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Oil Extraction from Pistacia Khinjuk - Experimental and Prediction by Computational Intelligence Models

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ABSTRACT: This study investigates the oil extraction from Pistacia Khinjuk by the application of enzyme. Artificial Neural Network (ANN) and Adaptive Neuro Fuzzy Inference System (ANFIS) were applied for modeling and prediction of oil extraction yield. 16 data points were collected and the ANN was trained with one hidden layer using various numbers of neurons. A two-layered ANN provides the best results, using application of ten neurons in the hidden layer. Moreover, process optimization were carried out by using both methods to predict the best operating conditions which resulted in the maximum extraction yield of the Pistacia Khinjuk. The maximum extraction yield of Pistacia Khinjuk was estimated by ANN method to be 56.52% under the operational conditions of temperature and enzyme concentration of 0.27, pH of 6, and the Ultrasonic time of 4.23 h, while the optimum oil extraction yield by ANFIS method was 55.8% by applying the operational circumstances of enzyme concentration of 0.30, pH of 6.5, and the Ultrasonic time of 4.55 h. In addition, mean-squared-error (MSE) and relative error methods were utilized to compare the predicted values of the oil extraction yield obtained for both models with the experimental data. The results of the comparisons revealed the superiority of ANN model as compared to ANFIS model.

Keywords: *Artificial Neural Network, Adaptive Neuro Fuzzy Inference System, Modeling, Optimization, Pistacia Khinjuk.*

Introduction

Pistacia is a genus of flowering plants and belongs to the family of Anacardiaceae (Shuraki & Sedgley, 1994), which comprises 11 species (Zohary *et al.,* 1996). Among them, Pistacia vera L., Pistacia atlantica subsp. mutica (Fisch. & C. A. Mey.). Pistacia mutica and Pistacia khinjuk Stocks, are the species that occur in Iran (Razavi, 2006). Pistacia vera has economical importance and its cultivation, as a traditional nut crop is extended to the dry land areas of the country. P. vera and P. khinjuk are the most primitive species and also postulated that P. khinjuk was directly descended from P. vera (Zohary *et al.,* 1996) as a bridge to other Pistacia species.

The largest producer of Pistacia spp. in the world is Iran, with over 44% of the world production, therefore, a few places such as Zagros Mountains, where wild pistachio persists in natural and extensively managed (i.e., semi-natural) stands (Razavi, 2006). They are the most important types of pistachio and for this reason, Iran is known as the origin of pistachios. Therefore the Pistacia khinjuk seed would be as a novel

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source of plant oil for the pharmaceutical industries.The essential oil extract of P. khinjuk seed showed antihelminthic effect against protoscoleces of E. granulosus and anti-echinococcal activity. The oil from plant seeds is conventionally extracted by either mechanical pressing or solvent extraction (SE) (Taran *et al.,* 2009; Mani *et al.,* 2007). Mechanical pressing is a very efficient process, leading to low oil recovery. In spite of the high efficiency of solvent extraction, this method suffers from poor quality of protein in oil cake (meal), high investment, and energy requirements. The commercial solvent for SE process is hexane which is listed among hazardous air pollutants associated with neurological and respiratory disorders on prolonged exposure (the International Standard Organization permits only 50 ppm residual hexane in oil seed meal) (Sharma *et al.,* 2001). It therefore is vital to explore alternative safe and efficient method of oil extraction.

Aqueous enzymatic extractions are potentially applied to the oil industries due to their high specificity and low operating temperatures. These are the reasons which make enzyme process more economical for oil extraction processes (Rosenthal *et al.,* 1996). Enzymes decompose the cell structure of plants. The cell wall of plants consists mainly of pectic substances, cellulase, hemicelluloses, lignin and protein, whereas lipid bodies are enveloped in a lipoprotein layer. Enzymes like cellulase, himicellulase and pectinase break down the cell, while proteases permeabilize the liposome membrane and facilitate oil release from the oil body (Rosenthal *et al.,* 1996; Fullbrook, 1983). Aqueous enzymatic oil extraction is one such alternatives-friendly process based on simultaneous isolation of oil and protein from oil seed by dispersing finely ground seed in water and separating the dispersion by centrifugation into oil, solid, and aqueous phases. Dobozi *et al.* (1988) reported that the treatment of mustard seeds with cellulolytic enzymes resulted in an increase (20–30%) in the yield of oil. Optimization of the enzymatic treatment during aqueous oil extraction with cellulases from sunflower seeds has been reported by Sineiroa *et al.* (1998). Latifa & Anwara (2011) reported oil and protein extraction from sesame seeds during an enzyme-assisted aqueous extraction process. Extraction of oil from watermelon seeds by aqueous enzymatic extraction method has been studied by Xiaonan *et al.* (2011). They obtained the optimum parameters form single-factor experiment and response surface methodology. Najafian *et al.* (2009) found that oil extraction from olive can be enhanced by enzyme hydrolysis and demonstrated that pre extraction enzyme digestion increases cellular degradation and significantly increases the oil recovery upon extraction. An aqueous enzymatic extraction of peanut oil and protein has been studied by Jiang *et al.* (2010). Hadj-Taieb *et al.* (2012) studied the effect of enzymatic formulation on Tunisian olive oil extraction yields. Also optimization of the aqueous enzymatic extraction of pine kernel oil by response surface methodology and extraction of olive oil using enzymatic formulations during malaxation has been reported by Yang *et al.* (2011). Liua *et al.* (2009) researched the extraction of pomegranate seed oil by using SC-CO_2. Response surface methodology was used to evaluate the effects of the process parameters. The extraction parameters were optimized with a central composite design experiment. The linear term of pressure, the linear term of CO_2 flow rate, the quadratic terms of pressure, temperature and CO_2 flow rate and the interactions between pressure and temperature, as well as CO_2 flow rate and temperature, had significant effects on the oil extraction yield. He *et al.* (2010) analyzed SC-CO_2 extraction of whole fruit oil from Gardenia jasminoides Ellis. This study revealed that the second-order polynomial model could be practiced to optimize extraction of gardenia fruit oil for maximizing the oil yield within the experimental ranges. The extraction of jatropha oil from the seeds was carried out with SC-CO_2 at different temperatures (308.15–328.15 K) and pressures (20–50 MPa) by Min *et al.* (2010). The Chrastil equation and a modified Chrastil equation were treated to correlate the solubility data. The values of average absolute relative deviation (AARD) were 10.1% and 3.47%, respectively, pointing out that the modified Chrastil equation was much greater than the Chrastil equation, basically due to the improvement of parameters. Li *et al.* (2011) investigated the oil extraction from red pepper seed by SC-CO_2. Three-level Box–Behnken factorial design (BBD) from RSM was practiced to optimize the principal extraction conditions comprising pressure, temperature and concentration of modifier (ethanol). Yin *et al.* (2005) applied ANN technology to simulate the supercritical fluid extraction process of vegetable oil. They established the kinetic model of artificial neural networks (ANN) supported on differential mass balance of packed bed. The results proved that the trained network was able to simulate the extraction rate of the Hippophae rhamnoides L. seed oils. Moreover, the network could produce a good estimation for the change of fluid concentration with the bed position and extraction time.

An empirical kinetic model was derived for the extraction of black cumin (Nigella sativa L.) seed oil with supercritical carbon dioxide as solvent by Fullana *et al.* (2000). An ANN model was considered to predict extraction yield. It incorporated a feed forward multilayer neural network appropriately trained with the back propagation algorithm. Inputs to the neural net were: pressure, temperature and time. The system squashed the yield of extraction as the only system response. The pseudohomogeneous model considered as suitable one for quantitatively outlining the supercritical fluid extraction of seeds packed in fixed beds. Excluding the variations in fluid flow rate and the solubility of Nigella sativa oil in the SC-CO_2, the model was achieved to be vigorous for the rest of parameters influencing the mass balance equation. Investigation concerned with optimization of supercritical carbon dioxide extraction of Passiflora seed oil has been carried out by Zahedi & Azarpour (2011). Response surface methodology (RSM) and artificial neural network (ANN) were used to evaluate the effects of the process parameters. Moreover, process optimization were carried out by using both methods to predict the best operating conditions, which resulted in the maximum extraction yield of the Passiflora seed oil. The maximum extraction yield of Passiflora seed oil was estimated by ANN to be 26.55% under the operational conditions of temperature (56.5°C), pressure (23.3 MPa), and the extraction time (3.72 h), whereas the optimum oil extraction yield was by 25.76% applying the operational circumstances of temperature (55.9 °C), pressure (25.8 MPa), and the extraction time (3.95 h) by RSM method. This paper focuses on the oil extraction utilizing enzyme. ANN and ANFIS were employed to model and optimize the extraction process. After introducing both methods and their applications, modeling and optimization of oil exteraction by enzyme were carried out along with comprehensive explanation of the procedure. Thereafter, the results of models were compared using mean-squared-error method (MSE) and the acquired values were reported in the tables and the figures.

Materials and Methods

Pistacia Khinjuk seeds were purchased from local market in Iran. The seeds were wrapped in plastic bags and stored at 4°C until usee. Seeds were ground and screened to select the fraction size. All the chemicals used were purchased from Merck (Darmstadt, Germany) or Sigma–Aldrich (Buchs, Switzerland) Chemical Companies. Cellulase preparation from Aspergillus niger was obtained from Sigma.

$$\%\ \text{Oil Recovery} = \frac{\text{Weight of oil extracted} \times 100}{\text{Total weight of oil estimated by soxhlet method}} \quad (1)$$

- Aqueous extraction of Pistacia Khinjuk

Pistacia Khinjuk was dispersed in distilled water to make slurry at a ratio of 1:6 w/v using a flask. Slurry pH was adjusted to the desired value with 0.1 N NaOH or 0.1 N HCl, and was stirred on a magnetic stirrer at 250 rpm for 30 min. The enzymes were added at different concentrations and the samples were incubated at various temperatures and times with the constant mixing speed. Afterwards, the samples were incubated at constant temperatures. A shaker-incubator (DK-S1060, DAIKI SCIENCE CO.) was used for temperature-controlled shaking of the sample solutions, followed by centrifugation (10000g, 30°C) for 20 min (MIKRO 200, HETTICH) yielding three distinct phases (i) an oil phase, (ii) creamy phase and (iii) aqueous phase. The upper oil layer was separated and weighed. Oil recovery was expressed relative to that obtained by Soxhlet extraction with hexane.

The total amount of extracted oil was determined with Soxhlet apparatus following the standard AOAC standard procedure (Vining, 1998). All experiments were repeated three times to render mistakes during experiments. The ranges of input and output parameters are shown in Table 1.

- *Artificial Neural Network (ANN)*

In order to find a relationship between the input and output data obtained from accelerated experimentations, a more sophisticated method than traditional method is necessary. ANN is a particularly efficient algorithm to approach any function with limited number of discontinuities by learning the relationships between the input and the output vectors (Vallés, 2006; Hagan, 1996). ANN techniques are peculiarly helpful for modeling highly nonlinear and complicated systems. ANNs are biologically inspired based on various characteristics of the brain functionality. Artificial neurons are simple computational devices, which are highly interconnected. An ANN determines an empirical relationship between the inputs and the outputs of a given system in which its inputs and outputs are the independent variables and dependent variables, respectively. A network is consisted of units or nodes, which represents the neuron body. The units are interconnected by links that act like axons and dendrites of their biological counterparts. A typical interconnected neural network is illustrated in Figure 1 (Zahedi *et al.,* 2005; Zahedi *et al.,* 2009; Zahedi *et al.,* 2010).

Table 1. The ranges of input and output parameters

Variable	Range of the parameter value
Input layer	
Enzymes concentration	0.2-0.6
pH	5-9
Ultrasonic time (h)	0-5
Percentage of oil extraction	0-1

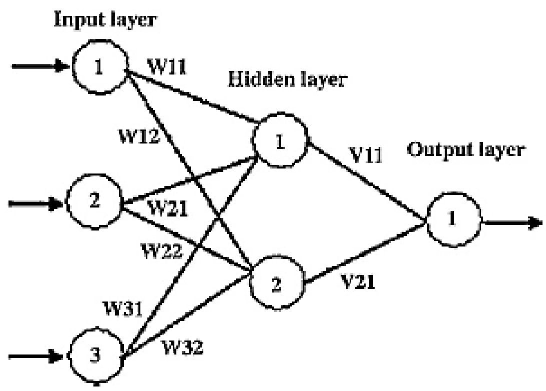


Fig. 1. A typical interconnected neural network

In Figure 1 an input layer, a central or hidden layer and an output layer can be seen. In a network, each connecting line has an associated weight. Supplying fast answers to a problem and generalizing answers providing acceptable results for unknown patterns are two important potentialities of the neural network (NN). As an outcome, they should learn about the problem under study and this learning is conventionally cited as training process. To devote random value for the weight of NN is the initial step of training. Therefore, NNs are provided with a set of patterns linking up with a problem domain to adapt the amount of their weights. There are various learning algorithms to train neural networks. One of the notable topologies of neural networks for learning is the multi-layer perceptron (MLP), which is applied to the categorization and the approximation problems (Zahedi *et al.,* 2005; Zahedi *et al.,* 2009; Zahedi *et al.,* 2010). An MLP is an NN with three layers, an input layer, a hidden layer, and an output layer. The input layer outlines the entering pattern and the output layer is the product of the network. Each layer is consisted of a series of nodes associated with weights. During the learning sequence, the MLP is acquainted with an input pattern on the input nodes and a target pattern on the output layer. The weights are then renovated so as to yield the favorable output for the network. Each node includes an activation function, which is a function identifying whether the neuron should fire relying upon its inputs. After training (when the network is applied to utilize), the values of the weights and the activation functions determine which nodes fire. These activation functions show up many different forms, the classics being threshold, sigmoid Gaussian, etc. (Lang, 2006). For more details of the various activation functions one can study Bulsari (Bulsari, 1995).

- Neural network applied to oil extraction yield predicting

Data evaluation before applying them for modeling by ANN is a crucial action in order to exclude insufficient data (Zahedi *et al.,* 2009). To make sure that the selected data for modeling present normal operating ranges, the unsatisfactory ones were excluded from the data source. The data, which were not in the normal course of the process, were removed. The back-propagation learning with one hidden layer network has been used in this work. Inputs and outputs are normalized between the values −1 and 1. Logistic Sigmoid and purelin transfer functions have been used in constructing ANNs. ANN has been trained with 70% of the data set and 30% of the data have been applied for testing the predictions of NN.

- *Adaptive Neuro Fuzzy Inference System (ANFIS)*

A structure of ANFIS is presented in Figure 2, in which a circle indicates a fixed node, whereas the square shows an adaptive node. For simplicity, it was assumed that the FIS has two inputs x and y and one output z. The ANFIS used in this study performs a first order Sugeno fuzzy model. A typical rule set with two fuzzy if–then rules for this model can be defined as follows:

Rule 1: If x is A_1 and y is B_1 then $z_1 = p_1 x + q_1 y + r_1$

Rule 2: If x is A_2 and y is B_2 then $z_2 = p_2 x + q_2 y + r_2$

Where A_i and B_i are the fuzzy sets in the antecedent, and p_i, q_i, and r_i are the design parameters determined during the training process.

Fuzzify inputs: Resolve all fuzzy statements in the antecedent to a degree of membership between 0 and 1. When there is only one part of the antecedent, this is the degree of support for the rule.

Apply fuzzy operator to multiple part antecedents: If there are multiple parts to the antecedent, use fuzzy logic operators and solve the antecedent to a single number between 0 and 1. This is the degree of support for the rule.

Apply implication method: Use the degree of support for the entire rule so as to shape the output fuzzy set. The consequence of a fuzzy rule assigns an entire fuzzy set to the output. This fuzzy set is indicated by a membership function that is selected to indicate the qualities of the consequent. If the antecedent is partially true, (i.e., Assigns a value less than 1), then the output fuzzy set is truncated based on the implication method.

As ANFIS structure is depicted in Figure 2, each node within the same layer performs functions of the same type. If a node parameter set is not empty, then its node function depends on the parameter values; a square is used to represent this type of adaptive node.

On the other hand, when a node has an empty parameter set, its function is fixed; a circle is used to specify this type of fixed node. The structure includes five layers:

Layer 1: Each node 'i' in this layer is a square node with a node function.

$$O_i^1 = \mu_{A_i} \tag{2}$$

Where x is the input to the node 'i', A_i is the linguistic label, and O_i^1 is the membership function of A_i. Parameters in this layer are expressed as premise parameters.

Layer 2: Circle nodes in this layer multiply the incoming signals and send the product out. This indicates the firing strength of a rule.

$$\omega_i = \mu_{A_i}(x) \times \mu_{B_i}(y), \quad \mathrm{i} = 1, 2 \tag{3}$$

Layer 3: Every node in this layer, labeled in Figure 2 with N, calculates the average ratio of 'ith' rule's firing strength.

$$\overline{\omega}_i = \frac{\omega_i}{\omega_1 + \omega_2} \qquad \mathrm{i} = 1, 2 \tag{4}$$

Layer 4: Every node 'i' in this layer is a square node with a node function.

$$O_i^4 = \overline{\omega}_i f_i = \overline{\omega}_i(p_i x + q_i y + r_i) \tag{5}$$

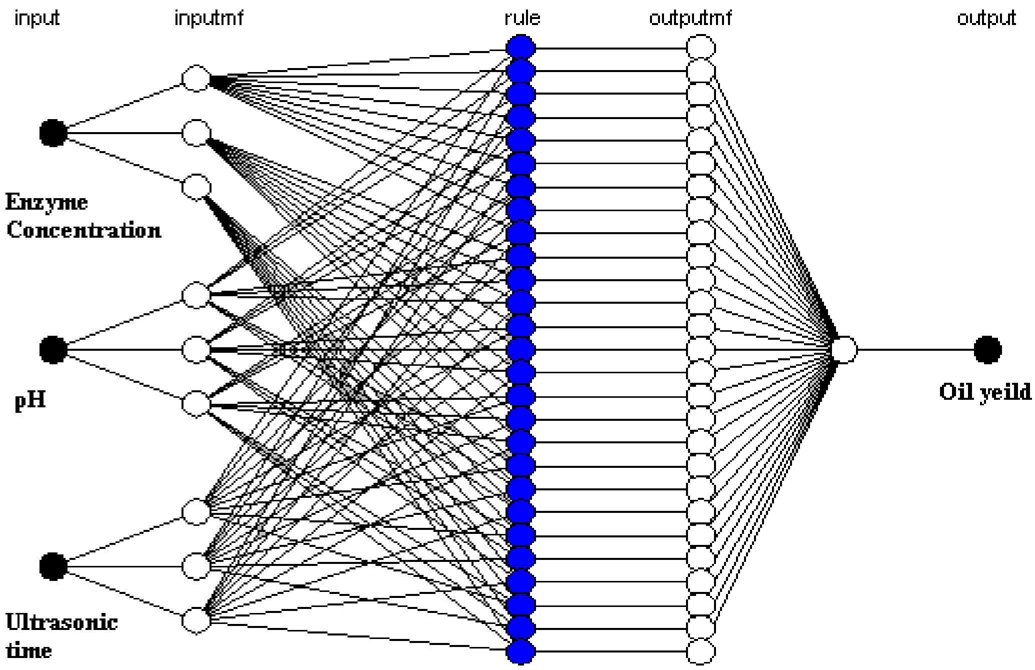


Fig. 2. Adaptive Neuro-fuzzy inference system structure

Where $\overline{\omega_i}$ is the output of layer 3 and parameters p_i, q_i and r_i are referred to as consequent parameters.

Layer 5: The node in this layer computes the overall output as the summation of all incoming signals:

$$O_i^5 = \sum_i \overline{\omega}_i f_i = \frac{\sum_i \omega_i f_i}{\sum_i \omega_i} \tag{6}$$

The oil extraction yield is estimated utilizing the above mentioned ANFIS based methodology. As the Gaussian membership functions are the most common, they are considered in this paper. Different values of the fuzzy exponent were analyzed and the value of 3 was found to be a system capable of a high performance. Furthermore, several numbers of membership functions were assumed to find optimum number. Based on many experiments, the number of membership functions (MFs) was selected as 3 for every input model. The training time increases with an increase in the number of MFs. Figure 2 shows the architecture of one proposed ANFIS model with 3 inputs, 1 output and 27 fuzzy rules. The initial value of the step size for training the ANFIS was fixed at 0.01. Based on the above settings, the training of the ANFIS model was conducted in MATLAB software version 2008a environment.

Results and Discussion

- The result of ANFIS model

The oil extraction yield was calculated using hybrid Neuro-fuzzy model. Defining a fuzzy membership function and the corresponding value is the most important step in the model. Gaussian and bell membership functions are the most commonly used methods for specifying the fuzzy set due to their smoothness and concise notation. Both membership functions have the advantages of being smooth and non-zero at each point. Since the bell membership function has a more parameter than Gaussian membership function, it can approach to non-fuzzy set, if the free parameter is tuned (Lotfi Zadeh, 1995). Hence the Gaussian membership function has been investigated (Figure 3).

The hybrid algorithm has been applied to the membership function of each input. The structure of desired rules and results of these rules is depicted in Figures 4 and 5, respectively.

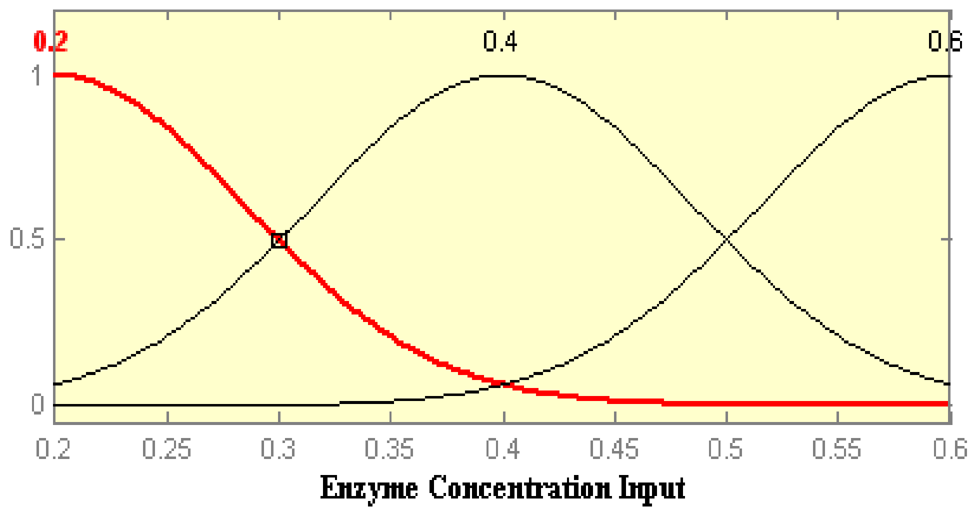


Fig. 3. Gaussian membership functions for input 1

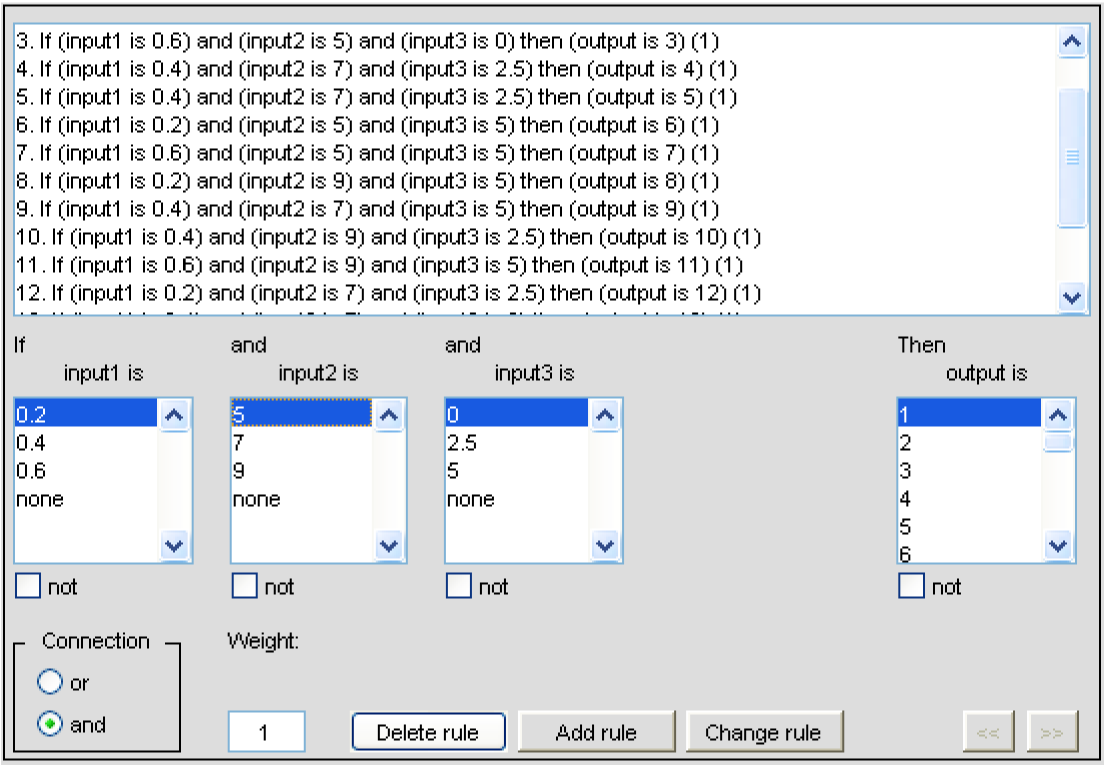


Fig. 4. Rule structure

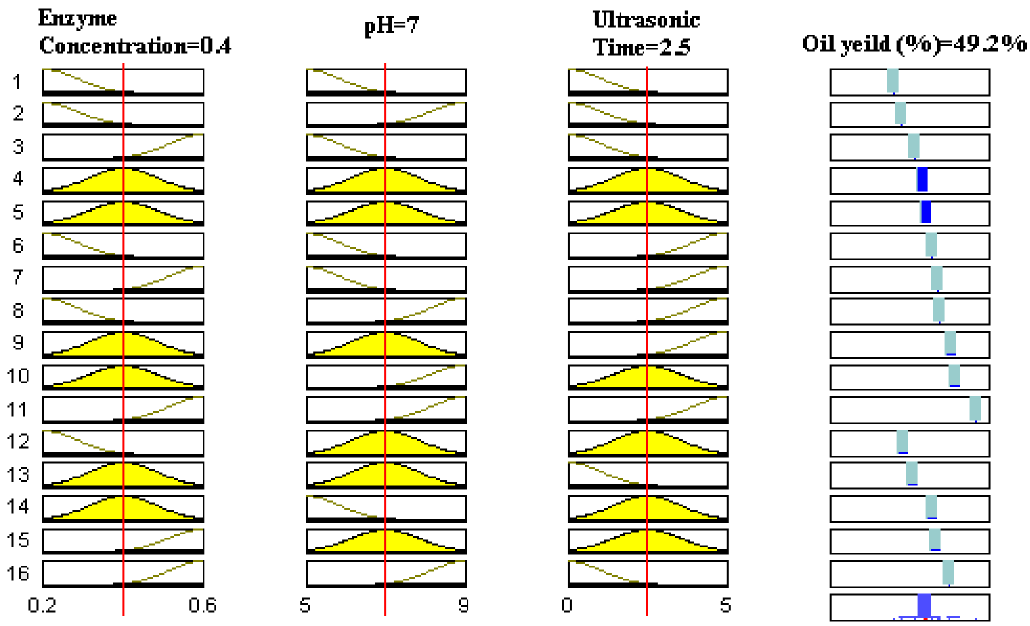


Fig. 5. ANFIS rule structure

A typical surface graph that shows the relationship between the effects of inputs on the output is shown in Figure 6.

The system performance indices correlation coefficient (R^2), 0.995 are shown in Figure 7.

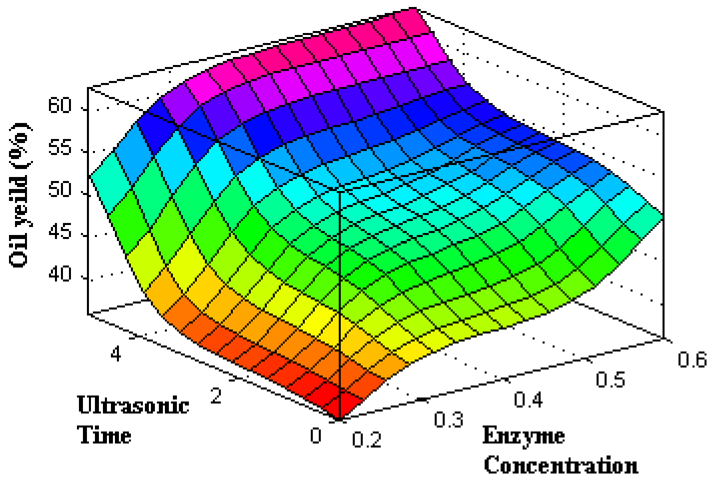


Fig. 6. Typical surface graph

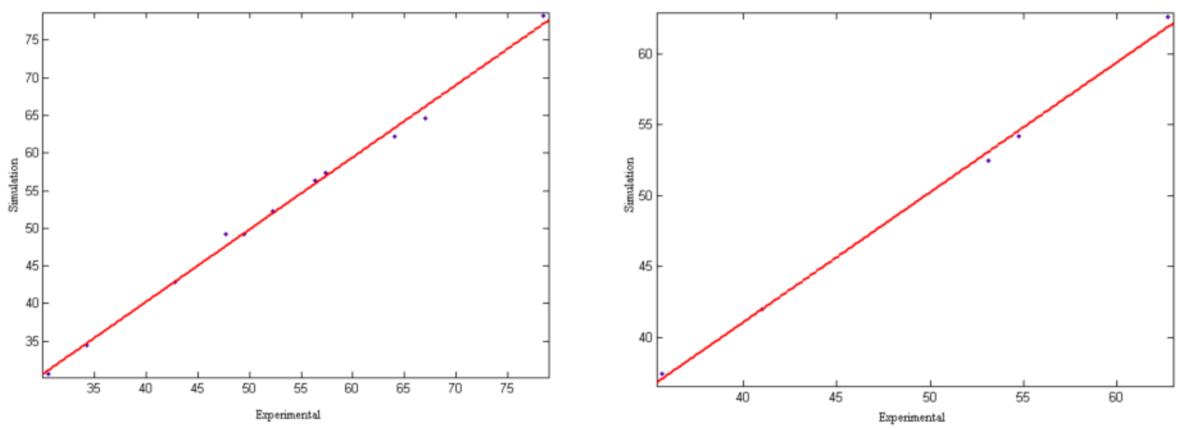
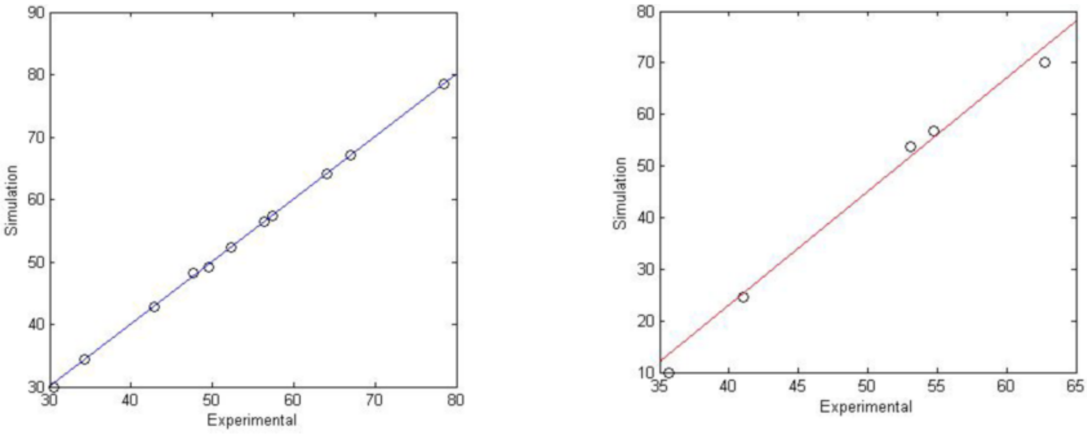


Fig. 7. Cross-correlation of experimental data (the oil extraction yield) vs. predicted values by ANFIS model for (a) train data, (b) test data

- The result of ANN model

The LM training algorithm was employed for modeling the oil extraction yield of Pistacia Khinjuk. There is no any general and precise method to achieve the optimum number of hidden layers of the neurons and it is obtained by trial and error. The optimum number of hidden layer neurons was determined to be 10 for this network. Two scatter plots of measured experimental data against the predicted values by ANN model were illustrated in Figure 8. Figure 8a gives information on the oil extraction yield by comparing the experimental data against the ANN model predicted values for training data. Figure 8b implies the experimental data versus the simulated ones derived by ANN model for testing data, which have not been applied for the training of the ANN (30% remaining data), for the extraction yield. The figures show that the data obtained from the model are in a very good agreement with the laboratory results. The predictions that match measured values should fall on the diagonal line. Almost all data fall close to this line, which confirms the accuracy of the ANN model.



(a) (b)

Fig. 8. Scatter plot of experimental data (the oil extraction yield) vs. predicted values by ANN model for (a) train data, (b) test data

Some statistical methods were used for the comparison. The criterion for the comparison in this work was mean-squared-error method (MSE) between the net output and the training data. MSE is defined as:

$$\mathrm{MSE} = \frac{\Sigma(\mathrm{X_{exp}} - \mathrm{X_{sim}})}{\mathrm{X_{exp}}} \quad (7)$$

where X_{exp} is the target value, X_{sim} is the output value, and n is the number of the experimental data. In addition to compare the results of the used methods by MSE, relative error has been used to evaluate the results and analogy as well. The percent relative error is defined as:

$$\text{Percente Ralative Error} = \frac{\text{Value} - \text{Approximate}}{\text{Value}} \times 100 \quad (8)$$

where "value" is the experimental value used to construct the model and "approximate" is the output of the neural networks at the same conditions. Regardless of the model, this error can have either positive or negative values. However, the better result is the convergence of relative error parameter to zero. Errors of measurements using this criterion has been shown in

Tables 2 and 3 (derived from ANN and ANFIS models for training and testing data, respectively). It is obvious that ANN has a superior overlap with the laboratory experimental data comparing to ANFIS. MSE values for ANN model and ANFIS method were calculated as 0.0005 and 0.0925, respectively. Therefore, the results derived from ANN model were significant and the error was incredibly low. It can be observed that the optimum extraction process parameters within the experimental ranges are Enzyme concentration of 0.27, pH=6, and Ultrasonic time of 4.23 h. Under these conditions, the oil extraction yield was 56.52% while the yield obtained from ANFIS model was 55.8%. As ANN is more accurate than ANFIS, it can be concluded that the optimum values from ANN model optimization are more reliable.

Conclusion

The comparison between ANN and ANFIS models has been implemented by MSE and relative error methods. For instance, MSE value for ANN model has been enumerated to be 0.0005, which is a great result in respect to the MSE value for ANFIS method which is 0.0925. From the results we can conclude that the ANN model by using MLP neural network architectures was the best for the estimation of the values of the targets in comparison with ANFIS model.

Table 2. Comparison of the ANN prediction with ANFIS for training data

Enzyme concentration	pH	Ultrasonic time (h)	Oil yield (exp.)	Predicted value (ANN)	Predicted value (ANFIS)	Relative (%)error (ANN)	Relative error (%) (ANFIS)
0.2	5	0	30.55	30.00	30.7	0.018003	-0.004909
0.2	9	0	34.31	34.39	34.5	-0.002331	-0.005537
0.6	5	0	42.81	42.89	42.9	-0.001868	-0.002102
0.4	7	2.5	47.71	48.21	49.2	-0.010479	-0.031230
0.4	7	2.5	49.51	49.22	49.2	0.005857	0.006261
0.2	5	5	52.29	52.37	52.3	-0.001530	-0.000191
0.6	5	5	56.37	56.46	56.4	-0.001596	-0.000532
0.2	9	5	57.35	57.44	57.3	-0.001569	0.000871
0.4	7	5	64.05	64.14	62.2	-0.001405	0.028883
0.4	9	2.5	66.99	67.08	64.6	-0.001343	0.035677
0.6	9	5	78.43	78.52	78.2	-0.001147	0.002932

Table 3. Comparison of the ANN with ANFIS for testing data

Enzyme concentration	pH	Ultrasonic time (h)	Oil yield (exp.)	Predicted value (ANN)	Predicted value (ANFIS)	Relative (%)error (ANN)	Relative error (%) (ANFIS)
0.2	7	2.5	35.62	10.00	37.4	0.719260	-0.049971
0.4	7	0	41.01	24.56	42.0	0.401121	-0.024140
0.4	5	2.5	53.10	53.83	52.5	-0.013747	0.011299
0.6	7	2.5	54.74	56.86	54.2	-0.038728	0.009864
0.6	9	0	62.74	70.00	62.6	-0.115715	0.002231

By applying ANN model a good conformity with the experimental data was earned. An important feature of the model is that it does not call for any theoretical knowledge or human experience during the training process. Therefore the former knowledge has not been utilized and the model has been only trained based on the experimental data. All unknown relationships have been embodied with NN instead of the traditional procedures.

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