

ARTIFICIAL NEURAL NETWORKS MODELING OF THE VISCOELASTIC PROPERTIES OF VAPOR-GROWN CARBON NANOFIBER/VINYL ESTER NANOCOMPOSITES

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Keywords: *Artificial neural networks, materials informatics, vapor-grown carbon nanofiber, vinyl ester, cross validation.*

1 Introduction

An artificial neural network (ANN) is a modeling approach to non-linear function approximation based on “artificial intelligence”. This technique is very useful in modeling phenomena that are not easily modeled analytically [1]. ANNs consist of a host of simple processors (neurons) that are interconnected in an organized architecture and are associated with a learning algorithm that resembles a biological process [1]. There are numeric values associated with the interconnections of the simple processors that are adjusted over time to emulate learning. The weights associated with the interconnections (a measure of the strength of the connections) encode knowledge about the problem domain. The architectures (neurons and their interconnections) provide a computational structure for simulating a biological neural network [2].

Learning in an ANN can occur in either a supervised or an unsupervised fashion [1]. A supervised approach uses a learning algorithm that creates an input/output mapping based on a labeled training set; thus, creating a mapping between an n -dimensional input space and m -dimensional output space. In this case, the network will learn a functional approximation from the input/output pairings and will have the ability to recognize or classify a new input vector into a correct output vector. On the other hand, an unsupervised learning architecture presents the network with only a set of unlabeled input vectors from which it must learn. In other words, the unsupervised ANN is expected to discover new knowledge as it characterizes the input vectors and produces outputs corresponding to learned patterns.

The use of the ANN techniques in the context of materials science and engineering is considered an important extension of materials informatics [3-6]. This interdisciplinary study integrates computer science, information science, and other domain areas to provide new understanding and to facilitate

knowledge discovery. Materials informatics is a tool for material scientists to interpret their acquired experimental data through the use of ANNs and machine learning approaches, integrated with new visualization schemes, more human-like interactions with the data, and guided by domain experts. It can also accelerate the research process and guide the development of new materials with desired engineering properties. Materials informatics is being fueled by the new and dynamic growth in information technology and is driving the interest in ANNs, data mining, machine learning, information retrieval, and other knowledge representation or discovery schemes in the engineering disciplines [7]. There are several recent published applications utilizing materials informatics and ANNs. Yassar, et al. [8] developed a novel computational model based on dislocation structures to predict the flow stress response of 6022 aluminum alloy. An ANN model was used to back-calculate the in-situ non-linear material parameters and flow stress for different dislocation microstructures [8]. Guessasma and Coddet [9] characterized the microstructural features of alumina-13 wt.% titania coating obtained under various atmospheric plasma spray (APS) conditions by implementing ANNs, which were detailed in the case of APS process parameters. These parameters were related to alumina, titania, porosity and unmolten particle contents. Yoshitake, et al. [10] used an ANN to model the changes in lattice phases as a function of their chemical composition and temperature. Reasonable phase predictions were made for several alloys, which agreed with X-ray measurements. The variation of the lattice constant with the concentration of individual alloying elements and temperature could be embodied into other computer programs, which deal with the partitioning of solutes between the different phases. Hu, et al. [11] used materials informatics to resolve the problem of materials science image data sharing. They presented an ontology-based approach that can

be used to develop annotation for non-structured materials science data with the aid of semantic web technologies. Sabin, et al. [12] evaluated an alternative statistical Gaussian process model, which infers a probability distribution over all of the training data and then interpolates to make predictions of microstructure evolution arising from static recrystallization in a non-uniform strain field. Strain, temperature, and annealing time were the inputs of the model and the mean logarithm of grain size was its output.

A class of advanced engineering materials, i.e., nano-enhanced polymer composites and polymer nanocomposites [13], has progressively emerged as candidates for light-weight high-performance automotive composite structural parts. In these applications, improved specific properties and energy absorption characteristics are sought and polymer composites meet these requirements [14]. The scope of this study is to apply the ANN technique as a knowledge discovery tool on a commercially viable thermosetting polymer nanocomposite material system, i.e., vapor-grown carbon nanofiber (VGCNF)/vinyl ester (VE) nanocomposites. VGCNFs are commercially available nanoreinforcements with first-rated mechanical properties [19]. VEs are thermosetting resins suitable for automotive structural composites due to their superior properties in comparison with unsaturated polyesters [16-18, 20, 21]. Incorporating VGCNFs into VEs provides improved mechanical properties relative to the neat VE matrix. Nouranian, et al. [15-17] and Torres, et al. [18] have developed a relatively large mechanical property dataset for this material system that are suitable for the ANN analysis. This study seeks to use this dataset to demonstrate the usefulness of knowledge discovery and ANN techniques for nanocomposite material property characterization. In this case, ANNs and knowledge discovery techniques help model and predict the viscoelastic responses (storage and loss moduli) and tan delta (ratio of loss to storage modulus) of VGCNF/VE nanocomposites, thereby aiding the nanocomposite design and characterization.

An ANN model was used to explore the VGCNF/VE dataset discussed above [15]. The dataset consisted of 240 data points, each corresponding to the combinations of the levels associated with five input design factors and three output responses, i.e., a total of eight “dimensions.” The dimensions are the combinations of both inputs and outputs of the developed ANN model. For the

VGCNF/VE dataset, the dimensions are five formulation and processing factors, i.e., VGCNF type, use or absence of dispersing agent, mixing method, VGCNF weight fraction, and temperature, and three responses, i.e., storage modulus, loss modulus, and tan delta. The last three dimensions correspond to measured material properties that were also successfully predicted using the ANN resubstitution and ANN 3-folds cross validation techniques.

2 Materials and Methods

A brief summary of the statistical experimental design utilized by Nouranian, et al. [17] in generating the VGCNF/VE dataset is given here. A more detailed discussion on the specific materials formulations and specimen preparation steps as well as testing procedures can be found in [15-17].

2.1 Statistical design of experiments

Five input design factors, i.e., VGCNF type (designated as A), use or absence of a dispersing agent (B), mixing method (C), VGCNF weight fraction (D), and temperature (E) (Table 1) were incorporated in a general mixed-level full factorial design [22] with storage modulus, loss modulus, and tan delta as the viscoelastic responses. The resulting $2 \times 2 \times 3 \times 5 \times 4 = 240$ “treatment combinations” (different combinations of the factor levels) were randomized before experimentation to eliminate bias in preparing the specimens. Dynamic mechanical analysis (DMA) was used to measure the viscoelastic responses. Each treatment combination resulted in three specimens for testing and the average value of the three test results was calculated for each response [16, 17]. Note that the storage and loss modulus are indicative of the nanocomposite’s stiffness and energy dissipation capability, respectively. The average storage and loss moduli for each of the 240 treatment combinations are given in [15].

3 Theory/Calculation

As mentioned in Section 2, this study incorporates five input design factors, i.e. factors A, B, C, D, and E and three output responses, i.e., storage modulus, loss modulus, and tan delta in the ANN. Therefore, the dataset represents an eight-dimensional (8-D) case for analysis. Since factors A, B, and C are considered *qualitative* factors, they are represented by a numeric code for analysis purposes. For two-level factors A and B, 0 and 1 are the coded values

for the first and second levels, respectively. For the three-level factor C, -1, 0, and 1 are the coded values for the first, second, and third levels, respectively. Factors D and E are considered *quantitative*, so their values can be used directly in the analysis (Table 1). However, all the quantitative values were normalized using standardized scores. ANN resubstitution and ANN 3-folds cross validation techniques were used with the 240 treatment combination dataset to predict and model

the three output responses of the nanocomposite data. This is particularly helpful in the case that the inputs of the VGCNF/VE system are known *a priori*, but not the outputs. Thus, using the developed ANN model, there is no need to conduct the experiments needed to analytically estimate these responses. Before applying these techniques, a brief explanation of the ANN resubstitution and cross validation techniques are introduced.

Table 1. The experimental design factors and their levels [15, 17].

Factor designation	Factors	Level				
		1	2	3	4	5
A	VGCNF type	Pristine	Oxidized	-	-	-
B	Use of dispersing agent	Yes	No	-	-	-
C	Mixing method	US ^a	HS ^b	HS/US	-	-
D	VGCNF weight fraction (phr ^c)	0.00	0.25	0.50	0.75	1.00
E	Temperature (°C)	30°C	60°C	90°C	120°C	-

^a Ultrasonication

^b High-shear mixing

^c Parts per hundred parts of resin

3.1 ANN resubstitution method

In the ANN resubstitution method [23], the whole dataset is used to train the ANN and the same dataset is used for testing (validation). This ensures that the designed ANN model generalizes well for the unseen data samples, when a combination of inputs is applied to the ANN and the outputs (responses) are not explicitly known. This method is computationally efficient. Good generalization is achieved when the mean squared error (MSE) between the actual responses of the ANN model and the desired (targeted) responses is minimal [23].

The MSE is defined as:

$$MSE = \frac{1}{N} \sum_{i=1}^N (e_i)^2 = \frac{1}{N} \sum_{i=1}^N (t_i - a_i)^2 \quad (1)$$

where N : is the total number of samples,

t_i : is the targeted response, and

a_i : is the actual ANN response

Although several network architectures and training algorithms are available, the feedforward neural network (FFNN) trained by the back-propagation (BP) algorithm is the most commonly used one and we have utilized it in this study. A FFNN is a layered network of artificially processing units, called neurons, in which connections between neurons are associated with weights that represent

the strength of the connections [1]. It includes an input layer, with one neuron corresponding to each of the inputs used in the model, and an output layer, with a single neuron corresponding to each output variable (response). The network also includes one or more “hidden” layer(s) of neurons. Because each neuron in the hidden layer is associated with an activation function (sigmoidal function in this study), the hidden layer(s) allow(s) a non-linear mapping from the values of the input variables to the value of the output variable [1].

A FFNN network is trained using a dataset of related input-output examples to estimate a non-linear relationship between the input variables and the output variable(s). Upon presenting the training samples to the network, the weighted connections between neurons are adjusted by BP to decrease the MSE between the network’s output and the targeted output. The process is repeated until the MSE has been reduced as much as possible [24].

3.2 Cross validation technique

Cross validation (CV) is a technique that can be used to better train the neural network with the available samples in the dataset. First, the available dataset is randomly partitioned into a training set and a test set. The training samples are further partitioned into two disjoint subsets: 1) the estimation subset, which is used to select the ANN model and determine the interconnection weights, and 2) the validation

subset, which is used to test or validate the developed ANN model [25].

The motivation to use the CV technique is to validate the model on a dataset different from the one used for parameter estimation. In this way, the training samples can be used to assess the performance of various candidate ANN models and thus the “best” one can be chosen [25]. There are four different CV methods and the following is a brief explanation of each of these methods [24].

1) **Holdout:** If a random number $r \in [0, 1]$, then $(1-r)N$ samples are allotted to the estimation subset, and the remaining rN samples for validation, where N is the total number of samples. This method is computationally expensive and the final ANN model is the one yielding the minimum validation error.

When the complexity of the target function (input-output mapping) is small compared to the sample size N , the validation performance is relatively insensitive to the choice of r , whereas when the target function becomes more complex relative to the sample size N , the choice of r has a more pronounced effect on cross-validation performance. However, a single fixed value of r (e.g., 0.2) works nearly optimally for a wide range of target-function complexity.

2) **Early-stopping method of training:** With good generalization as the goal, the training can be stopped earlier before the learning error becomes too low. The best point to stop training can be determined by the periodic “estimation-followed-by-validation” process as shown in Figure 1. After some periods of training, say five epochs, the ANN weights are fixed and the validation error is then measured. When the validation phase is completed, the training is resumed for another epoch(s). Finally, when the validation error starts to increase, it is the point to terminate the training process and finalize the weights, even if the error for the training samples continues to decrease.

3) **Multifold:** The disadvantage of holdout method is that not all samples are used for validation. Instead, in multifold validation, the N samples are divided into K subsets. Each time, one subset is used for validation and the remaining $K-1$ subsets for training. The process is continued until each subset is used for validation once. In this study, 3-folds cross validation was implemented and the performance was assessed by averaging the

validation error over all the trials. In Figure 2, an illustration of a 4-fold cross validation is shown.

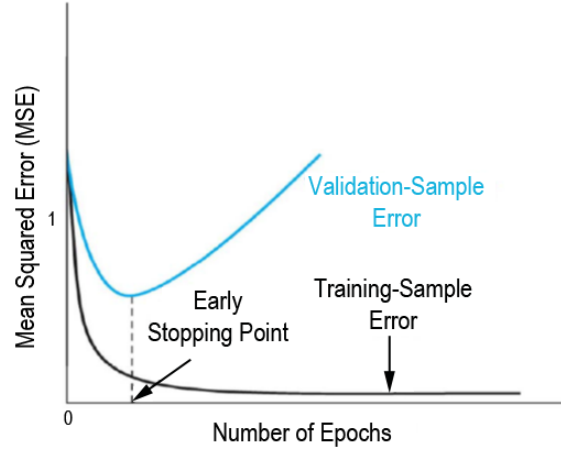


Figure 1. Illustration of the early-stopping rule based on cross validation.

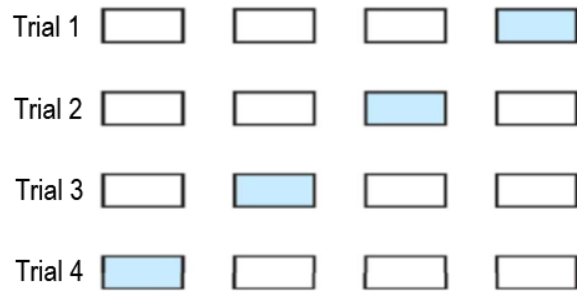


Figure 2. Illustration of the multifold method of cross validation. For a given trial, the subset of shaded data is used to validate the model and the remaining data is used to train the model.

4) **Leave-one-out:** When the available number of samples, N , is severely limited, an extreme form of multifold validation known as *leave-one-out* validation can be used. In each trial, $N-1$ samples are used for training and the one left out can be used for testing. The process is repeated N times until each sample is used for validation exactly once.

4 Results and discussion

Following the standard practice of the ANN analysis, the inputs were normalized using standardized scores, as their original value ranges were completely different from each other. This allowed the sigmoid functions to perform better. At the same time, the outputs were de-normalized. Because back-propagation is a gradient descent algorithm that can converge to a local optimum, the ANN model was trained six times with different initial weights (set randomly), and the best results are reported here.

The overall ANN architecture is shown in Figure 3. This structure was used when both the resubstitution and the 3-folds CV techniques were implemented.

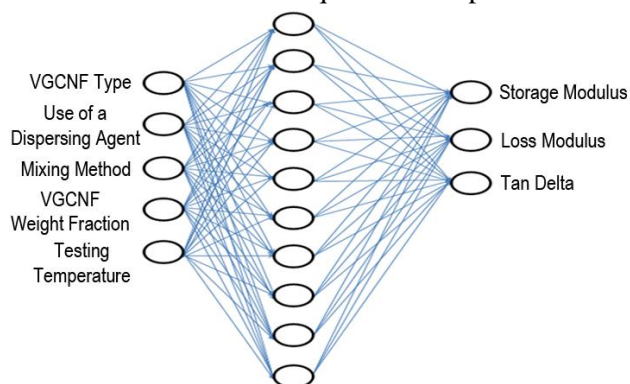


Figure 3. The architecture of ANN used in this study. Five neurons were used in the input layer; each for one input and three neurons were used in the output layer; each for one response. There is one hidden layer with ten neurons.

There are three layers utilized in this ANN architecture (Figure 3). The input layer consists of five neurons and each neuron carries one of the inputs used in this study (VGCNF type, use of a dispersing agent, mixing method, VGCNF weight fraction, and testing temperature). There is one hidden layer consisting of ten neurons. The hidden layer connects the input layer with the output layer via activation functions from *input-hidden* and from *hidden-output*. The output layer consists of three neurons, each for one of the responses used in this study (storage modulus, loss moduli, and tan delta). First, the ANN was trained using the resubstitution method, where all the 240 VGCNF/VE samples were used for training and testing. The ANN implementation details are illustrated in Table 2.

Table 2. Implementation details of the BPANN applied to the VGCNF/VE dataset using the resubstitution method.

Number of input neurons	5
Number of output neurons	3
Number of hidden layers	1
Number of neurons in the hidden layer	10
Mean Square Error (MSE)	0.0015
Learning rate	0.001
Input-hidden activation function	Sigmoidal
Hidden-output activation function	Sigmoidal
Number of epochs	23

Each input in the VGCNF/VE dataset (VGCNF type, use of a dispersing agent, mixing method, VGCNF weight fraction and testing temperature) is

associated with one input neuron in the input layer and each response (storage modulus, loss modulus, and tan delta) is associated with one output neuron in the output layer. Thus, the ANN architecture has five input neurons and three output neurons. The performance curve of the ANN implementation using the resubstitution method is shown in Figure 4.

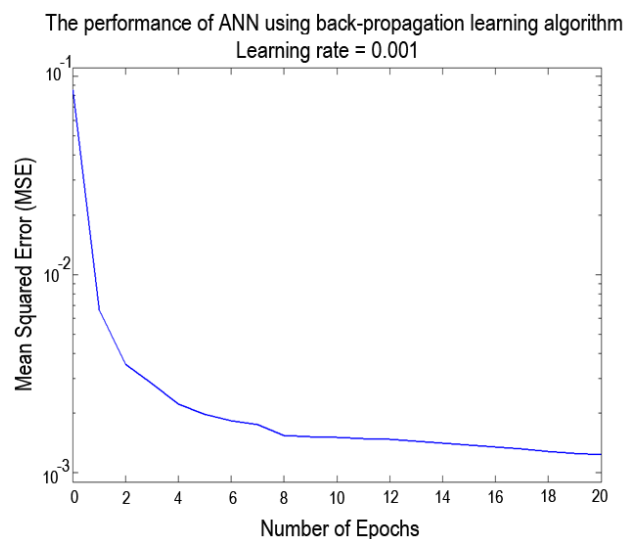


Figure 4. The performance curve of back-propagation ANN (BPANN) using the resubstitution method. The MSE gradually decreases with the increasing number of epochs and the best performance (lowest MSE) is achieved after running 23 epochs.

After the resubstitution method, the 3-folds CV technique was applied on the VGCNF/VE dataset. Since the total number of samples was 240 and three different trials were implemented, the size of the training and test sets in each trial were 160 and 80 samples, respectively. Each trial had different training and test samples than those of the other trials. This led to a more efficient training and testing of the ANN model and, therefore, the best performance and network structure was obtained. Similar to the ANN implementation using the resubstitution method, in 3-folds cross validation implementation, five neurons were used in the input layer (each VGCNF/VE input with one neuron) and three neurons in the output layer (each VGCNF/VE response with one neuron). The learning rate was 0.001 and the activation function implemented between the input-hidden and hidden-output layer was the sigmoidal function. There was one hidden layer with ten neurons.

The performance curve of the ANN implementation using the first fold of the 3-folds cross validation technique is shown in Figure 5.

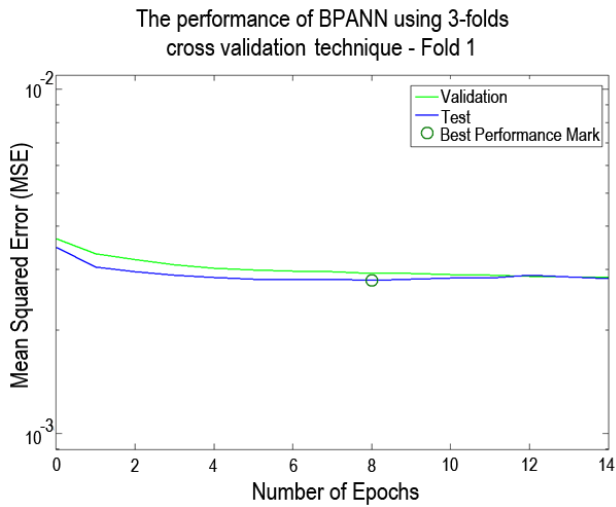


Figure 5. The performance curve using the data samples of the first fold when 3-folds cross validation technique was applied. The performance of the training set is slightly better than that of the test set and the best performance was achieved at epoch 8 when the MSE reached about 0.0028 mark.

As evident in Figure 5, the performance of both test (validation) and training sets were very close, even though the training set behaved better up to epoch 10. After that, the MSE of the training set began to increase and was equal to the MSE of the test set at epochs 12-14. The best performance of the ANN model using the first fold was achieved at epoch 8, when the MSE was minimal at about 0.0028.

The performance curve of the ANN implementation using the second fold of the 3-folds cross validation technique is shown in Figure 6.

The performance curves were almost steady for both training and test sets (Figure 6). However, the training set behaved better than the test set compared to the training and test sets in the first fold. The best performance of the ANN model using the second fold was achieved at epoch 17, when the MSE was minimal at about 0.0029. After that, the MSE tended to slightly increase and remained constant up to epoch 23.

The performance curve of the ANN implementation using the third fold of the 3-folds cross validation technique is shown in Figure 7. In this figure, the total number of epochs needed for the ANN model to converge was 25 epochs and the MSE was minimal at epoch 21. The performance of both the training and test sets was nearly the same at the beginning, but later it was slightly better for the training set than for the test set. However, both curves remained almost steady after running seven epochs of the analysis.

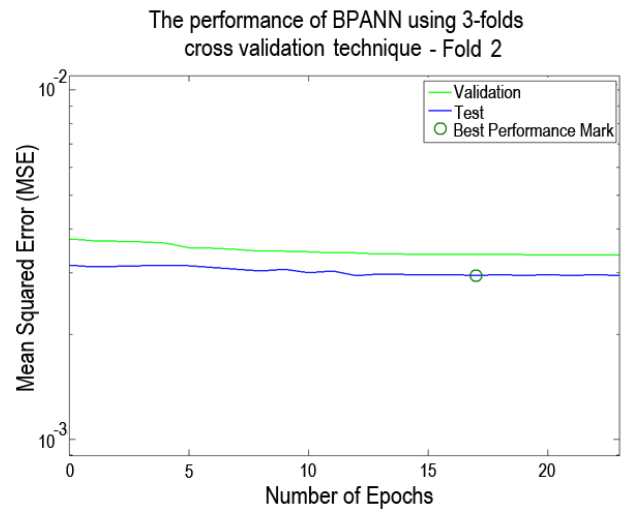


Figure 6. The performance curve using the data samples of the second fold when 3-folds cross validation technique was applied. The performance of the training set is slightly better than that of the test set and the best performance was achieved at epoch 17 when the MSE reached about 0.0029 mark.

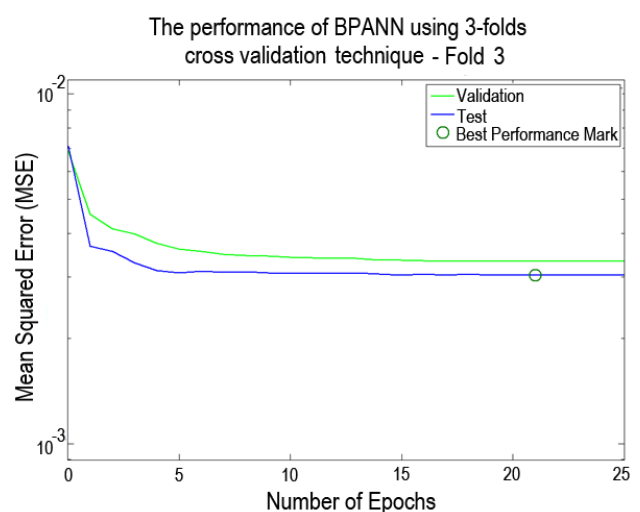


Fig. 7. The performance curve using the data samples of the third fold when 3-folds cross validation technique was applied. The performance of the training set is slightly better than that of the test set and the best performance was achieved at epoch 21 when the MSE reached about 0.0030 mark.

The number of epochs needed for the ANN model to converge was higher in the case where the resubstitution method was implemented. This is due to the fact that the number of training and test samples was higher than those of the 3-folds cross validation technique. However, this came at the expense of a lower MSE at 0.0015 using the resubstitution method, whereas the average MSE of all folds in the case where the 3-folds cross

validation technique was implemented was higher at about 0.0029.

5 Application to Materials Informatics

The results and analyses shown in section 4 validate that all of the five input design factors and the three responses form a good combination to design a VGCNF/VE material system. This observation can be proved by the small value of MSE obtained through the analysis as well as the relatively few number of epochs (an average of 17.25 epochs) required in order for the ANN to converge (i.e. to achieve the minimal MSE). However, when tan delta response was excluded from the analysis, the MSE started to increase (about 0.02). In addition, when the testing temperature was removed as an input design factor, the MSE started to significantly increase (about 0.24). At the same time, the number of epochs required for the ANN to converge was much higher (about 1000 epochs). On the other hand, when the VGCNF weight fraction was excluded as a design factor, the average MSE increased, but not as much as when the testing temperature was excluded.

These observations confirm that the testing temperature is the most dominant feature in the input design factors followed by VGCNF weight fraction. Tan delta is also very important as a response. However, all other input design factors had exhibited less sensitivity, as their impacts on the responses were much less than the testing temperature and the VGCNF weight fraction. The findings in this study confirm previous findings by Nouranian, et al. [17], where a response surface modeling approach was utilized to optimize the VGCNF/VE nanocomposite material system.

6 Summary and Conclusions

Artificial Neural Network (ANN) technique was applied to a vapor-grown carbon nanofiber (VGCNF)/vinyl ester (VE) nanocomposite dataset as a proof of concept for materials informatics. This dataset had previously been generated by a full factorial experimental design with 240 different design points. Each treatment combination in the design consisted of eight feature dimensions corresponding to the design factors, i.e., VGCNF type, use of a dispersing agent, mixing method, VGCNF weight fraction, and testing temperature as the inputs and storage modulus, loss modulus, and tan delta as the outputs. The ANN was trained using the resubstitution method and the 3-folds cross

validation (CV) technique to provide a predictive model for these responses when the inputs are fed to the ANN. The ANN was able to predict/model these responses with minimal mean square error (MSE) using both techniques. However, the MSE error was relatively lower in case of resubstitution method. This is due to the fact that more samples were used for training and testing when the resubstitution method was implemented. In the 3-folds CV technique, the dataset was split into two subsets: one for training and one for testing (validation), so the number of samples used for training and testing was lower. This came at the expense of more converging time (23 epochs) needed for the ANN when the resubstitution method was implemented.

The ANN applied here demonstrates the usefulness of data mining and knowledge discovery techniques in materials science and engineering. Specifically, the analysis of the dataset associated with a VGCNF/VE nanocomposite material system serves as proof of concept. It is expected that more knowledge discovery and data mining techniques will be employed within the rising field of materials informatics in near future.

Acknowledgments

The authors thank the Center for Advanced Vehicular Systems (CAVS) for supporting this research.

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