

Predicting mechanical properties of elastomers with neural networks

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Abstract

Despite the existence of a solid theoretical basis interrelating various mechanical properties of elastomers, the complexity of these materials and strong dependence of characteristic material parameters on deformational and temperature conditions cause insuperable difficulties in establishing accurate relations between the crosslinking properties of elastomeric compounds and crosslinked elastomers in practice. Since knowledge of such presumably nonlinear relations would be valuable for several reasons, this work attempts to uncover these relations using methods of soft computing, in particular neural networks. The resulting relations obtained by neural network analysis have proved to be incontestably good and completely in accordance with expectations, thus contributing to proficiency in dealing with elastomeric materials, as well as curtailing possibilities of testing redundancy.

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1. Introduction

Elastomers are a class of polymers characterized by the high, or rubber, elasticity of the compounds. Rubber, a basic elastomeric material, has a complex morphological structure, which becomes even more intricate in the case of compounds with other materials, e.g. active fillers. Although theoretical grounds for understanding and describing the behaviour of elastomers and the effects of various ingredients in elastomeric compounds are rather firmly established [1–3], the complexity of these materials makes it difficult to accurately predict the properties of the resulting rubber products in practice. For practical applications, elastomeric compounds must undergo a composite chemical process of crosslinking, by which their long, entangled molecules are covalently linked to form a three-dimensional network. Moreover, the material properties of elastomeric compounds are affected by additional factors, such as deformational and environmental conditions, and

thermal and chemical fatigue, all of which could hardly be encompassed in an analytical model, accurately predicting the behaviour of rubber products. Even if such models were feasible, they would be increasingly complicated, thus potentially obscuring their primary purpose. Assuring high-quality manufacturing of rubber products, therefore, invariably demands that a multitude of laboratory tests is performed on the elastomers, which is costly and time consuming.

Tests intended to assess the properties of elastomers and those of the end products are roughly of two types: rheometric examination is used to determine the resistance of elastomeric compounds to flow and thus their ability to crosslink, which is essential for compound processing, while diverse mechanical tests on crosslinked elastomers, or vulcanizates, are mainly used to predict the properties of the end products. These categories differ significantly in the time the tests take to perform: whereas the former are minute-scale tests, completion of the latter takes substantially longer, typically lasting hours or even days, which is primarily due to lengthy sample preparation and the particular requirements ordinarily imposed by standards. Thus, the main idea of this work is to search for strong relationships between rheometric testing and tests on

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vulcanizates, which, apart from a theoretical contribution, would also enable minimization of certain routine tests or even their complete abandonment.

Indeed, such relationships must exist, since all the properties identified in individual tests ultimately stem from a single source, i.e. elastomeric structure, regardless of how complex it may be. For example, all the mechanical properties of a vulcanizate unequivocally depend on crosslink density, the main morphological trait of crosslinked elastomers. Yet, in general, these relationships are not simple and are rarely expressible analytically. Particularly at high strains, where material response is nonlinear, the relationships are increasingly complicated and theoretically difficult to determine.

At present, however, there is an effective way to circumvent this obstacle. Instead of the tantalizing search for analytical solutions, modern techniques of data mining by soft computing methods can be used [4,5], enabling extraction of the knowledge implicitly stored in large databases. To find complex relations in the data that even experts may miss, various data mining models have been devised. Among them, neural networks [6–8], as general nonparametric and robust models designed to find nonlinear relationships between specified input–output data samples, have progressively gained popularity. To illustrate a recent application of neural networks in solving problems concerned with elastomers, it is worth mentioning the effective search of a relationship between the viscosity of elastomers and their molecular weight distribution [9]. Successful results obtained by neural networks have been further achieved in finding and simulating property–formulation relations, using nitrile [10] and styrene–butadiene [11] rubber-based compounds, respectively. There have also been attempts to use neural networks in rubber processing and quality control [12]. Due to the good generalization and adaptation properties of neural network models, as well as their ability to learn from examples, data mining by neural networks was applied in the present work for tackling the problem of relating rheometric and mechanical tests.

A brief overview of elastomeric testing is given in the Section 2. In Section 3, an applied neural network prediction model is outlined. In Section 4, the experimental setup and

results in terms of prediction accuracy are explained. The main findings are given in Section 5.

2. Testing of elastomers

Elastomeric compounds are tested in their raw or uncross-linked state, as well as in their covalently crosslinked state. The equipment for performing the typical tests on elastomers is shown in Fig. 1.

Crosslinking, or commonly vulcanization, is a process comprising several chemical reactions between an elastomer and various crosslinking agents, such as reaction activators, accelerators and link-forming agents, originally sulphur. The end result of crosslinking is the formation of covalent intermolecular linkages, resulting in a three-dimensional molecular network, popularly known as vulcanized rubber [13]. Traditionally, the process is thermally activated, i.e. taking place at elevated temperatures. In the case of sulphur as a link-forming agent, crosslinks of the type C–S_x–C are usually formed between carbon atoms of spatially adjacent elastomeric molecules, S_x being a chain or ring of *x* sulphur atoms, frequently eight. There are, of course, other agents, such as certain metal oxides and resins, that form crosslinks, whereas in the case of so-called peroxide vulcanization [14], the carbon atoms are linked directly, forming C–C type crosslinks.

Although all the details of this widespread vulcanization process are not entirely clear, perhaps the most comprehensive chemical account is given by the vulcanization scheme proposed by Coran [13,15]. The detailed chemical kinetics of crosslinking reactions is very complicated, and therefore not readily applicable. There are, however, efficient methods for tracing net vulcanization kinetics [16], which have proved to be extremely valuable in the rubber industry. One such method is a specific branch of rheometry called vulcametry. It is based on a theory of rubber elasticity [17] that stipulates the proportionality of crosslink density in an elastomer and its modulus of elasticity, or more generally, its resistance to strain. Thus, any possible measurement of increasing compound strain resistance with time during vulcanization implies indirectly ascertaining the compound's crosslink density. The method

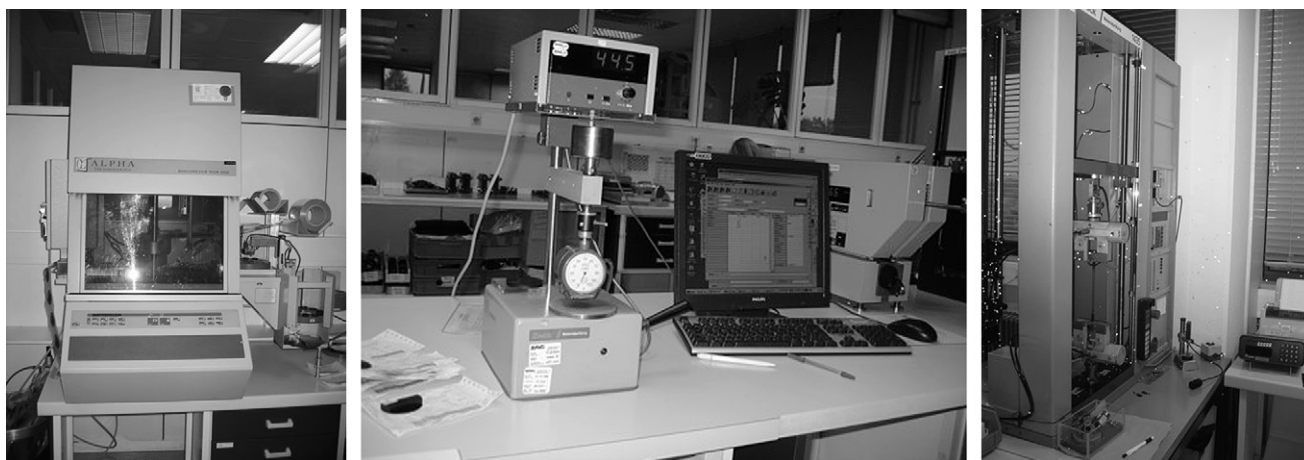


Fig. 1. Equipment used to measure rubber compound properties: moving die rheometer (left), hardness tester (centre) and tensile testing machine (right).

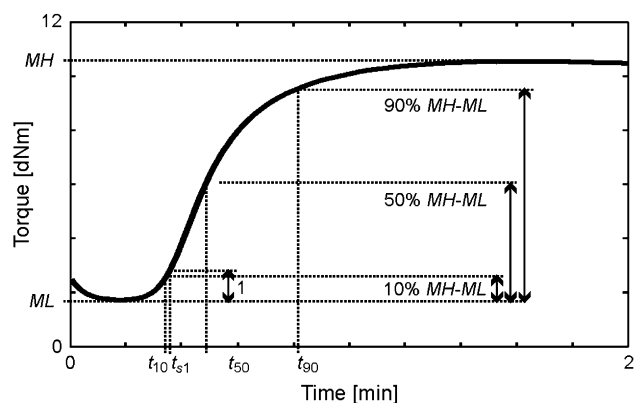


Fig. 2. Vulcanometric curve representing the crosslinking process as torque versus time, with characteristic point parameters ML, MH, t_{10} , t_{50} , t_{90} and t_{s1} .

is simple, fast and accurate, and hence widely used. Measurements are performed using devices called rheometers, or vulcameters (Fig. 1), where a die-confined compound sample at an elevated temperature is exposed to oscillating torsion. The sample's response due to increasing crosslink density is manifested in the form of time-dependent torque imposed on a strain gauge measuring system. The recorded torque as a function of time represents the so-called vulcanometric curve of the specific elastomeric compound, an example of which is shown in Fig. 2.

As can be seen from Fig. 2, the curve exhibits certain distinguishing features associated with processes occurring in the compound at different phases of vulcanization. At the beginning, the curve falls, due to a drop in viscosity of the heated compound, to reach the minimum torque denoted by ML. In accordance with the Coran vulcanization scheme, the conglomeration of chemical reactions runs from the minimum to about the point of inflection, where the reaction accelerator should just be used up. From here on, a single first-order reaction between elastomeric molecules and sulphur proceeds to the maximum torque, denoted by MH. Such elucidation of a vulcanometric curve seems plausible and is generally in good agreement with other experimental approaches [16]. Although differing in chemical mechanisms, vulcanization with other crosslinking systems may schematically be explained in a similar way. It should be noted, though, that apart from covalent crosslink density, the vulcanometric curve also encompasses the density of secondary linkages of the van der Waals type [18], such as those formed by active fillers in an elastomeric matrix.

There are other commonly used point parameters indicated on the vulcanometric curve that characterize vulcanization kinetics. The points t_{10} , t_{50} and t_{90} designated, represent the times the process reaches 10%, 50% and 90% values of MH–ML torque, respectively, or, accordingly, the corresponding percentages of crosslink density. The time t_{s1} is the standard-defined scorch time [19], characteristic of the onset of crosslink formation.

Tests on crosslinked elastomers, on the other hand, generally comprise determination of mechanical properties important for end products, since the latter are invariably in

a crosslinked state. In this context, the most frequently determined parameters are hardness (H), various moduli of elasticity (in this work expressed as tensile stresses at 50% and 100% of tensile strain, denoted by M_{50} and M_{100} , respectively), elongation at break (E) and tensile strength (TS), as part of overall elastomeric compound characterization as well as quality control. These parameters are typically ascertained by standardized testing [20], demanding time consuming sample preparation, including vulcanization, and scrupulous measurement. As all of these parameters involve the material's response to strain, they clearly depend on crosslink density.

Although information characterizing vulcanizates is inherently present in vulcanometric curves, precise analytically expressible relations between the curve's characteristic parameters and the aforementioned parameters do not exist, even if measured under comparable deformational and temperature conditions. Generally, however, these conditions are far from being similar, and thus nonlinearities become even more pronounced. Consequently, nonlinear models such as neural networks might be appropriate for relating the mechanical and vulcanometric properties of elastomeric materials.

3. Neural networks

As suggested by the name, the basic idea behind neural networks [6–8] is to replicate the biological structures and functions of the brain. Such networks are capable of learning by example and generalizing the acquired knowledge. Due to these abilities, neural networks are widely used to ascertain nonlinear relations between parameters when no other adequate analytical description is available or is too complex. The learned knowledge is unfortunately hidden in the structure and cannot readily be extracted and interpreted.

In the present work, a neural network called a multilayer perceptron, as shown in Fig. 3, is used. It comprises basic computational units, called neurons, which are organized in two layers. Each neuron receives data in its input either from inputs to the neural network or outputs of neurons in the previous layer. Its output value is calculated as

$$y_i^l = f \left(\sum_j w_{ij}^l y_j^{l-1} \right) \quad (1)$$

where y_i^l is the output of the i -th neuron in the l -th layer, w_{ij}^l the weight of connection between the i -th neuron in the l -th layer and the j -th neuron in the $(l-1)$ -th layer, and f a certain transfer function. For simpler notation, inputs to the neural networks (x_1, \dots, x_N) are denoted as $y_i^0 = x_i$, $i = 1, \dots, N$. The neurons in the hidden and output layers have a nonlinear transfer function, the hyperbolic tangent shown in detail on the right of Fig. 3.

The topological structure of the multilayered perceptron, i.e. the number of hidden layers and the number of neurons, determines its capacity, while knowledge about the relations between input and output data is stored in the weights of connections between neurons. The values of weights are updated

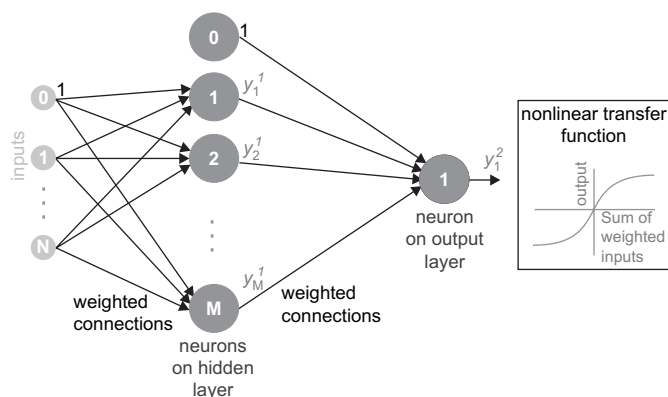


Fig. 3. A multilayer perceptron N - M -1 having N inputs, M neurons in a hidden layer and 1 output neuron. The number of weights is calculated as $(N + 1)M + (M + 1)$.

in the supervised training process with a set of known and representative values of input–output data samples. Input and output values are usually normalized for nonlinear transfer functions to operate in the active region. In this work, each input is normalized to zero mean and unit variance.

All available input–output samples are divided into training and testing sets. The training set is used to establish the values of the weights and thus build the model, whereas the testing set is used for testing of dependencies. At the beginning of the training process, the weights are set randomly. With respect to the squared difference between the target value and the calculated output value on the output neuron, the weights of all connections are updated with a minimization algorithm. The Levenberg–Marquardt back-propagation minimization algorithm [21], which involves performing computations backwards through the network, is used to update the neural network weights. Training itself is a multidimensional optimization process which is extremely sensitive to local minima. To achieve better results, the training process on a neural network model with the same topological structure is usually repeated many times, each time randomly setting initial model weights.

4. Experimental results and discussion

As the aim of this work is to find a single general purpose model applicable to as many elastomeric compounds as possible with no input data on molecular structure, a multitude of different compounds was included in the experiments, based on the following series of rubbers: natural rubber (NR), ethylene–propylene–diene (EPDM), butyl (IIR), styrene–butadiene (SBR), butadiene–acrylonitrile (NBR), polychloro-prene (CR), polybutadiene (BR), polyethylene (PE), synthetic polyisoprene (IR), chlorinated butyl (CIIR), and two classes of active filler: carbon black and silica.

In order to obtain a representative dataset, incomplete or inconsistent data was removed prior to modelling. Additionally, rare compounds were excluded, keeping only the most common ones with at least 100 measurements. The database was then divided into five separate datasets, each covering

the relation between all rheological parameters as inputs and one of the mechanical parameters as output. The size of each dataset depends on how frequently the parameter in question is assessed for quality control purposes. The hardness dataset, consisting of 18 649 input–output samples and covering sixty different compounds, was the largest in these experiments. The datasets from tensile testing, entailing eleven different compounds, consisted of 4072 input–output samples for the case of the moduli M_{50} , 4021 input–output samples for the case of the moduli M_{100} , and 4109 input–output samples for tensile strength TS and elongation at break E .

For the purpose of routine quality control, rheometric examination of elastomeric compounds also serves to resolve the appropriateness of rubber compounds for further processing. A compound is classified as appropriate for further production if all measured parameters are within a specified limit range [22]. An example of assessment of a compound for appropriateness for further production, together with specification limit ranges, is shown in Table 1.

The response of compounds with similar molecular structure to a testing procedure is expected to be similar, but on different scales. Therefore, it appears reasonable to represent each measured parameter by two values: its average for a given compound and a relative deviation, defined as the quotient of the divergence from the average and the average itself. The effect of such preprocessing on the hardness parameter for three different compounds is presented in Fig. 4. To grasp the idea of preprocessing, all samples of the same compound are plotted together, although they appear interchangeable in practice. The average values of measured parameters can be used by a model to properly identify compounds on a coarse scale. Besides, the relative deviations of parameters comprise information about discrepancies in vulcanometric parameters on input as well as mechanical parameters on output and are thus extremely important to establish a firm model on fine scale.

Each of five given datasets was prepared in its raw and pre-processed forms. In the raw form, all the datasets have rheometric parameters ML , MH , t_{s1} , t_{10} , t_{50} , t_{90} and measurement

Table 1

Measured values of parameters for a specific sample of a compound along with specification limit ranges and status of tests

Parameter	Unit	Measurement	Lower limit	Upper limit	Status
ML	dN m	2.29	1.99	2.35	Pass
MH	dN m	17.31	15.30	18.30	Pass
t_{10}	s	33.0	—	—	—
t_{50}	s	61.2	—	—	—
t_{90}	s	96.8	90.0	97.2	Pass
t_{s1}	s	28.8	—	—	—
H	Sh°A	71.0	65.9	70.1	Fail
M_{50}	MPa	1.67	1.36	1.84	Pass
M_{100}	MPa	3.13	2.55	3.45	Pass
TS	MPa	23.0	18.1	25.9	Pass
E	%	341	305	395	Pass

Since the hardness criterion (H) was not reached, the given compound is not appropriate for further production. For parameters without specification limits, the status cannot be obtained.

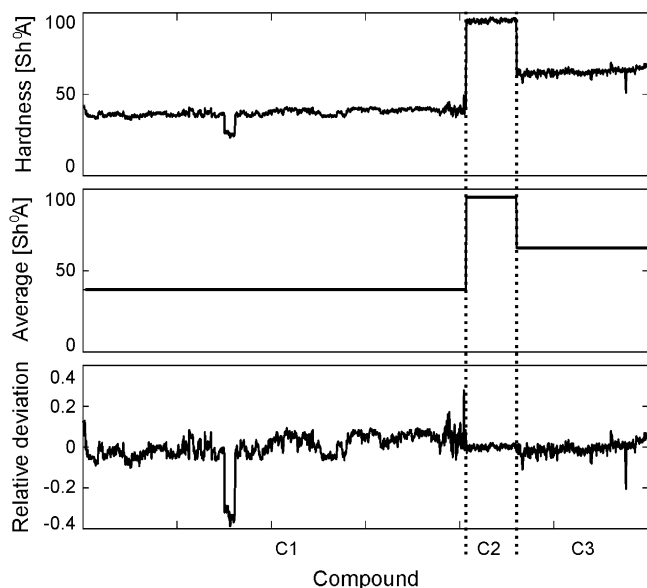


Fig. 4. Effect of preprocessing on the hardness parameter for compounds C1, C2 and C3: measured hardness (above), average over all samples of the same compound (middle) and relative deviation from the average (below).

time as inputs, and one of the observed mechanical parameters (H , $M50$, $M100$, E , TS) as output. In the preprocessed form, each rheological parameter is presented by its average and relative deviation, hence doubling the number of inputs, whereas only the relative deviation of the chosen mechanical parameter is used as output. It suffices to relate the rheological parameters only to the relative deviation, since the average value is known in advance from all previous tests.

For each dataset the input–output samples were divided into two sets: the training set, which included 80% of the input–output samples, and the testing set, with the remaining 20% of the input–output samples. The first 80% of the samples in the training set were used for training, while the last 20% of the samples were used only for validation purposes. When the error on the validation set starts to increase, the training process is stopped, preventing the model from adapting very accurately to the samples in the training set, thus retaining its generalization capabilities. Performance of the model on the testing set is used for model comparison. The proportions of input–output samples belonging to each compound were equal in both sets.

A multilayered perceptron with one hidden layer and one output neuron was used in all experiments, where the number of neurons in the hidden layer varied from three neurons to three times the number of inputs. Due to the stochastic nature

of the initial phase of the training process, the training of a model with a given topological structure was repeated 50 times, thus ensuring statistical significance of the results, on one hand, and diminishing the problem of local minima on the other.

To evaluate the models, three criteria were used: (i) the root mean square error, normalized to the standard deviation (NRMSE); (ii) the mean absolute percentage error (MAPE); and (iii) the percentage of correctly classified samples (% OK). The measured and/or predicted value of each parameter can fall into one of three categories: within, above or below the specified limit range. A given sample is correctly classified when the measurement and the prediction fall into the same category. The third criterion, the percentage of correctly classified samples, was used only in the case of hardness prediction, since only in the hardness test the specification upper and lower limits are defined for all compounds due to the quality control requirements.

4.1. Prediction results of hardness

The results of hardness prediction on raw and preprocessed datasets for the training and testing sets are given in Table 2. The values of all performance measures indicate that the model performed relatively well, i.e. was able to extract and quantize nonlinear relationships between the mechanical properties of vulcanizates and the rheometric parameters of compounds. Preprocessing plays an essential role in overall performance of the model — it reduces the NRMSE and MAPE, and increases the percentage of correctly classified samples. Considering all three criteria, it can be established that preprocessing contributed at least 15% to the improvement of the observed performance measures.

Although the number of model inputs is larger in the case of the preprocessed input data, the number of adaptive weights is smaller, indicating a less complex model. The impact of preprocessing is further illustrated in Fig. 5, showing that for the majority of compounds percentage of correctly classified samples on the preprocessed dataset are greater than 80%, which is not the case for the raw dataset.

The high percentage of correctly classified samples shows that the neural network model is capable of relating rheological parameters on one side and hardness on the other. Further analysis showed that the majority of incorrectly classified samples were relatively close to the boundary lines of the specified limit range. Moreover, preprocessing enables neural network models to find quite good relations even for compounds

Table 2
Prediction performance of hardness

Dataset	Model structure/weights	Performance measure					
		NRMSE		MAPE		% OK	
		Training	Testing	Training	Testing	Training	Testing
Raw	7-9-1/82	0.251	0.289	0.040	0.043	76.08%	72.53%
Preprocessed	14-3-1/49	0.186	0.200	0.028	0.029	86.68%	86.43%

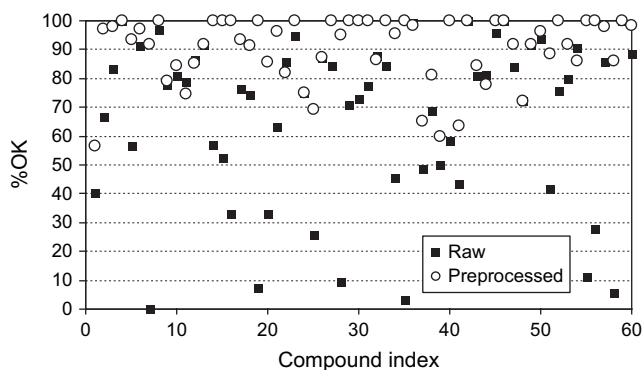


Fig. 5. Impact of preprocessing on the % OK measure for hardness prediction, given separately for each compound on testing set.

seemingly impossible to model from the raw dataset. The compounds with poor % OK performance measure were found to belong to uncommon molecular structure groups with a small number of input–output samples in the training set, thus making it very hard for the model to adapt to them. Furthermore, for many common compounds the classification rate was 100%.

The mean values of applied performance measures and corresponding 95% confidence intervals, shown in Fig. 6, were obtained from prediction results given by 50 different models with the same topological structure. The narrow confidence intervals in the case of the preprocessed dataset indicate easier modelling and more accurate prediction compared to the models based on the raw dataset.

4.2. Prediction results for tensile parameters

The prediction results for tensile parameters are summarized in Table 3. As the specification limits are not given for all compounds, only two performance measures are presented (NRMSE, MAPE).

Comparing the results with those of hardness modelling, it can be observed that the values of the performance measures are higher in the case of tensile parameters. Taking into account the number of model weights, the models of parameters *M50* and *E* are of comparable sizes to the hardness models, while the best performing models for parameters *M100* and *TS* are considerably larger, indicating far more complex dependencies. From this it may be concluded that tensile

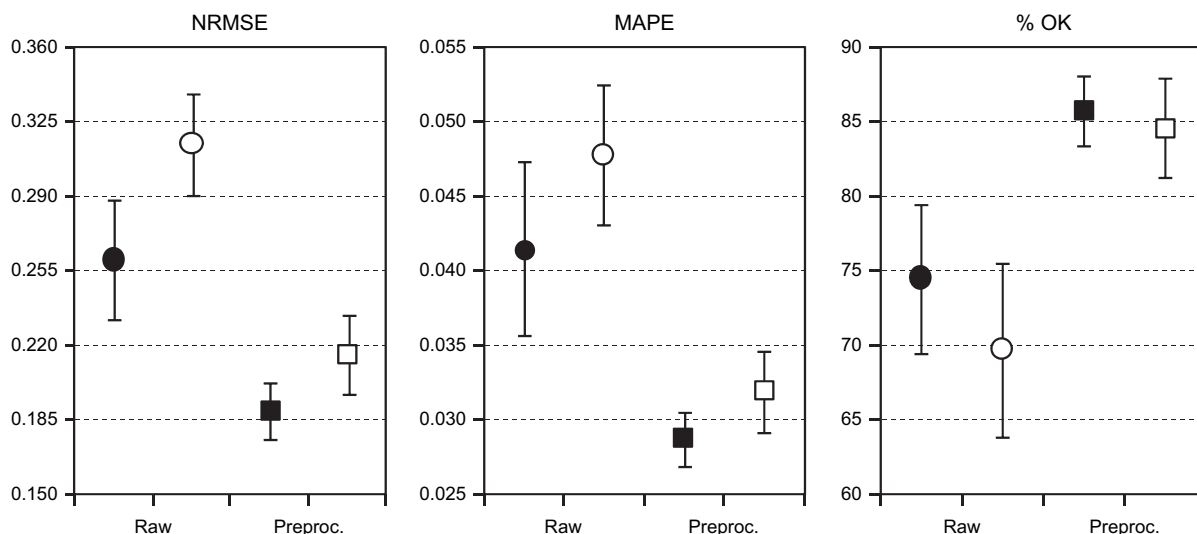


Fig. 6. Mean values and 95% confidence intervals for all three measures of hardness prediction for raw and preprocessed datasets. Black and white markers represent the average values for training and testing sets, respectively.

Table 3
Prediction performance of tensile parameters

Parameter	Dataset	Model structure/weights	Performance measure			
			NRMSE		MAPE	
			Training	Testing	Training	Testing
<i>M50</i>	Raw	7-11-1/100	0.564	0.390	0.124	0.075
	Preprocessed	14-3-1/49	0.441	0.302	0.094	0.058
<i>M100</i>	Raw	7-21-1/190	0.350	0.383	0.115	0.137
	Preprocessed	14-9-1/145	0.349	0.296	0.112	0.101
<i>TS</i>	Raw	7-19-1/172	0.229	0.258	0.137	0.188
	Preprocessed	14-13-1/209	0.234	0.222	0.124	0.134
<i>E</i>	Raw	7-15-1/136	0.725	0.365	0.095	0.094
	Preprocessed	14-3-1/49	0.721	0.345	0.091	0.085

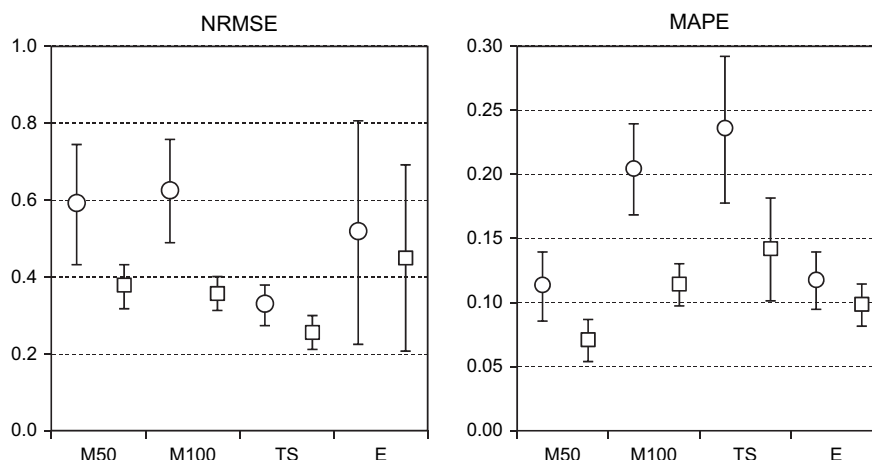


Fig. 7. Mean values and 95% confidence intervals for prediction of rheometric parameters on testing sets (circles and squares indicate results from raw and preprocessed datasets, respectively).

parameters are more difficult to model, clearly due to far more scattered values being highly dependent on the local structure of the tested materials.

Yet again, the prediction of all tensile parameters is improved by preprocessing the raw dataset. The number of model weights is smaller, with the exception of TS, where their prevalence indicates an exceedingly complex relation between TS and rheometric parameters.

Similarly as in the hardness case, the confidence intervals of tensile parameter models are also narrowed by preprocessing. Data for all four tensile parameters and the two types of datasets are given in Fig. 7.

The obtained prediction results are sufficient for the purposes of the rubber industry, since in practice measurement uncertainties due to the complex nature of the materials cannot be improved to below 5%. The achieved criteria can be especially perceived from the MAPE performance measure given in Figs. 6 and 7. The picture could be improved, however, if each compound was analyzed separately, but it would blur the aim of the work, which was to search for generality in polymeric features.

5. Conclusion

Theoretical predictions of the mechanical properties of crosslinked elastomers from the rheometric properties of elastomeric compounds are hampered by the high nonlinearities which generally arise due to multiple influences on already complex materials and, almost invariably, unequal testing conditions.

Specialized for precisely such problems, neural network predictive models in combination with data preprocessing yielded good results in an attempt to establish otherwise discouraging nonlinear relations. Special preprocessing of measured parameters was introduced in order to give representative data to the models and simplify their adaptation to the given data. Another confirmation of the successful application of neural networks in this work is the fact that the obtained results, together with

meticulous observations, are in perfect agreement with the generally known phenomena of elastomeric behaviour.

Revelation of newly instituted relations between material properties ascertained at high deformational and temperature differences contributes to the improvement of laboratory testing speed and quality, or efficiency in general. Owing to accurate classification of vulcanizate parameters from rheometric parameters, the multitude of long-duration mechanical tests on vulcanizates can be reduced to the minimum, also enabling prompt checking of the correlation of results for different characteristics of a given material, often required by quality standards of testing laboratories. Additionally, information on the suitability of testing methods can be gained by comparison of the results and the models.

To further improve the prediction and classification capabilities of the models, additional parameters should be retrieved from the MH portion of the vulcanometric curves, where the compound properties are close to those of vulcanizates used in mechanical tests. Furthermore, as molecular structure itself also plays a significant role in the behaviour of elastomeric compounds, as well as vulcanizates, the addition of this information might contribute to further improvement in prediction.

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