material, product bed temperature, atomizing air pressure, binder spray rate, air velocity and batch size | a leave-one-out cross-validation process| [10]  |
| (MLP) and a Generalized   |                                             |                                                                                                                                                                |                                         |       |
| Regression Neural Network  |                                             |                                                                                                                                                                |                                         |       |
| (GRNN)                    |                                             |                                                                                                                                                                |                                         |       |
| as a mean of developing   |                                             | parameters for the feed stream included flow rate, conductivity, feed pressure, pH and temperature                                                              | the internal validation step served as  | [16]  |
| data-driven models for    |                                             |                                                                                                                                                                | a stopping criterion for the ANN      |       |
| forecasting reverse       |                                             |                                                                                                                                                                | learning algorithm                     |       |
| osmosis (RO) plant        |                                             |                                                                                                                                                                |                                         |       |
| performance and potential |                                             |                                                                                                                                                                |                                         |       |
| use for operational       |                                             |                                                                                                                                                                |                                         |       |
| diagnostics               |                                             |                                                                                                                                                                |                                         |       |

Stages of modelling largely depend on the simulation object, but you can identify the general sequence of steps required for any modelled object (Fig. 1).

![Formulation of the problem](image)

**Figure 1.** Stages of modelling ANN for the purposes to predict the properties of objects

At I step of data preparation, an informative set $x \in \mathbb{R}^n$ and output $y \in \mathbb{R}^r$ quantities are constructed, the relationship between which will be modelled using a neural network. When modelling with neural networks, the choice of output attributes is important, since the incorrect selection of such characteristics can adversely affect the quality of the model [19].

In our case, when predicting the physicochemical properties of objects by technological parameters, this stage consists of collecting data during the experiment. Technological parameters are predetermined by an expert and determined from experience and analysis of the dependence of the PAN films properties on the conditions of the technological process of film fabrication. Further, a set of pairs $\{x_i, y_i\}$, are
formed, reflecting the real behaviour of the simulated system, which is determined during the experiment.

At the II step of preliminary data processing, data preparation is carried out: purification and transformation of information. The data collected as a result of the experiment, as a rule, are "raw", they must be processed: edit the gaps and abnormal emissions, eliminate contradictions, if necessary, transform into numerical data, eliminate insignificant factors, etc. The quality of the collected data, their completeness and reliability in many respects determine the choice of the type of model [3-17].

The data are normalized since the neural network activation function has the saturation property: starting with certain input values, the function will produce almost the same value. The neural network operates with numerical data, usually represented in the interval [0; 1]. There are different methods of normalization.

For example, linear scaling: for each j-th column, a range of values $[x_{j}^{min}, x_{j}^{max}]$, where

$$x_{j}^{min} = \min_{i=1}^{k} x_{ij} \quad \text{and} \quad x_{j}^{max} = \max_{i=1}^{k} x_{ij};$$

$$\tilde{x}_{ij} = \frac{x_{ij} - x_{j}^{min}}{x_{j}^{max} - x_{j}^{min}}$$

(3)

Note that normalization is not necessary, however, when using it, the output values to the user must necessarily be denormalized using reverse operations.

The main stage of prediction with the help of neural networks is the construction of a model, which includes two components: structural and parametric identification [20]. At the stage of structural identification, the question of the number of layers and neurons in layers is solved. At this stage, it is necessary to solve the problem connected with the fact that the increase in the number of layers and neurons in the network, although it leads to a reduction in learning error, can lead to retraining. The creation of the optimal complexity model is achieved in the selection of a test set from the set of input-output pre-processed data. The test set consists of data that is not used in the learning process. Usually, 20% of pairs are randomly selected.

The parametric identification stage consists in training the network, which consists of the selection of weights. The selection of network weights in the learning process is aimed at finding a combination of their values that best reproduces the sequence of expected training pairs $\{x_{i}, y_{i}\}$ [21]. There is a close relationship between the number of network weights and the number of training samples. To acquire the ability to generalize information, the network trains on an excessive set of data, since then the weights will not be adapted to unique samples, but their statistically averaged populations. The test sample should contain both positive and negative examples.

The optimal neural network structure and tuning of the learning algorithm are selected to achieve a minimum of the generalization error. The degree of correspondence of the synthesized model to the real object to exclude the error of the decision is confirmed on an independent test sample. A common way to confirm the adequacy of the synthesized model is to test the corresponding hypothesis based on statistical criteria. The evaluation procedure is based on a comparison of measurements on a real system and the results of experiments on a model [19-22].

The next important step is to assess the stability of the model – its ability to maintain adequacy at all possible levels of input parameters, as well as when making changes to the system configuration. The sensitivity of the model to the change of internal parameters – the weights of the inter-neural connections, is estimated through the quality functional or network error function. A good neural network model usually has for each parameter a relative stability corridor, within which the quality functional changes insignificantly due to the distribution of computing functions between network neurons.

The object of the study was silver-containing PAN films. The prepared film-forming solutions were applied to polycor substrates, which were previously degreased by boiling in isopropyl alcohol for 10 minutes. Then a part of the obtained samples was dried in the oven for 30 minutes at a temperature of 160°C. The samples were then allowed to stand for 24 hours at room temperature until they cooled down
completely. The other part of the samples in the oven was not dried but only kept in air for 24 hours at room temperature.

To fabricate the gas-sensitive layer, heating was used by incoherent IR radiation with a low vacuum \((8 \times 10^{-2} \text{ mm Hg})\). IR-annealing of samples was carried out in two stages: at a lower temperature for the preliminary structuring of PAN to form a system of conjugated -C = N- bonds and annealing at a higher temperature, during which the process of formation of both -C = N- and -C = C-bonds is intensified to form a polyconjugated chain polymer [23]. The intensity of the radiation in the first stage of IR annealing corresponded to temperatures of 250°C and 300°C, and the radiation intensity in the second stage of IR annealing was 250-500°C. The time of exposure to IR radiation at each temperature was varied (2 ÷ 20 minutes). The weight concentration of the modifying additive varied in the range of 0 ÷ 3 %.

The number of fabricated samples of Ag/PAN films was 38.

To analyze the experimental data, the neural network approach was applied, the software application Statistica Neural Networks 4.0 was used. To construct the neural network model, we used an ANN a two-layer perceptron model (Fig. 2), containing 5 input and 1 output neurons. Each input neuron corresponded to one of the technological parameters: temperature and time of the first and second annealing steps, as well as the weight concentration of the modifying additive \((\omega, \%)\); output – the coefficient of gas sensitivity. The data received as a result of training the network was processed using the program Statistica 6.0.

![Two-layer perceptron neural network](image)

The experimental data was divided randomly into 3 the training sets: learning, control and test (Table 2).

| №   | \(\omega, \%\) | \(T_{1}, ^{\circ} \text{C}\) | \(t_{1}, \text{ minute}\) | \(T_{2}, ^{\circ} \text{C}\) | \(t_{2}, \text{ minute}\) | \(\ln(S)\)     | set         |
|-----|----------------|-----------------------------|---------------------------|-----------------------------|---------------------------|---------------|-------------|
| 1   | 1.5            | 300                         | 5                         | 500                         | 20                        | 0.116534      | learning    |
| 2   | 1              | 300                         | 10                        | 400                         | 2                         | 0.001005      | learning    |
| 3   | 0.5            | 300                         | 10                        | 400                         | 2                         | 0.116534      | test        |
| 4   | 0.1            | 300                         | 15                        | 500                         | 5                         | 0.174353      | test        |
| 5   | 0.1            | 300                         | 10                        | 400                         | 2                         | 0.446287      | test        |
| 6   | 0.07           | 150                         | 3                         | 400                         | 2                         | 0.07257       | control     |
| 7   | 0.05           | 300                         | 10                        | 400                         | 2                         | 0.510826      | learning    |
| 8   | 0.05           | 300                         | 20                        | 515                         | 2                         | 0.235722      | control     |
| 9   | 0.02           | 300                         | 10                        | 400                         | 2                         | 0.494296      | learning    |
| 10  | 3              | 200                         | 10                        | 500                         | 10                        | 0.07257       | learning    |
| 11  | 1              | 200                         | 10                        | 500                         | 10                        | 0.061875      | test        |
| 12  | 0.5            | 200                         | 10                        | 500                         | 10                        | 0.04082       | learning    |
| 13  | 0.2            | 300                         | 10                        | 500                         | 5                         | 0.127833      | learning    |
| 14  | 0.2            | 300                         | 5                         | 500                         | 5                         | 0.105361      | learning    |
| 15  | 0.1            | 300                         | 20                        | 500                         | 20                        | 0.027371      | learning    |
| 16  | 0.1            | 300                         | 20                        | 500                         | 5                         | 0.116534      | control     |
| 17  | 0              | 200                         | 15                        | 500                         | 15                        | 0.755023      | control     |
| 18  | 0              | 200                         | 60                        | 500                         | 15                        | 1.139434      | learning    |
The choice of the number of layers of the ANN and the learning algorithm was carried out using 6 methods: backpropagation algorithm, Conjugate Gradients (CG) algorithm, Quasi-Newton (QN) algorithm, Levenberg-Marquardt learning rule, Radial Basis Function Networks (RBFN), Delta Bar Delta algorithm. Tables 3, 4 show the choice of training method and the number of neurons in the hidden layers.

**Table 3.** Values of statistical parameters for a two-layer network with a different number of neurons (training method-conjugate gradients)

| 1 layer | 2 layer | Statistical parameters |
|---------|---------|------------------------|
| Number of neurons | R | s_r | s_s |
| 1 | 3 | 5 | 0.76 | 0.135 | 0.35 |
| 2 | 4 | 5 | 0.78 | 0.2 | 0.135 |
| 3 | 5 | 5 | 0.79 | 0.188 | 0.11 |
| 4 | 6 | 5 | 0.82 | 0.186 | 0.12 |
| 5 | 7 | 5 | 0.81 | 0.16 | 0.12 |
| 6 | 8 | 5 | 0.96 | 0.05 | 0.09 |
| 7 | 9 | 5 | 0.77 | 0.21 | 0.08 |
| 8 | 10 | 5 | 0.81 | 0.18 | 0.16 |
| 9 | 11 | 5 | 0.84 | 0.15 | 0.13 |
| 10 | 12 | 5 | 0.78 | 0.17 | 0.09 |
| 11 | 8 | 0 | 0.89 | 0.12 | 0.09 |
| 12 | 8 | 1 | 0.92 | 0.1 | 0.14 |
| 13 | 8 | 2 | 0.8 | 0.18 | 0.1 |
| 14 | 8 | 3 | 0.93 | 0.06 | 0.15 |
| 15 | 8 | 4 | 0.93 | 0.11 | 0.1 |
| 16 | 8 | 6 | 0.8 | 0.02 | 0.15 |

Experimental data were normalized, 1 drop-out point was deleted. The data were normalized according to formula (3). Data on gas sensitivity did not need to be normalized since The value of S is in the interval [0, 1].
The lowest mean square error and the largest correlation coefficient were found in the ANN with 8 neurons in the first hidden layer and 5 neurons in the second hidden layer, the learning algorithm is the conjugate gradient method.

**Table 4.** The values of the statistical parameters for a two-layer network (the number of neurons in the first layer – 8, in the second layer – 5)

| Algorithm                  | \(R\)  | \(s_t\) | \(s_v\) |
|----------------------------|--------|--------|--------|
| backpropagation            | 0.78   | 0.17   | 0.14   |
| Conjugate Gradients        | 0.96   | 0.05   | 0.09   |
| Quasi-Newton               | 0.74   | 0.22   | 0.13   |
| Levenberg-Marquardt        | 0.83   | 0.03   | 0.22   |
| Radial Basis Function Networks | 0.95   | 0.06   | 0.09   |
| Delta Bar Delta            | 0.94   | 0.07   | 0.05   |

The quality of the ANN operation was determined by the root-mean-square error in predicting the values of the property on the learning sample \(s_t\), the correlation coefficient between the predicted and experimental values of the property on the training sample \(R\) and the standard error of the forecast in the test sample \(s_v\): \(s_t = 0.05, R = 0.96, s_v = 0.09\).

The correlation coefficient \(R\) determines the attributable fraction of the spread, \(R^2\) is the fraction of the total spread relative to the gas sensitivity \(S\), due to regression \((R^2 = 0.86)\). The fact that the model explains by 86% the spread of data relative to the average, allows us to speak about the reliability of the model.

The root-mean-square errors of training and test samples \((s_t = 0.05, s_v = 0.09)\) are approximately the same, which excludes retraining of the network. The accuracy of the calculations in the "learning" group and the "test" subgroup is the same, which indicates the stability of the model.

The sensitivity of the model was estimated using the mean square error of all data \(s = 0.09\).

The equation of the correlation dependence between the calculated and experimental values of the gas sensitivity of silver-containing PAN films is compiled:

\[
S_{\text{calculation}} = 0.913S_{\text{experiment}} + 0.1. \tag{4}
\]

It can be seen that the correlation line practically coincides with the graph, which has a tangent of the slope angle equal to one (Fig. 3). Thus, the applied method can be used to predict the sample's gas sensitivity, which will allow us to narrow the search area of the initially specified process parameters.

![Diagram of scattering of experimental and calculated values of the gas sensitivity coefficient of Ag/PAN films](image)

To test the adequacy of the model, it was tested. The optimum set of technological parameters \(\omega(\text{Ag}) = 0.05\%\), \(T_{\text{drying}} = 160^\circ\text{C}\), \(t_{\text{drying}} = 30\) minutes, \(T_1 = 250^\circ\text{C}\), \(t_1 = 2\) minutes, \(T_2 = 350^\circ\text{C}\), \(t_2 = 28\) minutes. According to the model, its experimental verification was carried out (Table 5).
Table 5. Results of testing the data of neural network modelling of the gas sensitivity coefficient

| ω (Ag), % | T\text{drying} – t\text{drying}, °C – min. | T_1 – t_1, °C – min. | T_2 – t_2, °C – min. | Gas sensitivity coefficient S, r.u. | Absolute and relative errors |
|----------|---------------------------------|-------------------|-------------------|-------------------------------|-----------------------------|
|          |                                 |                   |                   | calculation | experiment | δ (%) | Δ, % |
| 0.05     | 160 – 30                        | 250 – 2           | 350 – 28          | 0.75         | 0.69       | 0.06  | 8    |
| 0.75     | 160 – 30                        | 300 – 2           | 500 – 20          | 0             | 0          | 0     | 0    |
| 0.75     | 160 – 30                        | 150 – 3           | 400 – 2           | 0.13          | 0.15       | 0.02  | 13   |

3. Conclusion

A neural network model of the gas sensitivity of Ag-containing PAN films was constructed using the technological parameters of the formation of the film material (input data: temperature and time regimes of the first and second stages of IR annealing and the weight concentration of the modifying additive). Verification of the model allowed to prove the adequacy of the synthesized neural network.

It is shown that the use of the neural network approach allows to reduce the experiment and establish optimal technological parameters for obtaining gas-sensitive materials to create efficient gas sensors.

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