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Measurement and Modelling of the Rubber Resilience based on Ultrasonic Non-destructive Testing in Tires

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ABSTRACT: In tire industry, it is very crucial to evaluate physical and mechanical properties of rubbers which are used for production of tires, to ensure the quality of the final product. Resilience is an important property of a rubber, which cannot be evaluated through direct measurement in production cycle in this industry. Therefore, non-destructive ultrasonic testing, which has been used in many applications for examination of various material properties, can be used as an alternative approach for this purposes. In this study, the non-destructive ultrasonic testing method has been employed to investigate the resilience of nanoclay reinforced rubber compounds. By changing physical and mechanical properties of materials, ultrasonic wave velocities are changed. For this purpose, sixteen different samples of nanoclay reinforced rubber compounds with different formulations were prepared and both their resilience and the longitudinal ultrasonic wave velocity through them were measured. In the next step, using the relevance vector machine regression analysis, a mathematical expression for the rubber resilience based on the longitudinal ultrasonic wave velocity was developed, which was proven to be qualified with acceptable accuracy and generalization capability. The results of this research can be used for online evaluation of the rubber resilience in tire production cycle.

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1- Introduction

Tire is one of the most important products in automobile industry, which plays an important role in passengers' safety and comfort. Hence, it is greatly important to achieve the desired physical and mechanical properties based on available standards. To this end, several surveys have been conducted on the formulation of the mixture used for production of the tire which can improve its quality, such as presence of silica and absence of silane in the mixture [1] or using waste tires [2]. Given the fact that various factors influence the physical and mechanical properties of the mixture and enhancement of some properties may lead to degradation of some other features, the choice of a specific formulation for this mixture does not necessarily result in the desired properties and at the end of its production, it is necessary to measure its properties. One of the important dynamic properties of a tire is its resilience, and achieving a desired value of resilience along with other properties is of great importance. As the rubber resilience cannot be measured online in the production cycle, non-destructive testing investigating ultrasonic waves' velocities provides an alternative approach for this purpose.

In ultrasonic testing, as a non-destructive testing method, properties of a material are measured by calculating the time between sending ultrasonic waves and receiving it [3]. In this approach, ultrasonic waves and their reflections are displayed on the monitors and useful information is obtained by processing these signals, as shown in Fig. 1.

Ultrasonic wave's velocity changes and the changes in pulse height have been used to study the physical and mechanical properties of a wide range of materials. Vasconcelos et al. [4] evaluated the suitability of ultrasonic emission velocity method for investigation of physical and mechanical properties of granite. The mechanical properties, including resistance, compressive and elasticity modulus, and physical properties, including density and porosity,are obtained by measuring the propagation speed of longitudinal ultrasonic waves. The statistical correlation between the ultrasonic emission velocity, the mechanical and physical properties of the granite, and the factors that caused changes in ultrasound velocity revealed that the ultrasonic wave propagation velocity can be effectively used as a simple, cost effective and non-destructive method for initial prediction of mechanical and physical properties as well as assessment of the climate variations occurred in the lifetime of granite. In

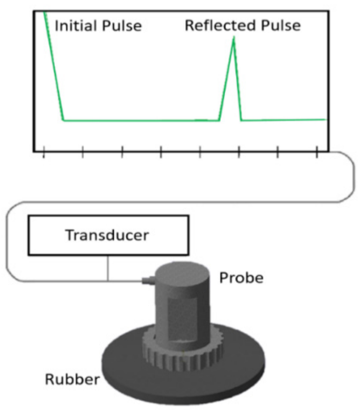

Fig. 1. Schematic of ultrasonic testing system

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addition, using statistical methods, they showed that there is a significant relationship between ultrasonic emission velocity and mechanical properties, i.e. tensile strength, compressive strength, elastic modulus and failure are significantly related.
A few numbers of surveys have also been reported for evaluation of the properties of rubbers by ultrasonic testing. In this field, Afifi et al. [5] have investigated the propagation velocity of longitudinal and transverse ultrasonic waves in Epoxidized Natural Rubber-Ethylene Propylene Diene Monomer with weight percentage of 0%, 25%, 50%, 75% and 100% of ethylene propylene rubber by ultrasonic waves at a frequency of 2 MHz and at room temperature using the ultrasonic wave reflection method. The mechanical properties of the rubber compound were evaluated, including density, longitudinal modulus, shear modulus, Young modulus, Poisson ratio and hardness. The results show that by increasing the weight percentage of ethylene propylene rubber to 75%, the velocity of ultrasonic waves increases but decreases from 75% to 100%, which indicates a stronger inter-molecular interaction in rubber compound components of epoxy resin up to 75% by weight of ethylene propylene rubber. According to their survey, appropriate mechanical properties are achieved in the range of 0% to 75% of the ethylene propylene rubber.
In another research conducted by El-Hadak [6], ultrasonic waves were used to determine the homogeneity of rubber plates. To achieve this goal, El-Hadak used a pulse-echo method and reported that rubber sheets with a percentage change of ±2.5% of longitudinal waves speed were homogeneous. In another survey in this field, Hourston and Hughes [7] concluded that the adaptability of polymer mixtures can be determined through measurement of the velocity of ultrasonic waves. The main advantage of RVM-based modeling over the previous works is the acceptable accuracy and generalization capability of this approach although the low number of measurements obtained based on the experiments and for the first time the ultrasonic non-destructive testing used for estimation of the rubber's resilience in rubber compounds.

2- Experimental details

2- 1- Materials

Generally, tires are reinforced by short fibers, carbon black, and silicates. For example, by adding 10% of the silicate as a filler to the natural rubber composition, there is a significant increase in its elastic modulus [8]. However, without considering any exception such as carbon black, the reinforcing effect of the filler materials is limited by the size of the particles and the agglomeration possibility.
Carbon black is widely used in rubber industry as the most important reinforcement factor. About 90% of global Carbon black production is used in tire industry. The carbon-smelted particles with suitable particle size and structure give better properties to rubber compounds, which is due to the nanoscale size of Carbon black particles. The significant properties of elastomers, despite the nanoclay's reinforcement, are attributed to the nanoscale dispersion of the silicate layers in the continuous rubber phase and the strong interactions between nanoparticles and polymer chains. A brief discussion about the relation between mechanical properties of polyamide-layered silicate nanocomposites and their structure can be found in [9]. Besides the structure, the melt conditions, namely factors such as mixing time and cutting severity, are found to play a crucial role in determining the distribution of clay particles in a polymer matrix and therefore their properties [10].
As a result of the high molecular weight of the tires, they exhibit a very high viscosity in the melt mixing state and thus significant shear forces can be produced during mixing, which can break the structure of the silicate plates and separate the layers from each other. Therefore, the melt mixing method is a suitable method for the preparation of nanocomposites based on rubber matrix [11]. However, if you use a melt mixing method for achieving the appropriate microstructure, it is essential to use the adapter.
In this study, sixteen different rubber formulations were used to investigate the relationship between Nanoclay reinforced rubber resilience and longitudinal ultrasonic emission velocity. The formulations were based on natural rubber, styrenine butadiene rubber and cescobutadiene filled with Carbon black. The organic clay was added to the formula, resulting in the following three modifications in formulation factors:

- Changing the amount of sulfur from 1.6 to 2.1 parts by weight to 100 parts by weight of rubber (phr),
- changing the amount of oil from 4.64 to 11.36 parts by weight to 100 parts by weight of rubber (phr),
- changing the ratio of the natural rubber to the rubberized butadiene system.

The final formulations are listed in Table 1. Preparation of the compounds was carried out in two stages. In the first stage, rubber, Carbon black, oil and chemicals were prepared except for cooking factors in an internal mixer (Banbury) at a capacity of 2 liters under identical mixing conditions. In the next step, the final blend was prepared by adding the cooking factors to the mixture on the rolls of two rollers. Samples were baked in a special form in a baking dish for 15 minutes at 151°C for 30 minutes. To test the resilience of three small samples, the standard punch and resiliency tests were used. The reported results are the mean value of three resiliency measurement tests.

2- 2- Resilience Test

Resiliency is the elastomer's ability to store and recover energy when exposed to rapid deformation. The resilience of a mixture is defined as a measure of elasticity and energy storage capacity of that mixture, therefore, a more elastic mixture benefits from a higher resilience and lower energy dissipation [12]. As a matter of fact, elasticity of an elastomer strongly depends on density of the transverse bonds in a way that as the transverse bonds decrease, elastic properties of the compounds also decrease [13].
In this study, the resin content of the compounds after baking was calculated as the resilience percentage according to the following equation [14].

$$R(\%) = \frac{1-\cos\theta_2}{1-\cos\theta_1} \times 100 \tag{1}$$

In the above equation, θ_1 is the angle of change of the pendulum of the thermistor device (45 °) and θ_2 is the angle of return.

2- 3- Ultrasonic Test

Ultrasonic waves always lose some part of their own energy propagating through a material because of the dispersion of microscopic interfaces as well as the effect of internal

Table 1. Weight of materials used in final formulations of sample rubbers

No.	Sulphor (g)	CBS (g)	Other Chemicals (g)	Oil (g)	NanoClay (g)	Soot N330 (g)	BR CIS (g)	SBR1500 (g)	SMR20 (g)	Total weight (g)
1	1.85	0.86	10.57	8.00	5.00	40.08	10.50	40.08	49.42	166.36
2	1.85	0.86	10.57	4.64	5.00	40.08	10.50	40.08	49.42	163.00
3	1.85	0.86	10.57	8.00	5.00	40.08	4.61	40.08	55.32	166.37
4	1.70	0.86	10.57	6.00	5.00	40.08	14.00	40.08	45.92	164.21
5	1.85	0.86	10.57	8.00	5.00	40.08	16.39	40.08	43.52	166.35
6	2.10	0.86	10.57	8.00	5.00	40.08	10.50	40.08	49.42	166.61
7	1.85	0.86	10.57	11.36	5.00	40.08	10.50	40.08	49.42	169.72
8	2.00	0.86	10.57	6.00	5.00	40.08	14.00	40.08	45.92	164.51
9	1.85	0.86	10.57	8.00	5.00	40.08	10.50	40.08	49.42	166.36
10	2.00	0.86	10.57	6.00	5.00	40.08	7.00	40.08	52.92	164.51
11	1.60	0.86	10.57	8.00	5.00	40.08	10.50	40.08	49.42	166.10
12	2.00	0.86	10.57	10.00	5.00	40.08	14.00	40.08	45.92	168.51
13	1.85	0.86	10.57	8.00	5.00	40.08	10.50	40.08	49.42	166.36
14	1.70	0.86	10.57	10.00	5.00	40.08	7.00	40.08	52.92	168.21
15	1.70	0.86	10.57	6.00	5.00	40.08	7.00	40.08	52.92	164.21
16	1.85	0.86	10.57	8.00	5.00	40.08	10.50	40.08	49.42	166.36

Table 2. Specifications of the device, samples and probe

Specification	Value
Manufacturer	tru-sonic
Probe diameter (mm)	12
Probe frequency (MHz)	4
Sample diameter (mm)	41
Sample thickness (mm)	6

friction in the material. The attenuation is in fact the sound waves' energy drop while the waves are traveling through the material.

In the present experimental study, the Tru-Sonic Ultrasonic Testing Device was employed to measure propagation velocity of ultrasonic waves. The specifications of the device, samples, and probe are shown in Table 2. With regard to more severe attenuation effects in rubbers, a probe with a frequency of 4 MHz was used to determine the velocity of ultrasonic waves [15]. The propagation velocity of ultrasonic waves for different samples was measured via measurement of the period between sending waves and reflecting them and then its appearance on the monitor with an accuracy of 1 m/s. The measurement system is illustrated in Fig. 2.

Given the fact that the calculated time between sending and receiving the waves include the time interval of wave propagation from the probe and the coupler, the calculated time has some errors which results in an incorrect calculated velocity.

Therefore, the transmission time of longitudinal ultrasonic waves through the probe and the coupler was measured by a standard calibration block, and it was omitted from the calculations. Calibration standard block is used for the calibration of transverse and longitudinal wave probes, time base and sensitivity settings.

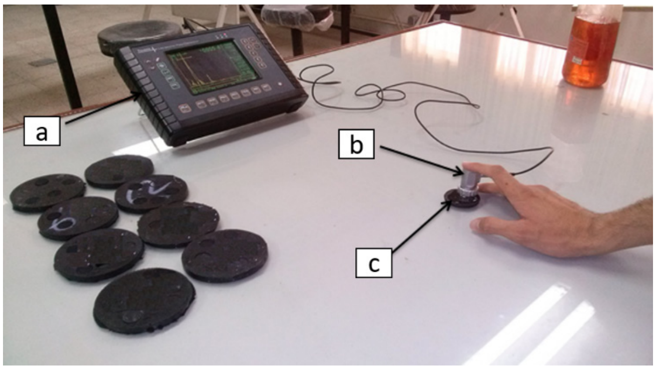

Fig. 2. The Ultrasound Wave Measurement System (a) Ultrasonic device, (b) Probe, (c) Rubber compound sample

3- Relevance Vector Machine

Thanks to the advances in machine learning in recent decades, many problems in engineering can be solved easier. Supervised learning can be described as generation of a model from dependency of the targets on the inputs based on a set of N observed input vectors $\{x_n\}_{n=1}^{N}$ and the corresponding targets $\{t_n\}_{n=1}^{N}$ in order to estimate the targets in case of inputs which have not been observed. Supervised learning algorithms can be used to establish a global model from the functional relationship between the outputs and the inputs based on a limited number of measurements [16].

Support vector machines (SVMs) are supervised learning models with associated learning algorithms used for classification and regression analysis [17] and have been proven to be efficient in many practical applications [18]. In SVM-based regression, the input space is mapped into a high dimensional feature space based on a set of kernel functions, and, then, a linear optimal regression is performed in this space which can be expressed as follows:

$$f(x) = y(x_i, w) = \sum_{i=1}^{N} w_i K(x, x_i) + w_0, \tag{2}$$

where $\{w_i\}$ are the model weights, $K(.,.)$ is a kernel function and N is the number of training samples. Despite its widespread success, the SVM suffers from some disadvantages, which have been overcome in a newer probabilistic approach named Relevance Vector Machine (RVM) proposed by Tipping [19]. RVM is a nonlinear pattern recognition model with simple structure based on Bayesian Theory and Marginal Likelihood. The main advantage of RVM over SVM in our application is the fact that besides the precision and sparseness, this method utilizes very fewer number of kernel functions and therefore it is suitable for definition of an explicit mathematical expression for description of the outputs based on the observed inputs.

In RVM-based regression, in order to achieve a function based on a set of N input-target pairs $\{x_n, t_n\}_{n=1}^{N}$, each target is modeled as a function of the corresponding inputs with additive white Gaussian noise to accommodate measurement error in the target,

$$t_i = y(x_i, w) + \varepsilon_i, \tag{3}$$

where ε_i is assumed to be mean-zero Gaussian with variance σ^2 and similar to the SVM, $y(x, w)$ is considered as a linear combination of N kernel functions centered at the training samples inputs, in form of (2). Therefore, with the assumption that we know $y(x_n)$, each target is independently distributed as Gaussian with the mean $y(x_n)$ and variance σ^2, expressed as :

$$p(t_n|x) = N(t_n|y(x_n), \sigma^2). \tag{4}$$

Due to the assumption that the targets are independent, the likelihood function of all samples can be obtained by multiplication of the probability distributions as

$$p(t|w, \sigma^2) = \frac{e^{\left\{\frac{-t-\varphi w^2}{2\pi\sigma^2}\right\}}}{(2\pi\sigma^2)^{\frac{N}{2}}}, \tag{5}$$

where

$$\boldsymbol{t} = (t_1 \dots t_N)^T, \tag{6}$$

$$\boldsymbol{w} = (w_0 \dots w_N)^T, \tag{7}$$

and φ is an $N \times (N+1)$ matrix calculated by,

$$\varphi_{N*(N+1)} = \begin{bmatrix} 1 & k(x_1, x_1) & k(x_1, x_2) & \cdots & k(x_1, x_N) \\ 1 & k(x_2, x_1) & k(x_2, x_2) & \cdots & k(x_2, x_N) \\ \vdots & \vdots & \vdots & \ddots & \vdots \\ 1 & k(x_N, x_1) & k(x_N, x_2) & \cdots & k(x_N, x_N) \end{bmatrix} \tag{8}$$

The vector of weights, $\boldsymbol{w}$, can be obtained by training such that across a given set of training data, the likelihood function (5) is maximized. However, this direct approach will lead to over-fitting such that generalization to new observations is not acceptable. Therefore, a 'prior' zero-mean Gaussian probability distribution is assumed for the weights as,

$$p(w|\alpha) = \prod_{i=0}^{N} N(w_i|0, \alpha_i^{-1}) \tag{9}$$

where $\boldsymbol{\alpha}$ is a vector of $N+1$ hyper-parameters [20]. The variance of this Gaussian probability distribution, α_i^{-1}, controls how far from zero each weight can deviate and when α_i tends to infinity. The corresponding weight w_i is peaked at *0* and therefore it can be estimated as *0*. Using the Bayesian inference [21], learning process of RVM is defined as a search for the parameters α and σ^2 which maximize the marginal likelihood $p(t|\alpha, \sigma^2)$ based on the training dataset [22]. In this procedure, many of hyper-parameters α_i tend to infinity, resulting in zero-valued weights, thus pruning many of the kernel functions used in (2), resulting in a shorter mathematical expression. The training set, which associates with the remaining nonzero weights is called the relevance vector.

4- Results and discussion

In this survey, non-destructive tests using ultrasonic waves were used to examine the resilience of rubber compounds and a database of sixteen values of resilience percentage and the corresponding ultrasonic longitudinal waves' speed was obtained, which is listed in Table 3. Thirteen samples of the measurements were used for training the RVM model and three of them, indicated by *, were applied to test the model accuracy. To improve the accuracy, the input and target values were normalized between −1 and +1 as

$$pn = 2 * \frac{p - \left(\frac{max + min}{2}\right)}{(max - min)}, \tag{10}$$

where *max* and *min* respectively represent the maximum and minimum value of the input or the output among the whole dataset; p is the input or output and pn is the corresponding normalized value. Therefore, the inputs are normalized as

$$xn = \frac{x-1579.6}{27.9165}, \tag{11}$$

and the outputs are normalized as

$$yn = \frac{y-32.3}{5.7}\ . \tag{12}$$

Based on the normalized dataset, RVM model was implemented by the Sparse Bayes package for Matlab [23] {Tipping, 2009 #186} and predicted outputs were scaled to their original range based on (11) as

$$\hat{y} = y_n * \left(\frac{max-min}{2}\right) + \left(\frac{max+min}{2}\right) = y_n * 5.7 + 32.3, \tag{13}$$

where $\hat{y}$ is the estimated output in the original range and y_n is the normalized estimated output. Using the Gaussian kernel function formulated as (14), with the parameter $\sigma^2 = 0.18$, the relevant vector contains only two of the training samples and therefore based on the calculated weights, the functional relationship between resilience and the longitudinal waves' speed can be expressed as (15).

$$K(x, x_i) = \exp\left(-\frac{x - x_i^2}{\sigma^2}\right) \tag{14}$$

$$\hat{y} = \left(-0.6031 * \exp\left(-\frac{(x_n+1)^2}{0.0324}\right) + 0.7605 * \exp\left(-\frac{(x_n-1)^2}{0.0324}\right)\right) * 5.7 + 32.3 \tag{15}$$

Table 3. Resilience and longitudinal ultrasonic waves' velocity of samples

No.	longitudinal ultrasonic waves' velocity (m/s)	Resilience (%)
1	1555.5	31.4
2	1575	34.6
3	1561	27.8
4	1551.667	26.6
5	1580.5	28.3
6	1559	33.3
7	1570.33	30.2
8	1571.667	32
9	1607.5	38
10	1578	32.7
11	1580.33	32
12	1581.5	34
13	1564.667	35.3
14*	1574	35.3
15*	1584.33	27.8
16*	1587.33	34

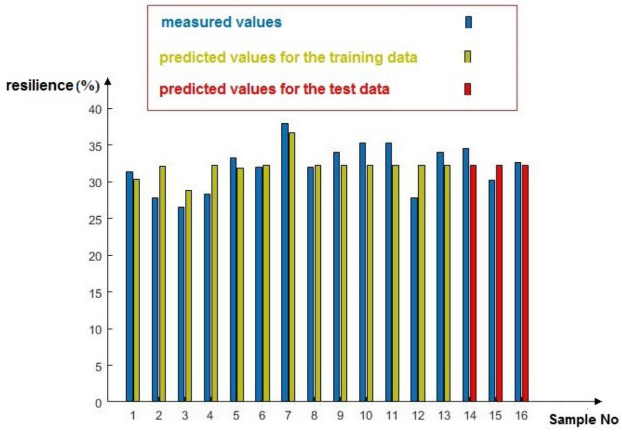

Fig. 3. The measured outputs and the outputs predicted by the RVM method

The accuracy of the results was evaluated based on the root means square error (RMSE) and normalized root means square error (NRMSE) respectively expressed by

$$RMSE = \sqrt{\frac{\sum_{i=1}^{N}\left(y_i - \hat{y}_i\right)^2}{N}}, \tag{16}$$

$$NRMSE = \frac{RMSE}{\bar{Y}}, \tag{17}$$

where y_i and $\hat{y}_i$ are the measured and the predicted outputs, respectively; N is the number of training samples, and $\bar{Y}$ is the mean value of the measured outputs. The calculated value of indices are listed in Table 4. As it can be observed, the RVM method provides a reasonable accuracy and generalization capability as well as possibility of defining an explicit mathematical expression. The measured outputs together with the outputs estimated by the RVM method are depicted in Fig. 3, showing a reasonable agreement between them.

Table 4. Statistical indices for evaluation of the RVM model

Database	RMSE	NRMSE
Training	2.6	0.0514
Testing	1.81	0.0447

5- Conclusion

In this paper, application of ultrasonic non-destructive testing for measurement and modelling of the rubber's resilience has been investigated. For this purpose, a database of resilience together with the corresponding measurements for the longitudinal ultrasonic waves' velocity is obtained. Based on the database, relevance vector machine regression analysis is used to define an explicit mathematical expression to model the relationship between resilience and waves' velocity, which is proven to provide a reasonable accuracy besides generalization capability. Based on the results, ultrasonic non-destructive testing can be used for online measurement of the rubber's resilience in tire industry.

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