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Machine learning approach for analysing and predicting the modulus response of the structural epoxy adhesive at elevated temperatures

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Abstract

For bonded Fibre Reinforced Polymer (FRP) strengthening systems in civil engineering projects, the adhesive joint performance is a key factor in the effectiveness of the strengthening; however, it is known that the material properties of structural epoxy adhesives change with temperature. This present paper examines the implied relationship between the curing regimes and the storage modulus response of the adhesive using a Machine Learning (ML) approach. A dataset containing 157 experimental data collected from the scientific papers and academic theses was used for training and testing an Artificial Neural Network (ANN) model. The sensitivity analysis reveals that the curing conditions have a significant effect on the glass transition temperatures (T_g) of the adhesive, and consequently on the storage modulus response at elevated temperatures. Curing at an extremely high temperature for a long time does not, however, guarantee a better thermal performance. For the studied adhesive, curing in a warm (⩾ 45 °C) and dry (near 0 % RH) environment for 21 days is recommended for practical applications. A software with a Graphical User Interface (GUI) was established, which can predict the storage modulus response of the adhesive, plot the corresponding response curve, and estimate the optimum curing condition.

Keywords: Structural epoxy adhesive; Curing conditions; Storage modulus; Machine learning; Artificial neural network.

1 Introduction

The civil engineering industry has witnessed a rapid growth in the use of externally bonded composite materials to strengthen structures across the world [1–3]. This has stimulated the use of structural epoxy adhesives in civil structures, as it can offer the advantages of low additional weight, more uniform stress distribution, and design flexibility, when compared to conventional joining techniques [4–6]. However, when the ambient temperature increases, adhesive joints may perform differently than at a normal temperature, as the glass transition behaviour of the adhesive layer will result in a reduction in its strength and stiffness, consequently reducing the stress transfer capacity of the joint [7–10].

Traditional experimental investigations on the performance of adhesive layers can sometimes inevitably lack representativeness and generalisation. Using Machine Learning (ML) approaches enables the incorporation of available experimental results into an unified model, without the need for individual testing at each data point, which provides a more comprehensive and systematic understanding on the existing results. Through extracting complicated relationships between parameters and outputs, ML can further summarise implicit nonlinear laws and apply them to make new predictions, without involving labour-intensive work [11–13].

The first concept of ML was introduced in 1959 by Arthur Samuael [14], who defined ML as a field of study that enables computers to have the ability to learn without being explicitly programmed [15]. Since the 1990s, engineers have been using ML techniques to explore the complex behaviour of structural materials, among which artificial neural network (described further in Section 2) in one of the most widely used ML methods. For example, Ghaboussi et al. [16] successfully used an Artificial Neural Network (ANN) in 1991 to predict the behaviour of concrete subjected to certain loading paths, while Rao and Mukherjee [17] used an ANN in 1996 to predict the micromechanical response of ceramic matrix composites [12,18].

Over the last decade, several studies have been carried out using ML ANN approaches to analyse the response of adhesively bonded joints, particularly for single lap-shear joints. A back-propagation ANN model for predicting the bond strength of Fibre Reinforced Polymer (FRP)-to-concrete joints was first proposed by Mashrei et al. [19] in 2013. Tosun and Çalık [20] developed an ANN model to estimate the strength of aluminum-to-aluminum single lap-shear joints based on their geometric dimensions in 2016, while in 2021, Gu et al. [5] further developed an upgraded ANN predictive model, which comprehensively considers the combined effects of continuous and discrete design (geometry and material) variables. Besides these, ANN approach can also be applied to develop constitutive material models for adhesive bonds. For instance, the rate dependent response of bonded joints was studied by Zgoul [21] using a proposed ANN constitutive model; and most recently, a ML material model that can be used in finite element analysis to estimate the element failure was developed by Sommer et al. [18]. These studies demonstrate the reliability of the ML ANN approach; however, they have been constrained to adhesively bonded joints rather than the structural adhesive itself and neither of them considers the effects of curing conditions or elevated environmental temperatures.

Studies using ML approaches to explore the thermal performance of structural epoxy adhesives are rare. Jha et al. [22] and Tao et al. [23] were dedicated to establishing correlations between the chemical structure and the glass transition behaviour of polymers. Szabelski et al. [24] applied the ANN method to investigate the influences of adhesive mixing ratios and curing conditions on the tensile strength of bonded joints. The results indicated that the higher curing temperature resulted in a relatively higher tensile strength, but the higher test temperature resulted in a significantly lower tensile strength. This demonstrates the importance of studying the mechanical behaviour of adhesives in relation to their curing conditions and service ambient temperatures. So far, there is a lack of research using machine learning approaches to analyse the implicit relationship between the modulus response of adhesives and the applied curing conditions.

The aims of this study are to explore the influence of elevated temperatures on the storage modulus response of the structural epoxy adhesive, and how the applied curing conditions related to the thermal performance of the adhesive. This is achieved by utilising the ML approach, as the traditional curve fitting method is not applicable due to the complexity of the T_g behaviour, the curing regimes, and the variety of different test data. The ML approach can enable a better understanding of experimental data and incorporate them into a unified model for further comprehensive analysis and prediction of the thermal response of the adhesive. The predictions obtained without conducting time-consuming and expensive experimental tests can provide reference values for practical engineering applications.

A predictive ANN model is proposed to map the relationship between the modulus response and the curing conditions. And for the first time, a user-friendly software with a Graphical User Interface (GUI) which implements the proposed ANN model is developed.

2 Artificial Neural Network (ANN)

ANN is a machine learning technique with flexible mathematical structures inspired by the networks of biological neurons in human brains. A typical ANN system comprises one input layer, one or more hidden layers and one output layer. After training, the system is capable of identifying complex nonlinear relationships between input and output data [9,25,26].

As shown in Figure 1, like biological neurons receiving signals from dendrites, all input parameters obtained from the dataset form the input layer of the ANN. The received parameters information is gathered in the hidden layer(s) and modulated through an activation function before being output to the output layer. This process can be expressed as follows [9,26,27]:

$$y_i = \sigma\left(\sum_{i=1}^{n} W_i^k I_i + b_i\right) \tag{1}$$

where y_i refers to the output of neuron i, I_i refers to its input, σ is the activation function, W_i^k represents the weight, and b_i represents the bias [26].

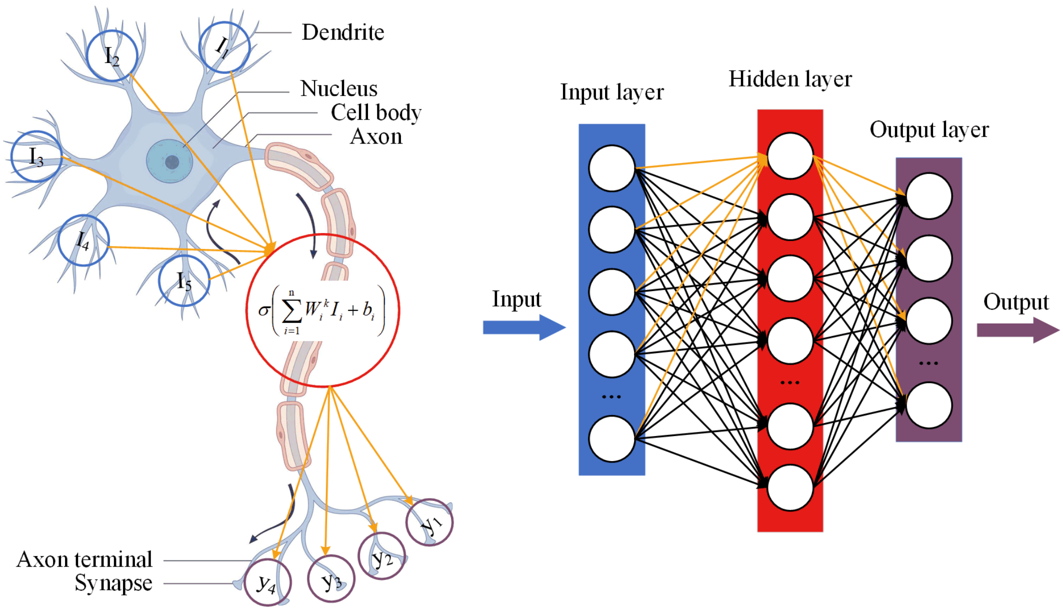


Figure 1: The schematic illustration of an ANN structure

The number of neurons varies greatly among different problem cases, and therefore, trial and error is necessary to determine the structure of the ANN network before the network training. The goal of training an ANN is to minimise the network error by optimising the weight factors (W_i^k) that represent the strength of connections between neurons. Mean Square Error (*MSE*) is one of the criteria used to evaluate the performance of a network [9,26,28]:

$$MSE = \frac{1}{N}\sum_{i=1}^{N} (t_i - \bar{y}_i)^2 \tag{2}$$

where N is the number of data, t_i is the target value, $\bar{y}_i$ is the predicted value. The training process is iterative, where $\bar{y}_i$ is estimated by assigning a random weight (W_i^k) value each time, and the corresponding *MSE* is calculated. The initial weight (W_i^k) value is continuously updated until the *MSE* falls within a satisfactory range, and this is known as a back-propagation algorithm [26,28,29].

The basic concepts of the ANN machine learning technique have been clarified above, and more detailed information can be found in the cited references. In the next section, the development of the datasets is presented.

3 Data acquisition and pre-processing

Dynamic Mechanical Analysis (DMA) is a widely used technique for measuring the storage modulus of materials as a function of temperature [30,31], which has been used in a number of studies to determine the thermal properties of adhesives. In the present paper, the DMA experimental results of a structural epoxy adhesive (Sikadur-330 [32]) were extracted from a total of 11 scientific papers and academic theses to form two datasets. Dataset A containing 157 experimental results was used to develop the ANN model, while dataset B containing 6 experimental results was used to carry out subsequent validation work.

The adhesive, Sikadur-330, selected for this study is an ambient-cured epoxy that is frequently used to apply FRP-bonded strengthening in infrastructure projects. The material consists of a thixotropic, solvent-free, bi-component epoxy-based adhesive, and its chemical structure comprises a bisphenol-A based epoxy resin and aliphatic amines as hardeners, with small amounts of silica-based fillers. The recommended mixing ratio in weight of the resin to the hardener is 4:1 in the material data sheet [32,33].

Establishing a ML model for all types of structural adhesives was not possible within the scope of the current project, which would require a separate research work to collect an extremely massive database. Nevertheless, the research methodology demonstrated in this paper and the obtained implied relationship between parameters and outputs can provide a useful reference for studying other structural adhesives.

3.1 Critical points for analysing the modulus response

An indirect data training approach was used in this study, as the modulus response is a continuous result that cannot be used directly to generate the output part of the dataset. A summative output part with limited variables is required to prevent overfitting issues [34]. Figure 2 illustrates an example of the temperature-dependent storage modulus curve (black line) of the adhesive obtained from a DMA test [35], where the five critical modulus points related to the glass transition temperature (T_g) of the adhesive are marked:

- Initial modulus (E1): the storage modulus at room temperature (around 20 °C);
- Modulus corresponding to Onset T_g (E2): the intersection of two lines tangent to the glassy and leathery portions of the modulus response curve [30,36];
- Modulus corresponding to Inflection point T_g (E3): the point of inflection of the leathery portion of the curve [30,36];
- Modulus corresponding to Peak Tan δ T_g (E4): the modulus at which the maximum Tan δ (the ratio of loss modulus to storage modulus) occurs [30,36];
- Final modulus (E5): the storage modulus stabilises at the highest temperature (approximately 100 °C).

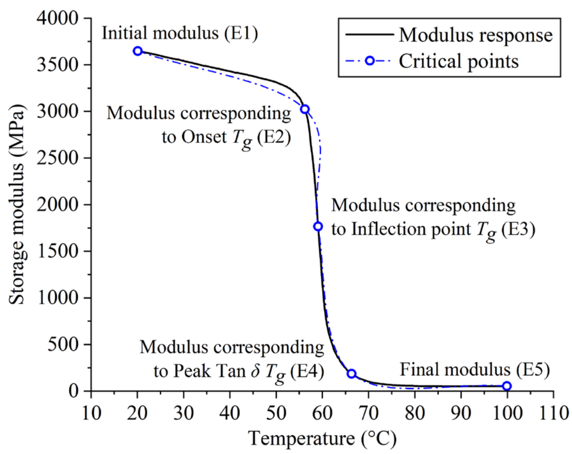


Figure 2: Simplify the modulus variation at elevated temperatures using the critical points

The entire temperature-dependent modulus response curve can be estimated by connecting the five critical points. As shown by the blue line in the figure, the estimated curve was generated using the modified Bezier line connection method in the Origin Lab software [37]. The corresponding five critical points data were therefore extracted from the experimental results in the literature to form the summative output parts of the datasets. Note that the obtained five critical points data were not filtered, which ensured the authenticity of the created dataset, although there may have been slight errors during the process of extracting the data (E1-E5) by drawing the auxiliary lines manually.

3.2 The development of the datasets

Table 1 summarizes the literature used to construct the dataset A and dataset B. 157 DMA experimental results from 9 references (dataset A) were used to develop (through training and testing) the ANN model (described in Section 4), and the other 6 results from 2 references (dataset B) were used to validate the established user-friendly software (described in Section 6). The DMA tests were carried out by applying dynamic flexural loads to the samples. A more detailed description of each test can be found in the cited references listed in Table 1. The experimental data from Othman's research at the University of Edinburgh [38] forms a large part of the datasets, which is due to the lack of other comprehensive experimental studies on the effect of different curing conditions on the modulus response of the studied adhesive. Whilst a more comprehensive dataset would be preferable, additional experimental work was beyond the scope of this study.

Table 1: Reviewed literature for constructing the datasets

No.	Reviewed Literature	No. of samples
1	Othman [38]	144
2	Wang et al. [10]	3
3	Wang et al. [35]	1
4	Sousa et al. [33]	1
5	Seif et al. [39]	1
6	Sun et al. [40]	3
7	Verdet et al. [41]	1
8	Lartigau et al. [42]	1
9	Savvilotidou et al. [43]	2
(Dataset A: 157 samples for developing the ANN model)		
10	Stratford and Bisby [7]	5
11	Burke et al. [44]	1
(Dataset B: 6 samples for validating the established software)		

The raw data of the dataset A and dataset B are listed in the supplementary material. Table 2 presents the features of the dataset A used for developing the ANN model.

Table 2: Statistical summary of the dataset A used for developing the ANN model

Variables	Min	Mean	Max	Coefficient of variation
Inputs				
Curing temperature (°C)	13	43.6	80	52.1%
Curing time (days)	3	13.2	34	73.5%
Curing humidity (%)	0	49.5	100	97.0%
Outputs				
E1: Initial modulus (MPa)	1952.9	3136.4	4172.9	14.2%
Onset T_g (°C)	28.5	52.3	74.9	22.6%
E2: Modulus corresponding to Onset T_g (MPa)	1111.6	2273.0	3281.7	21.3%
Inflection point T_g (°C)	34.9	58.8	80.3	21.1%
E3: Modulus corresponding to Inflection point T_g (MPa)	649.9	1289.4	2107.3	22.9%
Peak Tan δ T_g (°C)	46.5	67.3	89.1	17.5%
E4: Modulus corresponding to Peak Tan δ T_g (MPa)	66.5	234.6	575.6	46.2%
E5: Final modulus (MPa)	19.9	40.2	78.2	34.8%

The raw data needs to be normalised at the pre-processing stage, which can accelerate ANN training and improve prediction accuracy, especially for this study where the ranges of values of the variables (inputs and outputs) are significantly different. The min-max scaling method is applied [5,9]:

$$X' = \frac{X - X_{min}}{X_{max} - X_{min}} \tag{3}$$

where X represents the raw data, X' represents the normalised data, X_{min} and X_{max} refer to the minimum and maximum values of the target variable in the whole dataset. The full normalised dataset A is listed in the supplementary material as well.

4 Establishment of the ANN model

The construction process of the ANN model is shown in Figure 3. The Bayesian Regularization algorithm was used to train the ANN, which randomly divided the applied dataset A into two parts, 80% (126 samples) for training and 20% (31 samples) for testing. Whilst this algorithm usually requires more time, it can minimise the combination of squared errors and weights to train an ANN that performs reasonable generalisation for difficult, small, or noisy datasets [45,46]. The proposed ANN consists of one input layer, one hidden layer and one output layer. The activation functions for the hidden and output layers adopted the TANSIG (Equation (4)) function and the PURELIN function (Equation (5)), respectively. The TANSIG function produces data values between [-1, +1], while the PURELIN function keeps the inputs constant [28,45].

$$y = \mathrm{TANSIG}(x) = \frac{2}{(1 + e^{-2x})} - 1 \tag{4}$$

$$y = \mathrm{PURELIN}(x) = x \tag{5}$$

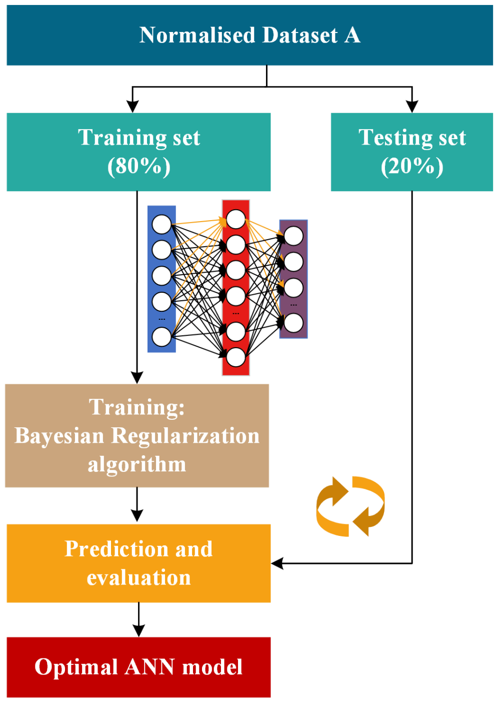


Figure 3: Construction process of the ANN model

The number of hidden-layer neurons was determined through trial and error. As shown in Figure 4, 20 neurons were applied to construct the ANN model as this resulted in the lowest MSE (Equation (2)).

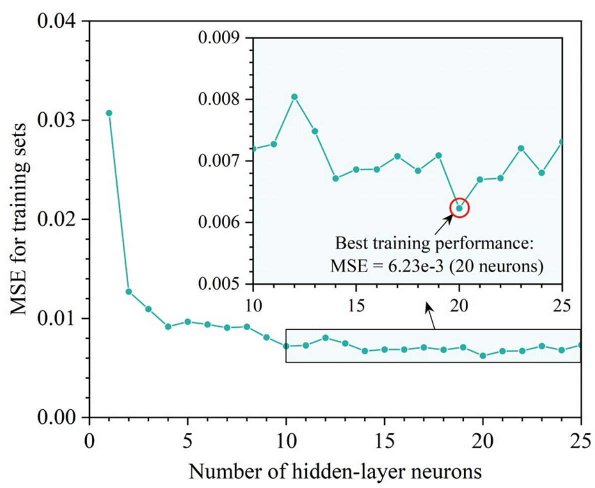


Figure 4: Training mean squared error with different numbers of hidden-layer neurons

5 Results and discussions

In this section, the performance of the ANN model is presented, and the predicted results of the modulus response are compared with the experimental results from the literature to verify the model. The complex relationship between the curing conditions and the modulus response is then analysed using the developed ANN model.

5.1 Model performance evaluation

Figure 5 illustrates the best training performance of the ANN model, which was obtained at the 363rd epochs with the MSE (Equation (2)) equals to 6.23e-3. The corresponding regression diagrams of the training set and testing set at the 363rd epochs are shown in Figure 6, where Y and T refer to the normalised predicted values and target values.

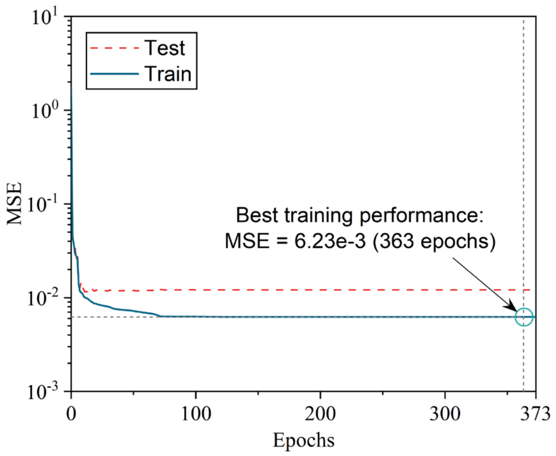
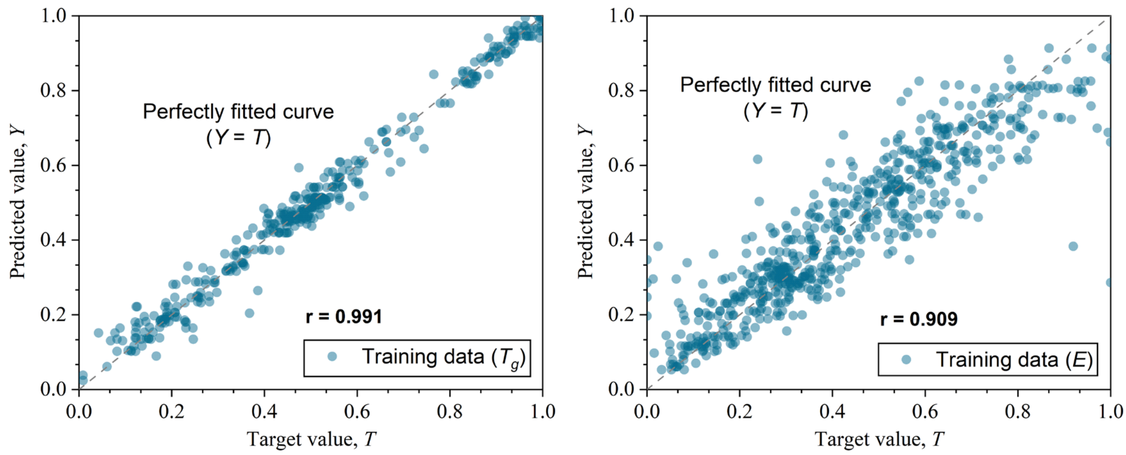
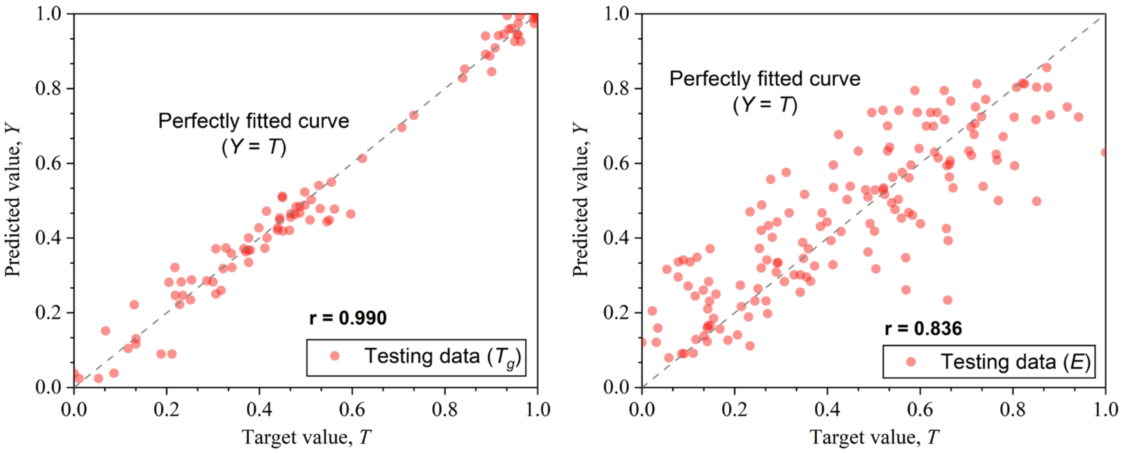


Figure 5: ANN training performance



(a) Training data (T_g) (b) Training data (E)



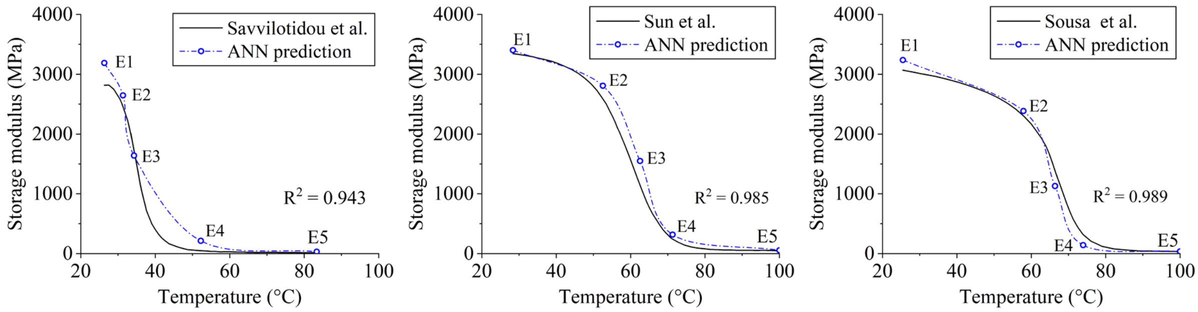
(c) Testing data (T_g) (d) Testing data (E)

Figure 6: Regression of the developed ANN model

The correlation coefficients (r) corresponding to the T_g values (Onset T_g, Inflection point T_g, and Peak Tan δ T_g) were higher than those corresponding to the modulus values (E1 – E5), which indicates that the trained ANN model was able to predict the adhesive's T_g values more accurately. Nevertheless, the overall response with the correlation coefficients close to 1.0 suggested that the training produced the optimal results.

5.2 Verification of the developed ANN model

The predicted values need to be post-processed to obtain the modulus response of the adhesive due to the indirect data training approach used in developing the ANN model. The comparison of the predicted modulus response curves (using the modified Bezier line connection method in the Origin Lab software [37]) and the experimental results (storage modulus versus temperature curves) from scientific papers are shown in Figure 7. The three selected representative comparison results correspond to the glass transition behaviour occurring in the low, medium, and high temperature ranges. The coefficient of determination (R^2) of each comparison is illustrated in Figure 7.



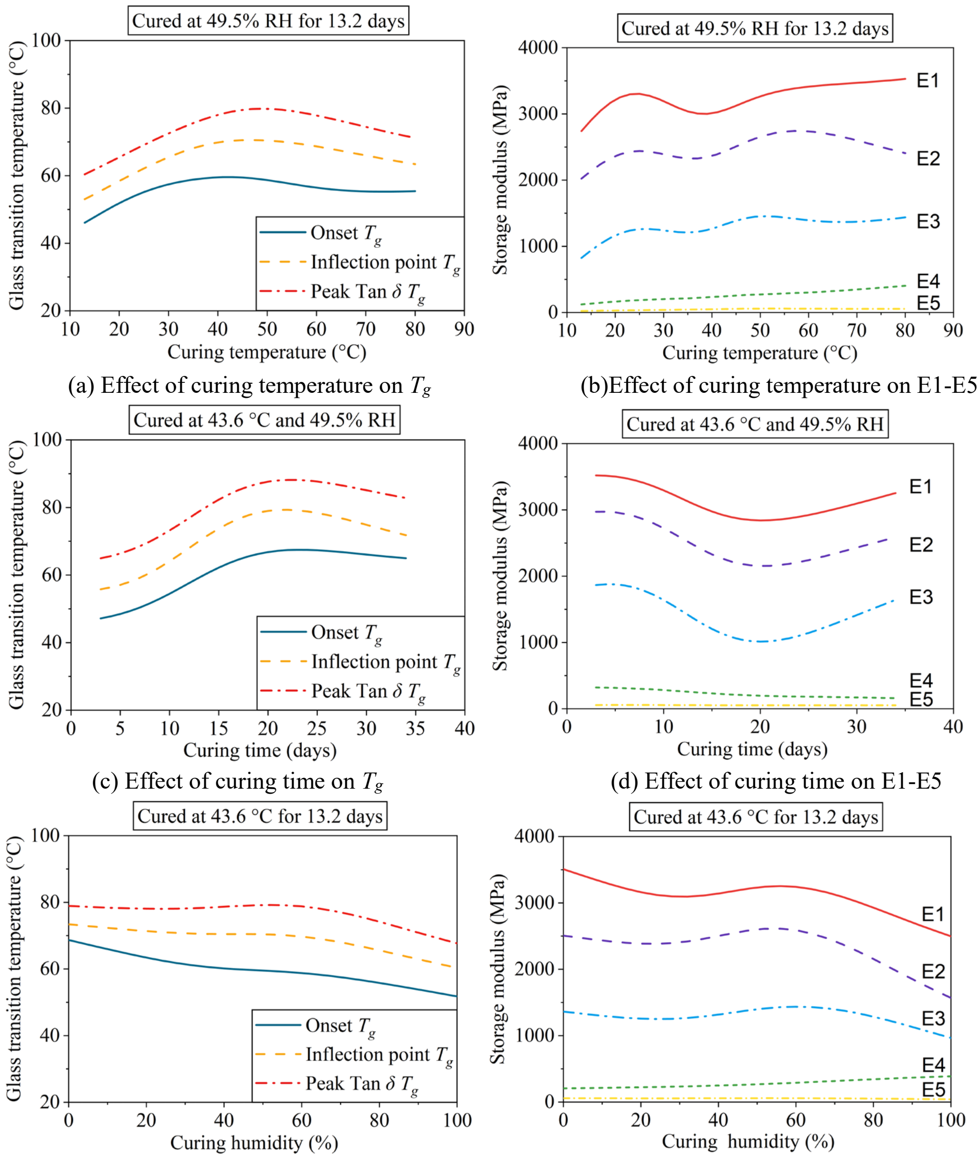
(a) In the low temperature range (b) In the medium temperature range (c) In the high temperature range

Figure 7: Obtained predicted results compared with published results

The predicted modulus response of the adhesive at elevated temperatures is suitably accurate from the viewpoint of engineering practice. The ANN model developed using the dataset A (Table 1), which contains a large proportion of data from Othman's experiments [38], can also be used to accurately predict the results from other experiments, demonstrating the great generality of the ANN machine learning approach. Note, however, that for the glass transition behaviour occurring in the low temperature range, if the precise modulus response is of interests, an extra experiment may still be necessary.

5.3 The effect of curing conditions on the modulus response at elevated temperatures

The developed ANN model made it possible to obtain a substantial number of predictions conveniently, which allowed for a comprehensive analysis of the relationship between the curing conditions (inputs) and the modulus response at high temperatures (outputs). The effects of curing temperature, curing time, and curing humidity, respectively, on the T_g and modulus values of the adhesive are visualised in Figure 8. The benchmark curing condition was set at 43.6 °C and 49.5% RH for 13.2 days, based on the mean values of the variables in the dataset A (see Table 2).



(a) Effect of curing temperature on T_g (b)Effect of curing temperature on E1-E5

(c) Effect of curing time on T_g (d) Effect of curing time on E1-E5

(e) Effect of curing humidity on T_g (f) Effect of curing humidity on E1-E5

Figure 8: The effect of curing conditions on the critical points of modulus response at elevated temperatures

- As shown in Figure 8 (a), by keeping the benchmark curing time and curing humidity constant, as the curing temperature increases from 13 °C, the crosslinking of the epoxy adhesive increases and the free volume decreases, which results in the T_g values growing until an approximate plateau is reached. Note that the slight reduction occurring during the plateau period is due to the thermal degradation or oxidative crosslinking behaviour caused by high curing temperatures. This was also observed experimentally by Carbas et al. [31]. The variation in the modulus values (Figure 8 (b)) is relatively small (noting the different vertical scale ratios), with a general tendency to increase with increasing curing temperature. The fluctuations in the modulus values are partly caused by the combined errors in the collected different experimental data and the training process (see Figure 6 (b) and (d)).

- Similarly, as the curing time increases from 3 days, the predicted T_g values continue to increase until they approach an approximate plateau, as shown in Figure 8 (c). The modulus values (mainly E1, E2, and E3), however, decrease firstly and then increase (see Figure 8 (d)).
- The humidity is detrimental to the thermal performance of the adhesive, as indicated in Figure 8 (e) and (f). Both the T_g and the modulus reduce as the curing humidity increases from 0% RH to 100% RH.

The curing conditions have a significantly impact on the temperature at which the glass transition behaviour of the adhesive begins, however, the impact on the specific modulus values at different stages of its transition from the glassy state to the rubbery state is limited. Curing the adhesive at an extremely high temperature over a long period of time is not conducive to practical engineering applications and this does not necessarily lead to a better thermal performance due to the thermal degradation or oxidative crosslinking effect. As a result, cured at an adequate warm temperature (≥ 45 °C) for around 21 days is sufficient for the studied adhesive, and the curing is preferably conducted in a dry environment.

6 Graphical User Interface (GUI) design

Applying the original ANN model requires further coding work, which could be an obstacle for civil engineers. Establishing a user-friendly software that implements the developed ANN model can facilitate turning machine learning results into practical applications. The designed software with a GUI was programmed using MATLAB, and the source code is publicly available in the supplementary material.

As shown in Figure 9, the software is capable of predicting the different modulus responses of the adhesive according to the different applied curing conditions. The modulus response curve can be generated based on the selected set of output values. In addition, the optimum curing condition can be calculated with respect to the maximum onset, T_g, at which the modulus of the adhesive starts to drop significantly. The optimum curing condition is estimated to be cured at 56.6 °C and 0% RH for 23.2 days, which agrees with the recommendation proposed at the end of Section 5. Utilising these functions of the software can provide more comprehensive results on the response of the adhesive.

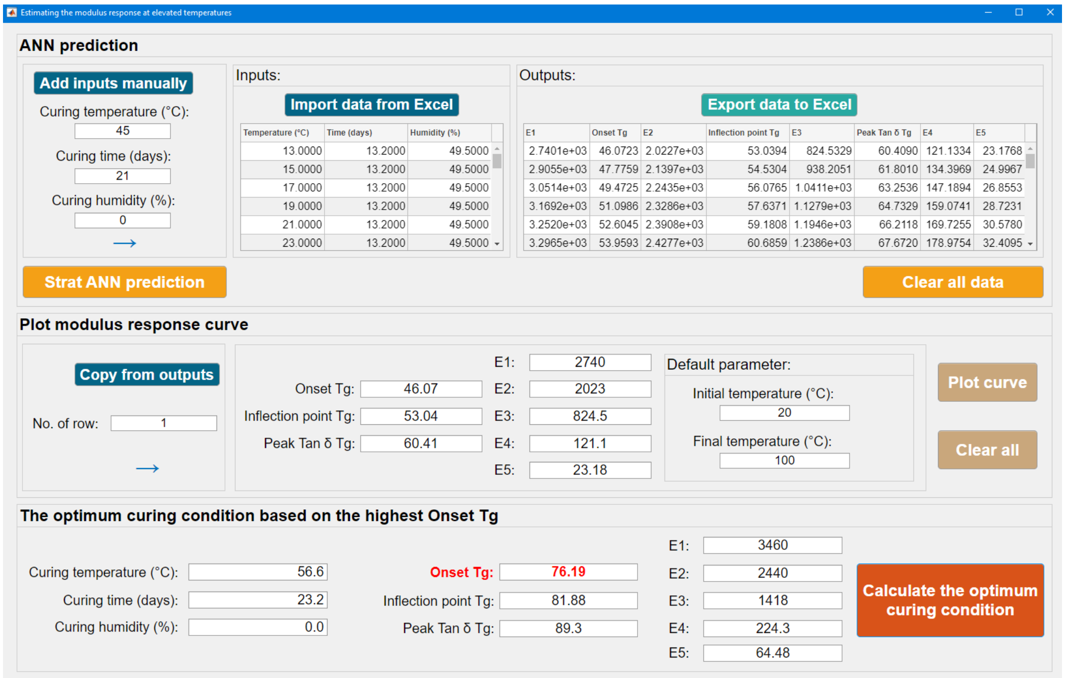
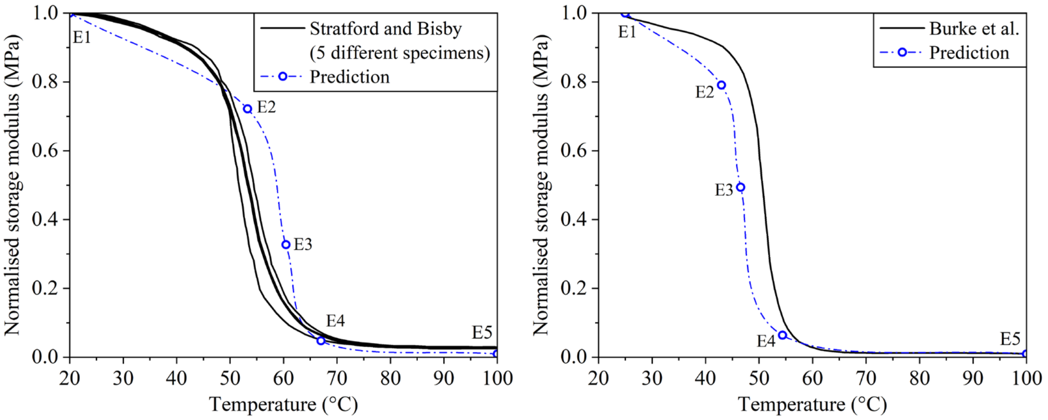


Figure 9: GUI for estimation of the modulus response at elevated temperatures

The effectiveness of the software was validated by comparing the predicted results with the experimental results from two references separately, as shown in Figure 10. The results in these 2 references were not used in training the ANN model (unlike in Figure 7), which ensures a reasonable validation.



(a) compared with the experimental results in [7] (b) compared with the experimental results in [44]

Figure 10: Validation of the established software

Whilst the errors in the specific T_g values are approximately ± 5 °C, the shape of the curve representing the tendency of the modulus to decrease at elevated temperatures can be well estimated. The software can be upgraded when more data is available to train a more robust ANN model and to include consideration of different chemical formulations for different types of structural adhesives. The current version can be used as a simple tool to conveniently estimate the approximate modulus response of the adhesive based on the applied curing condition, without the need for experimentation; however, caution is necessary in applying this software to obtain specific T_g or modulus values.

7 Conclusions

This study proposes using the machine learning approach for analysing and predicting the storage modulus response of the structural epoxy adhesive (Sikadur-330 [32]) at elevated temperatures. By utilising its powerful data processing and inductive learning capabilities, the links between parameters and results are inferred and analysed, which enables us to gain a deeper understanding of the patterns implied in the extensive experimental results.

An artificial neural network model was developed to map the relationship between the modulus response and the applied curing conditions, and to provide predicted reference values for practical engineering applications. The analytical results of the ANN model were verified against the published experimental studies, demonstrating the powerful generalisation and prediction capabilities of the ML model. The sensitivity analysis of variables was performed based on the massive prediction results generated using the ANN model.

- The effects of curing conditions on the T_g results of the adhesive are more significant than those on the modulus results. As the curing temperature or curing time increases, the T_g values will increase until the thermal energy is sufficient to complete the crosslinking, leading to an approximate plateau.
- Increasing curing humidity can result in a decrease in the T_g and modulus, which is detrimental to the thermal performance of the adhesive.
- It is recommended to cure the studied adhesive at a warm temperature (≥ 45 °C) and in a dry environment (close to 0% RH) for around 21 days in practical applications.

A user-friendly software with a Graphical User Interface (GUI) has been established by implementing the ANN model. The efficiency and capability of this simple tool have been illustrated. Using its powerful computational capabilities, the optimum curing condition for the examined adhesive was estimated to be 23.2 days at 56.6 °C and 0% RH. However, experimental confirmation would be required when using the software for design purposes.

Like any other machine learning method, the generalisability of the ANN model might be limited to the range of the dataset used. The dataset is expected to be updated as more experimental data becomes available to train a more robust ANN model and to consider different chemical formulations for different types of structural adhesives. Nevertheless, the present study demonstrates the use of a machine learning approach to comprehensively analyse and predict the modulus response of a structural epoxy adhesive according to the applied curing condition.

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Disclosure statement

The authors report there are no competing interests to declare.

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Data availability statement

The supplementary material that supports the findings of this study is publicly available in *figshare* at *https://doi.org/10.6084/m9.figshare.21640772.v1*.

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