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Sensitivity analysis: A tool for tailoring environmentally friendly materials

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ABSTRACT

In this article, we examine the use of sensitivity analysis for the optimization of selected physical properties in rubber compounds and determine objective criteria which allow for the reduction of environmental load during rubber compound production. The sensitivity analysis shows how significantly each input value affects the output value, and the response graphs express the effect of the selected parameter on the output value. The solutions described in the article are applicable to other production technologies. We present a sensitivity analysis based on the prediction of selected mechanical properties of rubber mixtures composed of Standard Malaysian Rubber (SMR). Two blends were pre- pared by mixing SMR and oleic acid and different concentrations of surfactant (2, 4, 6, 8, 10, 20, 30 wt%). Tensile strength R_m and moduli M100, M200, M300 were measured and evaluated. The sensitivity analysis showed the significance of certain ingredients which affect the measured mechanical properties.

1. Introduction

Technological treatments and the agents or fillers added to rubber have a strong effect on several properties of rubber mixtures. Carbon black is an active filler which is used to increase electrical and thermal properties due to its strong interaction with natural rubber. Plasticizers play an important technological role in the rubber industry. Rubber is a multicomponent material which is very sensitive to many chemical species, and especially to technological treatment. For multicomponent system inputs and outputs associated with the optimization of chemistry or technological processes, it is convenient to use an artificial neural network (ANN).

ANNs are frequently applied in material analysis, and in some cases also have successfully replaced destructive testing in fiber composites (Farhana et al., 2016).

True stress/strain curves were successfully calculated for the automobile industry (Doh, Lee, & Lee, 2016), where the authors optimized interconnection weights obtained with hidden layers and output layers. A mathematical model of the material’s behavior was suggested through this feed-forward neural network.

The study Le, Yvonnet, and He (2015) applied a finite element method and an ANN to describe surface stress in materials.

A combination of an ANN and FEM was applied in a study of the dynamic properties in glass laminates with different material shaping (Seidl et al., 2011). Shape modes were presented for different vibrational excitations.

ANNs were used with success to optimize the composition of optical glasses (Seidl et al., 2011) and obtain required optical properties.

Feed forward neural networks are used for the classification of layers of coal and shale coal according to ash and moisture content (Jančíková, Bošák, Zimný, Legouera, Minárik, Koštial, & Poulain, 2014).

The work Ghosh, Chatterjee, and Shanker (2016) applied an ANN and neuro-fuzzy interference system which separated permeate flux and salt content.

A multi resolution analysis of time-dependent data sets obtained through Wavelet de- composition and evaluated using an ANN was applied to the management of renewable energy sources in the study Salehi and Razavi (2016).

A sound statistical model analysis was developed based on deep neural networks. Using a new algorithm on conventional data, the authors attained substantially better results (Doucoure, Agbossou, & Cardenas, 2016).

A discoloration process analysis was conducted with the application of three intermediate layers, a backpropagation learning algorithm, and a sigmoid activation function implemented in Fortran. Three neurons in the intermediate layer provided the best results Hwang, Park, and Chang (2016).

Technological treatments and the agents or fillers added to rubber

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Table 1
Chemical composition of standard rubber mixture.

Ingredient	PHR content
SMR	100
Sulphur	2
ZnO	5
Stearine	2
Sulfenax	2
N339 CB	50
Gumodex	10

have a strong effect on several properties of rubber mixtures. The main additives and their functions were described in Lenzi et al. (2016).

The paper Jonšta et al. (2011) examines the effect of nano-styrene butadiene rubber in phenolic nanocomposites. When applied in the amount of only several percent, these nanoparticles substantially increase the notched impact strength without a significant change in flexural performance.

The application of ultrasound during the extrusion process of different rubber types showed changes due to die pressure (Liu, Ma, Zhan, & Wang, 2015). Increasing the ultrasound amplitude decreased the die pressure for both natural rubber and mixtures of natural and styrene butadiene rubber. In the case of styrene-butadiene rubber, the die pressure increased.

In (Choi & Isayev, 2015), the authors presented liquid isoprene as a replacement for oil plasticizer. In this case, liquid isoprene reduced both Mooney and apparent viscosity and suppressed plasticizer migration.

The work (Ren, Zhao, Li, Zhang, & Zhang, 2015) presented an improvement in the mechanical properties of silica-filled rubber caused by a ring opening between rubber chains and Si-OH groups. Ring opening processes occurred during both the vulcanization and mixing processes.

Polyethylene-co-vinyl acetate, natural rubber and organoclay mixtures were studied in (Xu, Jia, Luo, Jia, & Peng, 2015). The addition of organoclay suppressed the natural rubber amount and raised Young's modulus and yield stress. Elongation at break and stress at break were decreased.

The paper (Razavi-Nouri & Karami, 2014) studied the percolation effect and further electric transport phenomena caused by the presence of graphite.

Mansour, Hussein, and Moharram (2014) described the influence of fillers on natural rubber properties. The results for different fillers (including carbon nanotubes) showed an increase in viscosity cross-link density, modulus and hardness and a decrease in cure time. The plasticizers in a rubber blend increased their processing properties. Nevertheless, they often migrated from the rubber matrix and caused poor stability in the rubber blend in the long term. The adverse environmental impact of this process was also substantial.

Kopal et al. (2022) have predicted curing characteristics which play a significant role in the vulcanization process of rubber blends using ANN. Although some studies, e.g. (Lubura et al., 2021), (Safar et al., 2019), (Vodka & Pogrebnyak et al., 2020) and (Lopes, Silva, & Machado, 2021), have used ANN networks for the prediction of different processes in rubber technology, they focused on the prediction of different parameters but none of them take into account the environmental aspect of rubber technology. As the production of rubber compounds also has its environmental impact, it is necessary to focus on the optimization of chemical compounds.

The aim of this paper is to evaluate the influence of existing chemicals on the mechanical properties of the mixture in order to reduce environmental effects in the production process of rubber compounds, where the environmental aspect is the main contribution of this research. In the research, we deal with the effect of plasticizer and the amount of surfactant on certain mechanical properties such as strength R_m and moduli M100, M200, M300. The analysis method we applied was based on our previous experimental work (Ružiak et al., 2018).

The article is structured as follows: Section 2 summarizes the theory of artificial neural networks. Section 3 describes the experimental conditions. Section 4 gives a detailed overview of results, and Section 5 lists the conclusions arising from the research.

2. Theory

Artificial neural networks are used to predict material properties when an analytical mathematical approximation cannot be found. Using this very robust mathematical tool, material properties can be predicted.

Artificial neural networks use different topologies which, in most cases, contain three or four layers. Three types of neurons occur in these networks:

- input neurons
- hidden neurons
- output neurons.

Input neurons contain information about the parameters which change in our datasheet, such as material composition, thermal treatment, and others. Output neurons correspond to the material properties we want to predict. Some of the input neurons are used for training and others are used for generalizing.

The advantage of a neural network is that it can both learn and generalize. The main disadvantage of a neural network is that it needs more values for one or more parameters which change in each input dataset than required by standard fitting procedures such as the least squares and other methods.

The use of an ANN is tested with the statistical parameters REL_RMSE and R^2, which are defined by Eqs. (1) and (2).

$$RMSE = \sqrt{\frac{\sum_{i=1}^{n}(y_i - o_i)^2}{n.(n-1)}} \tag{1}$$

$$R^2 = \left(\frac{E(y).E(o)}{E(y.o)}\right)^2 \tag{2}$$

where:

y_i – measured values o_i – predicted values.
n – number of input values.
E (y) – statistical mean value of inputs.
E (o) – statistical mean value of predicted values.
E (y.o) – mean value of multiplication of predicted and input values.

3. Experimental conditions

3.1. Materials

Plasticizers play an important role in rubber technology and have an effect on the blend mixing. We therefore present the effect of the amount of plasticizer on selected mechanical properties such as strength $R_{m,}$ modulus M100, M200 and M300. The experimental values were measured on the apparatus Z250 AllroundLine (250 kN). The tensile strength R_m and moduli M100, M200, M300 measurements were performed on this device by tensile test.

The chemical composition of standard rubber mixture in PHR (parts per hundred of rubber) is shown in Table 1. The rubber was SMR (Standard Malaysian Rubber).

Sulfenax is used in rubber industry in processing of natural and synthetic rubber in rubber compounds as a fast accelerator of vulcanization with delayed action. It provides good physical and mechanical properties, high crosslinking efficiency and good modulus. N339 CB is a type of carbon black (CB) and Gumodex is a plasticizer.

The general procedure for the rubber vulcanizates preparation is as follows and is protected by the manufacturer. In the two-stage mixing, which we used in our case, the mixture is prepared in the first phase at a

Table 2
Number of hidden units.

Type of network	Minimum hidden inputs	Maximum hidden inputs
Radial basis function RBF	1	8
Three-layer MLP, layer 2	1	16
Four-layer MLP, layer 2	1	16
Four-layer MLP, layer 3	1	16

Table 3
Best neural networks.

Property	Details of network
R_m [MPa]	MLP 2:2–16-6:6
M100 [MPa]	MLP 2:2–16-6:6
M200 [MPa]	MLP 2:2–16-16–6:6
M300 [MPa]	MLP 2:2–16-16–6:6

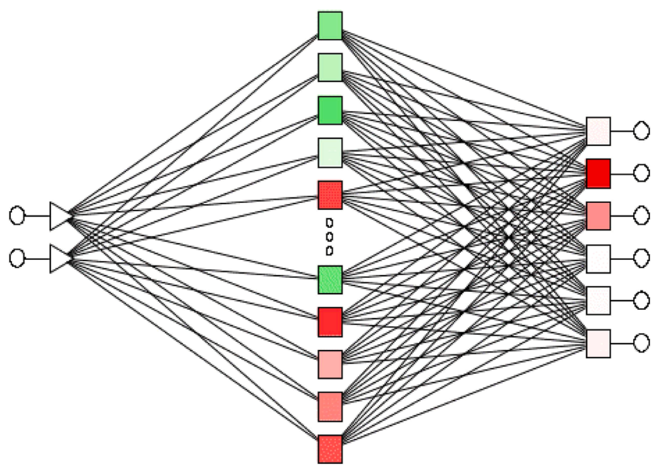

Fig. 1. MLP2:2-16-6:6 is best network for the prediction of tensile strength R_m and modulus M100.

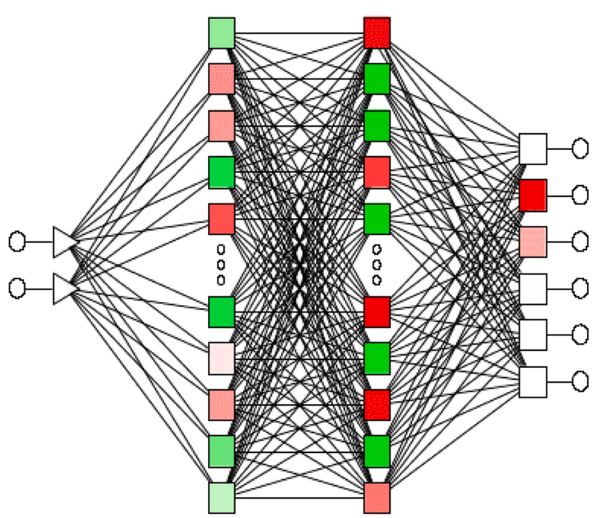

Fig. 2. MLP2:2-16-16-6:6 id the best network for moduli M200 and M300.

temperature of about 150° C. The pressure exerted by the kneading wedge is about 3 MPa and the mixing time is 4 to 4.5 min. These parameters depend on the type of mixture. The thickness of the belt is determined by the gap between the rollers. To prevent the mixture from sticking, it is immersed in a wetting bath containing a separation solution. The temperature of the effluent mixture is a maximum of 36° C.

In the second stage of mixing the mixture (final mixture), the mixture is weighed again in the form of a chopped honeycomb. It is first processed in a mixer, and when the required plasticity is reached, the vulcanizing agent and other substances are added. The temperature of the mixing chamber is lower than in the previous mixing (about 110° C) and the mixing takes a very short time.

The reference samples were mixed with oleic acid plasticizer with a content of 1 PHR and 3 PHR, together with weight percentage of surfactant (0, 2, 4, 6, 8, 10, 20, 30 wt%). The surfactant was sodium 1-

Table 4
Slope between the predicted and measured characteristics, percentage deviance, correlation coefficient R and mean squared error RMSE.

Property	Slope	Percentage difference (%)	R	RMSE (MPa)
R_m [MPa]	1,048	+4,8	0,982	0,082
M100 [MPa]	0,983	−1,7	0,985	0,028
M200 [MPa]	0,999	−0,1	0,978	0,068
M300 [MPa]	0,938	−6,2	0,981	0,095

Table 5
Sensitivity coefficients of the input parameters for all the studied mechanical characteristics.

Property/Sensitivity coefficient	Surfactant wt %	Oleic acid amount
R_m [MPa]	2.491	1.163
M100 [MPa]	2.632	1.159
M200 [MPa]	2.931	1.203
M300 [MPa]	2.514	1.073

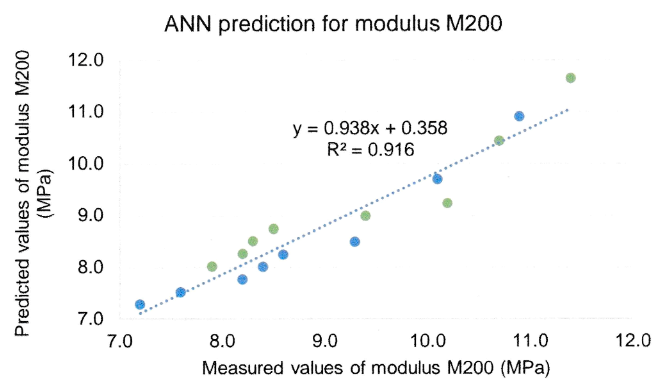

Fig. 3. Estimated R_m values versus the real measured values in the subgroup of 16 values.

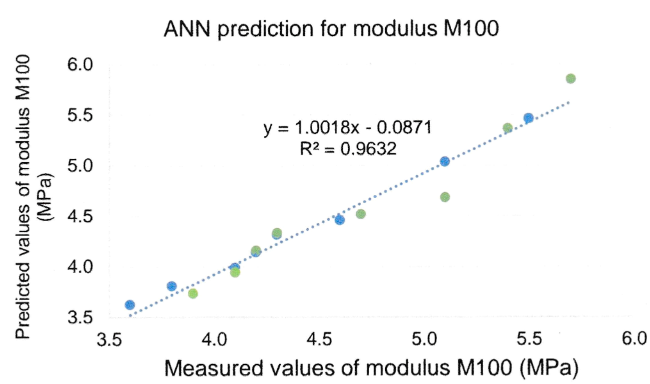

Fig. 4. Estimated M100 values versus the real measured values in the subgroup of 16 values.

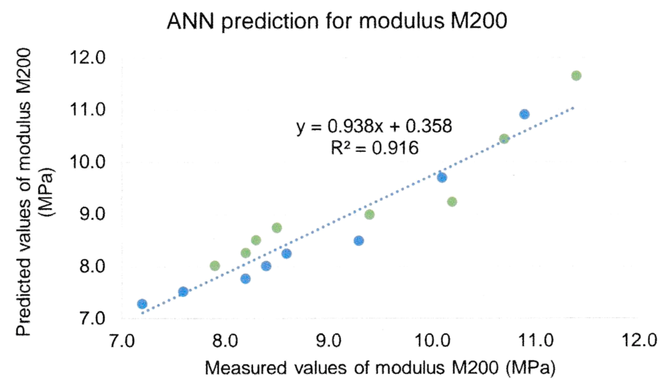

Fig. 5. Estimated M200 values versus the real measured values in the subgroup of 16 values.

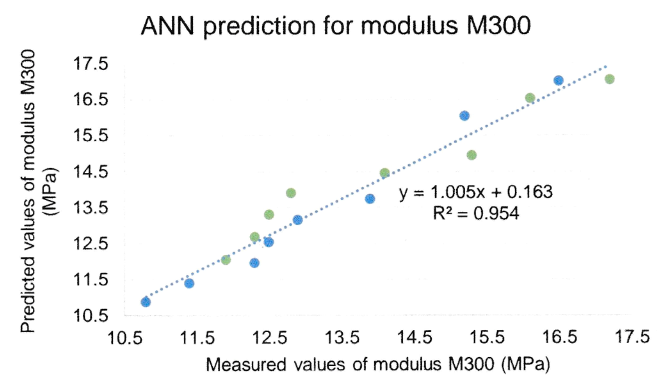

Fig. 6. Estimated M300 values versus the real measured values in the subgroup of 16 values.

nonyl-2-(2-nonylphenoxy benzene sulfate). These amphiphilic substances reduce the surface tension of the fluids and the interfacial tension between two liquids. The use of oleic acid plasticizer as a substitute for gumodex has a lower ecological impact.

3.2. Sensitivity analysis

The sensitivity analysis method applied in the study allows for a statistical analysis of the effect of technological processes on the mechanical properties of the rubber blend (in this case). The response graphs, which are additional results from the ANN procedures, indicate the values of the studied properties in combinations of input parameters which were not measured.

Neural networks were created in STATISTICA – Neural Networks software. Neural network software enables the execution of a sensitivity analysis and the creation of response graphs. The sensitivity analysis reveals how significantly each input value affects the output value, and the response graphs express the effect of the selected parameter on the output value.

A neural network conducts a sensitivity analysis by treating each input variable in turn as if it were "unavailable". Each model defines a missing value substitution procedure to allow predictions in the absence of values for one or more inputs. To define the sensitivity of a particular variable, we first run the network on a set of test cases and accumulate the network error. We then run the network again using the same cases, but this time replace the observed values with the value estimated by the missing value procedure, and again the network error accumulates.

Given that we effectively remove some information which the network presumably uses (i.e., one of its input variables), we would reasonably expect some deterioration in error to occur. The basic measure of sensitivity is the ratio of the error with the missing value substitution and the original error. The more sensitive the network is to a particular input, the greater the deterioration we can expect and therefore the greater the ratio. If the ratio is one or less, then making the variable "unavailable" either has no effect on the performance of the network or enhances its performance. This means that the studied property is not a function of the "missing" input parameter.

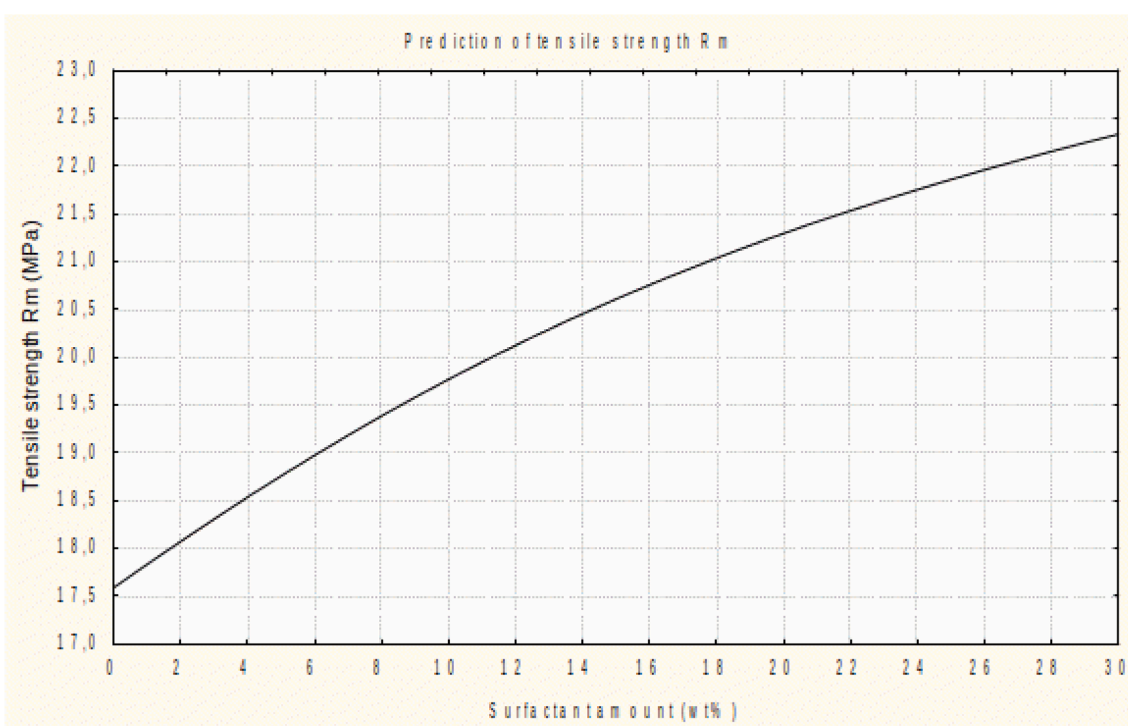

Fig. 7. Prediction of tensile strength R_m versus surfactant amount.

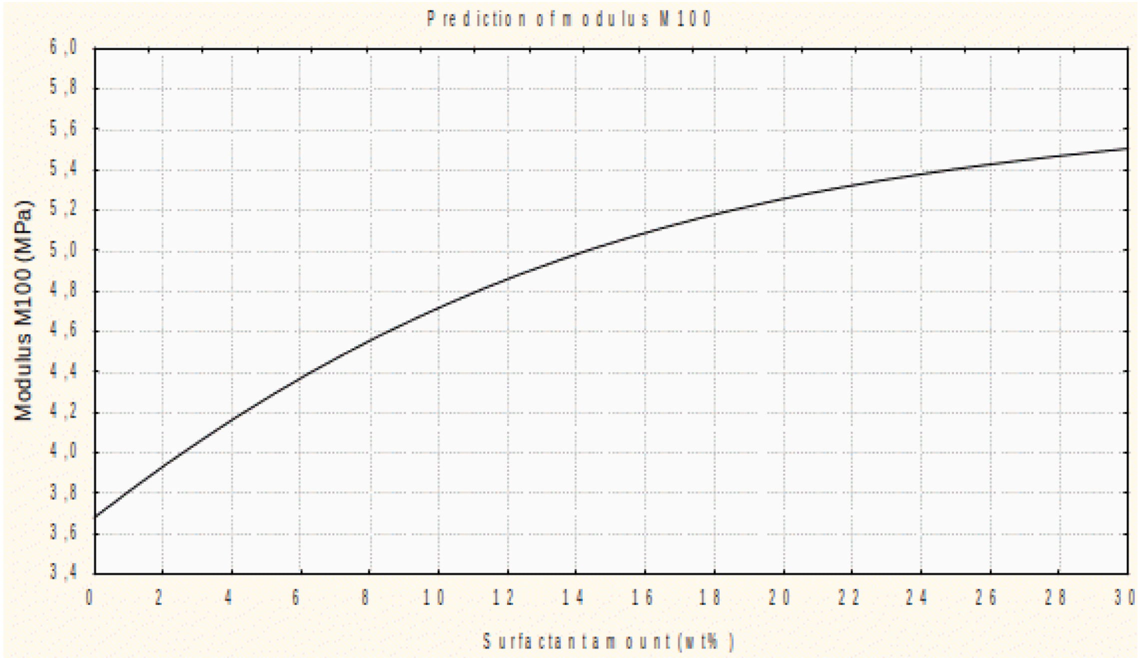

Fig. 8. Prediction of modulus M100 versus surfactant amount.

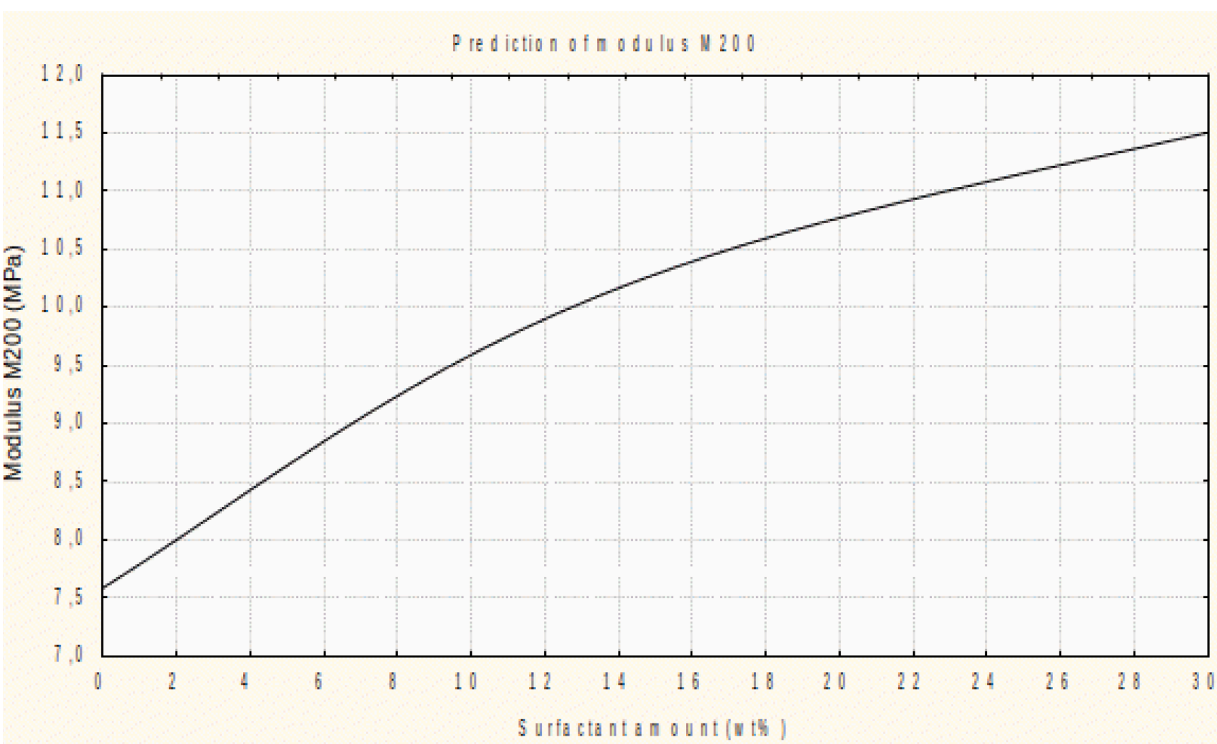

Fig. 9. Prediction of modulus M200 versus surfactant amount.

For the neural network input parameters, we used the surfactant amount as a continuous neuron input and the oleic acid amount as a categorical neuron input. We selected oleic acid as a categorical input because the studied materials only contain two different values for this input parameter, which is a very low level for learning and generalization. The measured values of the mechanical properties were used as continuous neuron outputs. Since the concentrations of the surfactant equaled 0, 2, 4, 6, 8, 10, 20, and 30 wt% and the two concentrations of oleic acid were 1 PHR and 3 PHR, the neural networks learned with 16 input neurons.

The total analyzed neural networks for each variable and each state consisted of ten neural networks, from which we used the best five neural networks. The selection criterion to retain a network was the lowest error. The types of network which we applied were linear, PNN or GRNN, Radial Basis function, three layer and four-layer perceptron.

The minimum and maximum values for the hidden inputs of each

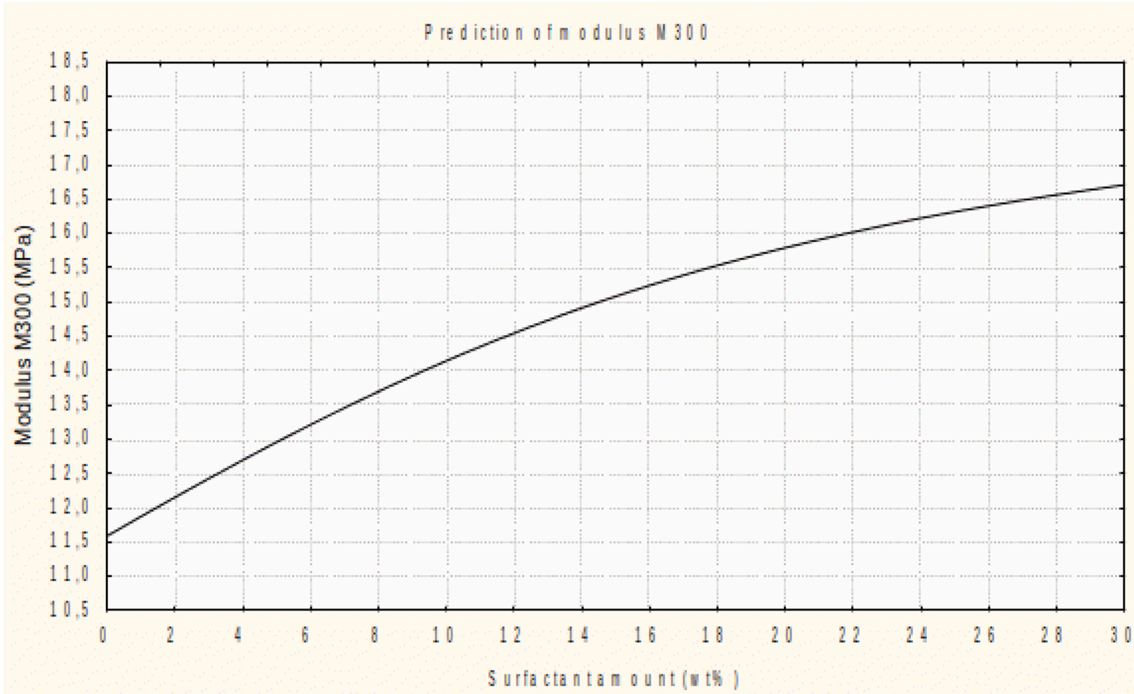

Fig. 10. Prediction of modulus M300 versus surfactant amount.

Table 6
Total share of the share of the surfactant and the volume of oleic acid of the studied mechanical characteristics.

Property/Sensitivity coefficient	Surfactant wt %	Oleic acid amount
R_m [MPa]	68.2 %	31.8 %
M100 [MPa]	69.4 %	30.6 %
M200 [MPa]	70.9 %	29.1 %
M300 [MPa]	70.1 %	29.9 %

network are shown in Table 2.

4. Results

We present details of the improved real time networks.

In the final step, we computed the predictions for all five best neural networks and com- pared them to the measured values in the teaching subset. As the best neural network, we used a neural network in which the correlation between the measured and ANN ap- proximated network were closest to 1 by sustaining the root mean square error between the measured and approximated values closest to zero. The best neural networks for each variable are listed in Table 3.

where MLP refers to the multilayer perceptron network.

Figs. 1 and 2 present the best network for moduli 100, 200 and 300.

Table 4 presents the slope values between the predicted values of the studied mechanical characteristics and the real measured values. The table also presents deviances of this slope from the value of 1 (it characterizes the deviance) and the correlation coefficient, together with the mean squared error RMS.

Based on the results presented in Table 4, we can draw the following conclusions:

The maximal size of the error in determining the mechanical characteristics by neuron networks in a 16-member base is 6.2% (for the M300 characteristic), which is below the measuring error limit of the stated mechanical characteristics of rubber compounds.

Since the error in determining mechanical characteristics of neural networks is smaller than the measuring error, we can state that ANN are able to predict tensile strength and strength of the M100, M200, M300 samples before ageing.

The stated conclusion is also supported by high values of the correlation coefficient – at least 0.978.

The maximal mean squared error between the predicted and measured values of the mechanical characteristics is 0.095 MPa, which is at the measurability limit of the given characteristics.

More useful parameter for assessing the differences between the predicted and measured values of mechanical characteristics is the relative mean squared error, defined as a quotient of the mean squared error and the minimal value of the given.

This parameter amounts to 0.005; 0.008; 0.009 and 0.009 for the tensile strength and the M100, M200 and M300 moduli, which correspond to the percentage deviation of 0.5% for the tensile strength, 0.8% for the M100 modulus and 0.9% for the M200 and M300 moduli.

- These values represent the maximal deviance between the predicted and measured char-acteristics, with the stipulation that they are at least 10 times smaller than the uncertainties in determining the studied mechanical characteristics.
- The theoretical error in determining the mechanical properties of rubber compounds based on at least 10 test specimens is approximately equal to 5%. The experimentally detected errors in the determination of Rm, M100, M200 and M300 for all measured sample types and all properties do not exceed the error value of 5%.

Based on diagrams 3 to 6, we can draw the following conclusions:

All the studied material properties increased along with a rise in the amount of oleic acid and surfactant.

- Tensile strength increased approx. 29% for 1 PHR and 22% for 3 PHR.
- Modulus M100 increased approx. 53% for 1 PHR and 46% for 3PHR.
- Modulus M200 increases approx. 51% for 1 PHR and 44% for 3PHR.
- Modulus M300 increases approx. 53% for 1 PHR and 45% for 3PHR.

- All these increases are highly above the property measurement uncertainty; therefore, the surfactant weight percentage has a significant role in the tensile strength and moduli.
- The differences between 1 PHR and 3 PHR in increase were 7%, 7%, 7%, and 8%, which are all below the measurement errors for these types of rubber blends, and we can therefore conclude that the effect of surfactant weight percentage is approximately the same for 1 PHR and 3 PHR mixtures, which is also supported by a very high correlation coefficient.
- between the predictions and measurements for both 1 PHR and 3 PHR mixtures.
- The increase in R_m was probably caused by a higher degree of structure cross- linking Skrobak et al. (2016).

In the following results, we show predictions of the studied properties. Because of same increase in the studied mechanical properties for 1 PHR and 3 PHR oleic acid mixtures, we show the prediction of the average values of property versus surfactant amount.

For each neural model response, graphs were created. The response graphs express the influence of the chosen input parameter on the predicted output parameter – see Figs. 7-10. These graphs express the influence of the surfactant amount on the predicted mechanical properties. Fig. 7 shows the prediction of tensile strength, while Figs. 8-10 show the prediction results for modulus M100, M200 and M300 respectively.

Based on Figs. 7-10, we can draw the following conclusions:

- The biggest change in the studied mechanical characteristics with a change in the share of the surfactant can be observed within the weight percentage range of 0 to 10%; the effect of the surfactant is reduced over 10%.
- An increase in the values of the studied mechanical characteristics of up to 10% repre- sents 12%; 27%; 26% and 22%, while the increase of up to 30% in the weight percentage of the surfactant only corresponds to increases of 25.5%; 49.5%; 47.5% and 49%.
- An increase in the share of the surfactant by 1/3 of the overall increase leads to a 48% increase of the total increase of the tensile strength R_m.
- An increase in the share of the surfactant by 1/3 of the overall increase leads to a 55% increase in the total increase of the M100 modulus.
- An increase in the share of the surfactant by 1/3 of the overall increase leads to a 55% increase in the total increase of the M200 modulus.
- An increase in the share of the surfactant by 1/3 of the overall increase leads to a 45% increase in the total increase of the M300 modulus.
- On the other hand, an increase in the surfactant by 2/3 of the overall increase leads to an increase in the tensile strength and of the M100, M200 and M300 moduli by 82.5%; 85%; 87.5% and 91%.
- Such an increase of a 20% share of the surfactant is sufficient for improving the studied mechanical characteristics and, at the same time, reducing the share of the surfactant has a positive impact on the environmental burden of the final mixture over time since the final mixture as well as surfactant age due to the impact of heat and time.

Below, we discuss the sensitivity analysis obtained from the prediction using the ANN networks. Table 5 shows the sensitivity coefficients of the input parameters for all the studied mechanical characteristics.

Based on Table 5, we can state that:

- The increase in the error by omitting the share of the oleic acid (in the way the sensitivity coefficients of neural networks are calculated) amounts to 16%, 16%, 20% and 7% for R_m, M100, M200 and M300, which is close to the measuring error, i.e., the change in the mechanical characteristics with a change in the share of the surfactant can be described by a single function for both concentrations of the oleic acid, which can be also deduced from Figs. 3-6.
- On the other hand, the same operation for the share of the surfactant leads to, at least, a 149% increase in the mechanical characteristics, which means that the surfactant has a significantly greater effect on the mechanical characteristics.

In Table 6 we calculated the overall sensitivity coefficients for both input parameters and all studied mechanical characteristics.

It is clear based on Table 6 that the share of the surfactant has approximately a 70% effect on the studied mechanical characteristics.

Oleic acid has a negative environmental impact especially during the ageing process. Based on the results presented in the table, we can thus recommend a reduction in the share of the softener with a simultaneous increase in the share of the surfactant for better environmental mixtures with the same mechanical characteristics.

5. Conclusion

Based on the results of the prediction of the mechanical characteristics of rubber com- pounds with various volume shares of oleic acid and various weight shares of the surfactant, using a statistical set of 16 samples, we can draw the following conclusions:

- ANN networks are suitable tools for the prediction of tensile strength and modulus for rubber mixtures containing oleic acid, with a correlation of at least 0.98 and REL_RMSE maximum 0.8 %.
- The dependence of mechanical properties versus surfactant amount can be described by a single function for 1 PHR and 3 PHR mixtures.
- ANN networks can also predict the mechanical properties of non-measured concentra- tions of surfactant.
- From the prediction of mechanical properties, we can conclude that the level of sur- factant amount at 20 wt% increases the mechanical properties to at least 80 % of the level of increase to 30 wt% of surfactant, and therefore with the aid of ANNs, we can lower the environmental impact of the mixtures, especially as the material ages.
- From the sensitivity analysis, we can conclude that truncating the changes in the oleic acid amount leads only to a maximum 20 % increase in error, therefore we can conclude that the oleic acid amount in the region of 1 PHR to 3 PHR does not have a significant effect on the studied mechanical properties.
- Truncating the changes of surfactant amount, however, leads to an increase in error of at least 150 %, therefore we can conclude that the surfactant amounts between 3 wt% and 30 wt% have a very large effect on the mechanical properties.
- Normalizing the sensitivity coefficients leads to a 70 % effect on mechanical properties by the surfactant amount and only a 30 % effect by the oleic acid.
- The lesser effect of oleic acid is because oleic acid is mainly used in rubber mixtures to improve the processing of mixtures which are not created for any specific mechanical properties.
- Surfactants, however, bond with fillers, and therefore the amount of surfactant has a significant effect on mechanical properties.
- The submitted study demonstrates the possibilities of how to optimize the material composition of products for material tailoring with an accent on the environmental burden.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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