**The Effect of Varying Types of *Curing Agent* EPDM on IRM 903 *Swelling Resistance* Using Fractional Factorial and *Artificial Neural Network Methods***

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**Abstract.** Ethylene-Propylene-Diene-Monomer (EPDM) is a type of synthetic elastomer that has a drawback in its resistance to oil and polar fluids because EPDM is a nonpolar elastomer. However, this property can be enhanced using other additives or curing system formulations. Therefore, this study selected the experimental design method to obtain optimal values in terms of oil resistance by determining the curing system in the EPDM elastomer formula using fractional factorial design and Artificial Neural Network (ANN) methods. The parameter kept constant was the EPDM Compound masterbatch formula. The process parameters used were variations in the curing system designed using a fractional factorial approach with Minitab21. The response parameters studied were mechanical properties, heat aging, and IRM 901 oil resistance. The results of this study showed that the fractional factorial experimental design model created using Minitab21 identified significant process parameters affecting the response parameters, with only the mechanical properties being significantly influenced, affected by the DPTT70 and sulfur parameters. Therefore, regression methods cannot be used to analyze the influence of curing agents on oil resistance and accelerated aging, which will be further analyzed using the main effects plot diagram. Data from the experimental design model used for ANN prediction simulation input showed that the ANN model was well-trained and is expected to have satisfactory predictive performance and generalization capability without overfitting.

**Keywords:** EPDM, Curing Agent, ANN

# INTRODUCTION

The elastomer industry is predicted to experience significant growth in the next few years, especially in the automotive sector which is looking for ways to reduce vehicle weight for fuel efficiency. Among the various types of elastomers, Ethylene-Propylene-Diene-Monomer (EPDM) is one of the most advantageous due to its stability against heat, oxidation, ozone and weather aging. However, EPDM has a major disadvantage in terms of resistance to oil and polar fluids due to its nonpolar nature [1].

Previous research shows that mixing EPDM with certain additives can improve its physical and chemical properties [2]. Mixtures such as stearic acid, zinc oxide as an activator, carbon black filler FEF N- 550, CBS accelerator, and sulfur as a vulcanizing agent have been proven effective. Various curing systems such as sulfur, peroxide, metal oxides, phenolic resins, and quinones have also been developed, where the type of curing system applied determines the structure and cross-link density of the elastomer [3].

Weaknesses in oil resistance in EPDM can be solved through experimentation and optimized using the *Artificial Neural Network* (ANN) method [4]. This method has been proven to have high accuracy in predicting elastomer mechanical properties, such as modulus and tensile strength, with better results than conventional methods such as *Central Composite Design* (CCD). ANN is also used to characterize the mechanical capabilities of natural rubber under accelerated aging conditions, with excellent prediction results [5].

This research aims to determine the optimal oil resistance characteristics of EPDM with variations in curing system composition using fractional factorial and ANN experimental design methods. This research will identify the combination of curing parameters that most significantly influence EPDM oil durability and utilize this data as input for the ANN model.

By combining fractional factorial and ANN experimental design methods, this research is expected to make a significant contribution to the development of EPDM compound characteristics. The results of this research can also be used as a reference for the manufacturing industry in research and development of EPDM compound products that are more resistant to oil and polar fluids.

# LITERATURE REVIEW

## Previous Research

Research related to the formulation of oil-resistant EPDM compounds has widely used the Artificial Neural Network (ANN) method and full factorial design. For example, Ruziak I. et al. [7] used ANN to predict the mechanical properties of NRs with high RMSE and R2 statistical functions. Research by Vijayabaskar V. et al. [8] also showed that ANN produces accurate predictions for the tensile strength and volume fraction of rubber with a high coefficient of determination. Previous research has explored various aspects of EPDM elastomers and compounds. For example, research conducted by Boden et al. [9] studied the influence of varying sulfur and peroxide as cross-linking agents on the mechanical properties and thermal resistance of EPDM vulcanizates. They found that the combination of these two agents provided optimal results in terms of tensile strength and elongation. Apart from that, research by Mayasari HE et al. [10] discussed the use of various accelerators in the EPDM vulcanization process and their impact on scorch time and curing time, showing that Thiurams-based accelerators provide significant results in reducing the vulcanization process time.

Another study by Ghaffarian N. et al. [11] investigated the effect of temperature and pressure in the vulcanization process on the final mechanical properties of EPDM. This research shows that proper vulcanization conditions are critical to achieving desired mechanical properties, such as hardness and abrasion resistance. The results of this research provide a strong basis for further development in the field of EPDM-based elastomer formulation and production processes.

## Elastomer

Elastomers are polymers that have elastic properties, allowing the material to return to its original shape after experiencing deformation. Elastomers are commonly used in a variety of industrial applications due to their flexibility and resistance to various environmental conditions. According to Persson et al. [12], elastomers such as natural rubber and synthetic rubber are widely used in the manufacture of tires, seals, and other automotive components due to their ability to withstand high deformations without losing structural integrity.

The main characteristics of elastomers include low elastic modulus and high deformation ability. The vulcanization process is used to improve the mechanical properties and thermal resistance of elastomers. Vulcanization involves the formation of cross-links between polymer chains, which increases tensile strength and resistance to various chemical agents. Research by Maciejewska et al. [13] showed that the type and amount of cross-linking agent used in the vulcanization process greatly influences the final properties of the elastomer.

## Ethylene-Propylene-Diene Monomer (EPDM)

Ethylene-Propylene-Diene Monomer (EPDM) is a type of synthetic rubber that is widely used in the automotive and construction industries because of its resistance to ozone, heat and weather. EPDM has a chemical structure consisting of ethylene, propylene, and diene, which provides flexibility and resistance to various environmental conditions. According to research by Gungor et al. [2], EPDM also has good resistance to water and chemicals, making it ideal for applications that require resistance to moisture and corrosive substances.

One of the main advantages of EPDM is its ability to maintain elasticity at low to high temperatures. Research by Williams et al. demonstrated that EPDM can be used in applications requiring flexibility at temperatures as low as - 40°C and as high as 150°C. This makes EPDM very useful in a variety of industrial applications, from car door seals to roof membranes.

## Curing System

Curing is an important process in elastomer vulcanization, which involves the formation of cross-links between polymer chains to improve mechanical properties and thermal resistance. According to research by Maciejewska et al. [12], curing systems can be classified into conventional (CV), semi-efficient (semi-EV), and efficient (EV) based on the sulfur/accelerator ratio used. CV systems have high sulfur content and low accelerators, resulting in longer polysulfide cross-links.

The semi-EV system has a balanced sulfur and accelerator content, providing optimal mechanical properties and thermal stability. Meanwhile, the EV system has a low sulfur content and a high accelerator, resulting in a short scorch time, high vulcanization rate, and higher cross-link density compared to the CV system. Research by Boonkerd et al.

[13] showed that the selection of an appropriate curing system is very important to achieve the desired final properties in elastomer products.

## Physio-mechanical characteristics

Physio-mechanical characteristics of elastomers include tensile strength, elongation, hardness, and abrasion resistance. Research by Piersol et al., [14] shows that tensile strength and elongation are strongly influenced by the type and amount of cross-linking agent used. Elastomers with more cross-linking tend to have higher tensile strength and abrasion resistance. Elastomer hardness is measured using the Shore A scale and is related to the density of cross- links in the material. Research by Nasruddin [15] shows that increasing the accelerator content in the vulcanization process can increase the hardness of the elastomer. Abrasion resistance is also an important characteristic that determines the longevity of the material in applications involving friction and contact with rough surfaces.

## Rheometry

Rheometry is a technique used to measure the flow and deformation properties of materials, which are important in determining the viscosity and elasticity of elastomers during the vulcanization process. Research by Nakajima [16] shows that rheometry can provide information about minimum (ML) and maximum (MH) torque, which is related to compound viscosity and stiffness modulus of vulcanized rubber. Scorch time (Ts2) and cure time 90% (Tc90) are important parameters in rheometry that determine the efficiency of the vulcanization process. Research by Mayasari et al. [9] showed that the sulfur concentration and type of accelerator greatly influence Ts2 and Tc90. A low Ts2 is desired to increase production efficiency, while an optimal Tc90 is important to ensure the quality of the final vulcanisate.

## Oil Resistance

Oil resistance is the ability of an elastomer to withstand the effects of oil solvents without experiencing significant degradation. Research by Mayasari et al. [9] showed that the oil resistance of elastomers is mainly influenced by the density of cross-links in the material. The higher the cross-link density, the lower the swelling percentage of the elastomer when immersed in oil, indicating better resistance to solvents.

Swelling is a phenomenon in which an elastomer absorbs a solvent and expands, which can affect the mechanical properties of the material. Research by Darko [17] shows that elastomers with higher cross-linking have better swelling resistance, because cross-linking reduces the free volume in the rubber matrix and makes it more difficult for solvents to penetrate the material.

## Experimental Design

Experimental design is a methodology used to design and analyze experiments with the goal of maximizing the information obtained from the data. According to Montgomery [18], good experimental design can help identify important factors that influence the results and interactions between these factors. Factorial and response surface designs are two common approaches to experimental design.

The factorial design approach involves testing all combinations of levels of several factors to understand their interactions. Response surface design, on the other hand, is used to model and optimize responses based on changes in factor levels. Research by Jones et al. [19] shows that the use of appropriate experimental design can reduce the number of experiments required and increase the efficiency of the product development process

## Factorial Design Method

The factorial design method is a statistical approach to studying the effects of several factors and their interactions on a response. According to Eriksson et al. [20], this method is very effective in identifying key factors and their interactions that influence the results. Full and partial factorial designs are the two main types used in experimental research

## Artificial Neural Network (ANN)

Artificial Neural Network (ANN) is a computational model that imitates the way the human brain works in processing information. ANNs are used in a variety of applications, including prediction and optimization of industrial processes. Research by Hertz (1992) shows that ANNs can learn patterns from training data and make accurate predictions for new data. ANN is used to model non-linear relationships between input and output, and can be integrated with factorial design methods to optimize industrial processes.

# RESEARCH METHOD

## Tools and Materials

This research was conducted in the Mixing Department of PT. Velasto Indonesia for six months, starting from February 2023 to August 2023. The research location was chosen because the facilities and tools available were in accordance with the needs of the experiments being carried out. Various tools used in this research include AND GF- 300 Analytical Scales for material weighing and specific gravity testing, Moriyama Laboratory Kneader DS3- 10MWB-E for compound master batch preparation, Yi Tzung Laboratory Open Mill D03-1 for making samples with a variety of systems curing, and Tung Yu 50-ton Open Press Molding Machine for making CS Block and Dumbbell test pieces. Apart from that, Teclock Durometer Shore A was also used for hardness testing, ZPM Z-10 Tensile Tester Machine for testing tensile strength and elongation at fracture, Ektron MDR Rheometer to measure the viscoelastic ability of rubber compounds during the vulcanization process, Yamato Scientific Drying Oven for aging testing. accelerated, test tubes for oil resistance tests, and Yasuda-Seiki Test Tube Aging Oven for heating test tubes containing specimens and IRM 903 oil.

All Materials used in this research are industrial grade supplied by Indonesian and foreign maker. Following materials that used in this research are EPDM Vistalon 7001 (Exxon Mobil Chem), Carbon Black FEF N-550 (Cabot)

, Paraffinic Oil 95 (Pertamina), ZNO NC 105 (Global Chem), Stearic Acid (Sumi Asih), Hirenol KPT-1250K (Kolon Chem), Rhenogran DPTT-70 (Rhein Chemie), TMTD (Akrochem), CBS (Kemai Chem), Accel EM33 (Kawaguchi Chem), Dicumyl Peroxide (Hanwha Chem), ZDBC (Puyang Willing Chem), Midas SP-325 (Miwon Chem), Rhenofit TRIM-S (Rhein Chemie), and Omyacarb (Omya).

## Shore Hardness Testing A

Shore A hardness testing is carried out to measure the mechanical properties of elastomers, namely hardness, based on the JIS K6253 standard. In this research, testing was carried out using a Teclock Durometer Shore A to ensure consistency of results and measure changes in hardness that occurred after the vulcanization and accelerated aging processes.

## Tensile Strength and Elongation at Break Testing

Tensile strength testing was carried out using a ZPM Z-10 Tensile Tester Machine to measure the ability of elastomers to withstand tensile loads until they break, according to JIS K6251 standards. While the elongation at break test measures how far the elastomer can stretch before breaking, it is also carried out based on the same standards. These two tests are important to determine the strength and flexibility of the resulting material

## Rheometry and Oil Resistance Testing

Rheometry testing was carried out using the Ektron MDR Rheometer to measure the viscosity and elasticity of rubber during vulcanization, producing ML, MH, Ts2, and Tc90 data. For oil resistance, test tubes and the Yasuda- Seiki Test Tube Aging Oven were used to test changes in the mechanical properties of elastomers after being immersed in IRM 903 oil at high temperatures, following established oil resistance test procedures.

## Fractional Factorial Design Simulation

In the Minitab software, the fractional factorial method was chosen with 15 runs. The experimental design table is presented in the following table:

**TABLE 1.** Experimental Design Matrix as Independent Variable

|  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **Run** | **HS** | **TB** | **E.B** | **M.L** | **MH** | **T s 2** | **T c 90** | **Aging HS** | **Aging TB** | **Aging EB** | **Oil Res HS** | **Oil Res TB** | **Oil Res EB** | **Oil Swell** |
| 1 | H 1 | T 1 | E 1 | ML 1 | MH 1 | TS 1 | TC 1 | AH 1 | AT 1 | AE 1 | OH 1 | OT 1 | OE 1 | SW 1 |
| 2 | H 2 | T 2 | E 2 | ML 2 | MH 2 | TS 2 | TC 2 | AH 2 | AT 2 | AE 2 | OH 2 | OT 2 | OE 2 | SW 2 |
| 3 | H 3 | T 3 | E 3 | ML 3 | MH 3 | TS 3 | TC 3 | AH 3 | AT 3 | AE 3 | OH 3 | OT 3 | OE 3 | SW 3 |
| 4 | H 4 | T 4 | E 4 | ML 4 | MH 4 | TS 4 | TC 4 | AH 4 | AT 4 | AE 4 | OH 4 | OT 4 | OE 4 | SW 4 |
| 5 | H 5 | T 5 | E 5 | ML 5 | MH 5 | TS 5 | TC 5 | AH 5 | AT 5 | AE 5 | OH 5 | OT 5 | OE 5 | SW 5 |
| 6 | H 6 | T 6 | E 6 | ML 6 | MH 6 | TS 6 | TC 6 | AH 6 | AT 6 | AE 6 | OH 6 | OT 6 | OE 6 | SW 6 |
| 7 | H 7 | T 7 | E 7 | ML 7 | MH 7 | TS 7 | TC 7 | AH 7 | AT 7 | AE 7 | OH 7 | OT 7 | OE 7 | SW 7 |
| 8 | H 8 | T 8 | E 8 | ML 8 | MH 8 | TS 8 | TC 8 | AH 8 | AT 8 | AE 8 | OH 8 | OT 8 | OE 8 | SW 8 |
| 9 | H 9 | T 9 | E 9 | ML 9 | MH 9 | TS 9 | TC 9 | AH 9 | AT 9 | AE 9 | OH 9 | OT 9 | OE 9 | SW 9 |
| 10 | H 10 | T 10 | E 10 | ML 10 | MH 10 | TS 10 | TC 10 | AH 10 | AT 10 | AE 10 | OH 10 | OT 10 | OE 10 | SW 10 |
| 11 | H 11 | T 11 | E 11 | ML 11 | MH 11 | TS 11 | TC 11 | AH 11 | AT 11 | AE 11 | OH 11 | OT 11 | OE 11 | SW 11 |
| 12 | H 12 | T 12 | E 12 | ML 12 | MH 12 | TS 12 | TC 12 | AH 12 | AT 12 | AE 12 | OH 12 | OT 12 | OE 12 | SW 12 |
| 13 | H 13 | T 13 | E 13 | ML 13 | MH 13 | TS 13 | TC 13 | AH 13 | AT 13 | AE 13 | OH 13 | OT 13 | OE 13 | SW 13 |
| 14 | H 14 | T 14 | E 14 | ML 14 | MH 14 | TS 14 | TC 14 | AH 14 | AT 14 | AE 14 | OH 14 | OT 14 | OE 14 | SW 14 |
| 15 | H 15 | T 15 | E 15 | ML 15 | MH 15 | TS 15 | TC 15 | AH 15 | AT 15 | AE 15 | OH 15 | OT 15 | OE 15 | SW 15 |

The validity of the response parameters obtained from the experiment was tested using the one-way ANOVA method in Minitab software to evaluate the influence of each process parameter on the experimental results obtained. Then predictive analysis was carried out using the regression method in Minitab software. Regression analysis is used to fit a linear model between process parameters and response parameters.

## Artificial Neural Network (ANN) Simulation

*Data Normalization*

Data or response parameters obtained from experiments are normalized or converted into values with a limit of 0 to 1 so that the data is not dynamic. The calculation formula for data normalization is as follows:

N = Data−Dmi𝚗

Dmax −Dmi𝚗

With:

(1 )

* N = Normalized data
* D = Data to be normalized
* D max = Upper limit of data
* D min = Lower limit of data

*Artificial Neural Network (ANN) Design*

The ANN model was processed using Matlab software with the neural tool (nnstart) toolbox. The stages of using this toolbox begin by opening the neural network tool, then entering process parameters as input and response parameters as output. After that, a neural network model was created and continued with training using the Bayesian regularization algorithm. After training, simulation is carried out to produce simulated output. This process is repeated by carrying out training and re-simulation, using the number of neurons as a fixed variable for each different type of response parameter.

*Data Denormalization*

The simulated output obtained from ANN training and simulation is converted into actual values using the following formula:

𝐷 = 𝐻 𝑥 (𝐷𝑚𝑎𝑥 − 𝐷𝑚𝑖𝑛) + 𝐷𝑚𝑖𝑛 ( 2)

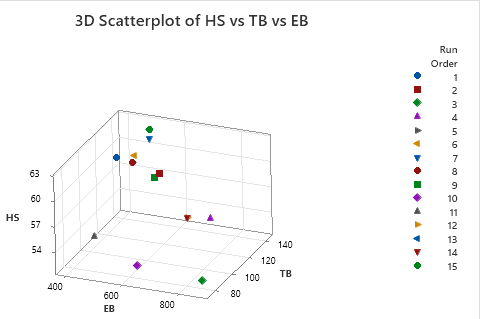
With:

* D = Normalized data
* H = Simulation Results Data
* D max = Upper limit of data
* D min = Lower limit of data

# RESULTS AND DISCUSSION

## Discussion of Fractional Factorial Experimental Design Results

In this research, the fractional factorial experimental design was carried out using Minitab 15 software. Experiments carried out based on the experimental design model will analyze the influence between process parameters and response parameters. In addition, the experimental results obtained are used as input in the ANN prediction simulation. The experimental results are presented in the figure below.



**FIGURE 1** . 3D Scatterplot of Mechanical Properties Test Results



**(a)**

Mooney Low

**(b)**

Mooney High

0.80 20.00

0.60 15.00

0.40 10.00

0.20 5.00

0.00 0.00

0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15

Run Order Run Order

**(c)**

Scorch Time

**(d)**

Cure Time 90%

250

200

150

100

50

0

450

400

350

300

250

200

150

100

50

0

0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15

Run Order Run Order

ML (dNm)

MH (dNm)

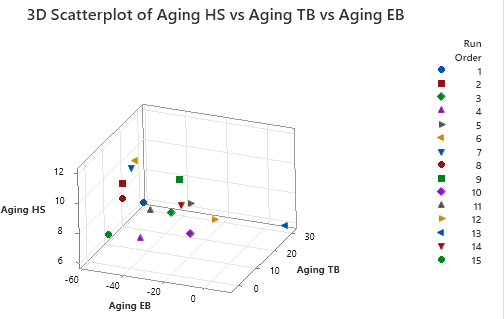
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**FIGURE 2.** Rheometry Test Results (a) ML value; (b) MH value; (c) Ts2 value; (d) Tc90 value

Ts2 (sec)

Tc90 (sec)



**FIGURE 3.** 3D Scatterplot of Aging Test Results



**(a)** Oil Res. HS Change

**(b)**

Oil Res. TB Change

0

-10 0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15

-20

-30

-40

-50

Run Order

0

0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15

-50

-100

-150

Run Order

HS Change (Point)

TB Change (%)

|  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| 0 1 |  | 2 | 3 | 4 | 5 | 6 | 7 8 |  | 9 10 | 11 | 12 | 13 | 14 | 15 |
|  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |

**FIGURE 4.** Oil Resistance Test Results (a) Hardness Change Value; (b) Tensile Change Value; (c) Elongation Change



**(c)** Oil Res. EB Change

**(d)**

Oil Res. Volume Swell

0

-20

-40

-60

250

200

150

100

50

0

-80

Run Order

0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15

Run Order

EB Change (%)

Volume Change (%)

Value; (d) Volume Swell Value

*Regression Analysis of Experimental Design Results*

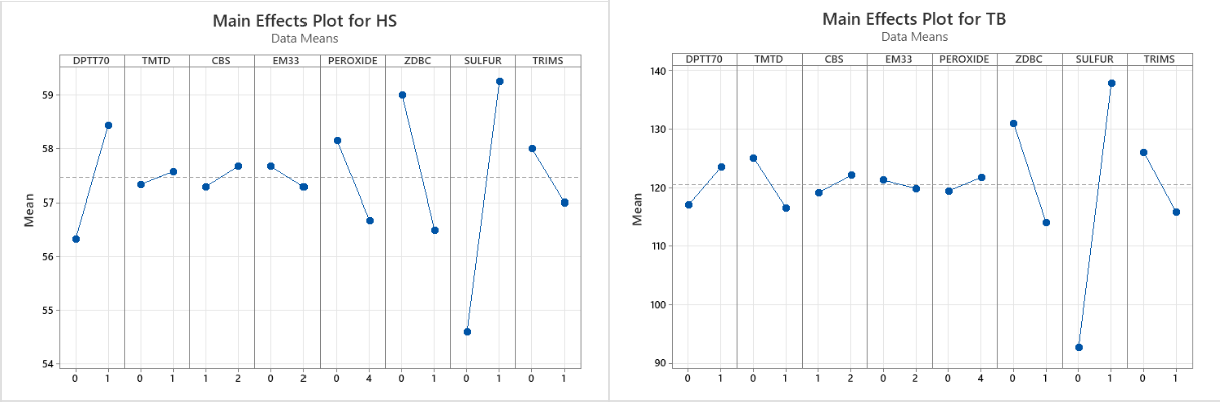
The results obtained through experiments were processed using Minitab 15 software to obtain ANOVA (Analysis of Variance) values as well as mathematical models of response parameters to process parameters. ANOVA can be useful in determining the significance of each linear, quadratic and interaction term in the model through probability values (P-value).

**TABLE 2.** P-Value Value from ANOVA

|  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **P-value** | **DPTT70** | **TMTD** | **CBS** | **EM33** | **PEROXIDE** | **ZDBC** | **SULFUR** | **TRIMS** |
| HS | 0.006 | 0.587 | 0.616 | 0.724 | 0.018 | 0.304 | 0 | 0.724 |
| TB | 0.006 | 0.587 | 0.616 | 0.724 | 0.018 | 0.304 | 0 | 0.724 |
| E.B | 0.029 | 0.227 | 0.643 | 0.771 | 0.55 | 0.344 | 0 | 0.336 |
| S.G | 0.001 | 0.666 | 0.254 | 0.021 | 0.701 | 0.962 | 0.001 | 0.007 |
| M.L | 0.981 | 0.439 | 0.272 | 0.448 | 0.463 | 0.128 | 0.817 | 0.551 |
| MH | 0.012 | 0.651 | 0.227 | 0.898 | 0.965 | 0.121 | 0.002 | 0.672 |
| TS2 | 0.109 | 0.913 | 0.387 | 0.235 | 0.275 | 0.967 | 0.02 | 0.51 |
| TC90 | 0.282 | 0.122 | 0.513 | 0.076 | 0.695 | 0.208 | 0.518 | 0.411 |
| Aging HS | 0.925 | 0.245 | 0.179 | 0.912 | 0.664 | 0.556 | 0.05 | 0.874 |
| Aging TB | 0.942 | 0.844 | 0.719 | 0.575 | 0.852 | 0.475 | 0.474 | 0.403 |
| Aging EB | 0.833 | 0.735 | 0.876 | 0.573 | 0.564 | 0.396 | 0.464 | 0.828 |
| Oil Res HS | 0.621 | 0.359 | 0.443 | 0.777 | 0.761 | 0.173 | 0.301 | 0.816 |
| Oil Res TB | 0.561 | 0.184 | 0.704 | 0.549 | 0.942 | 0.447 | 0.585 | 0.881 |
| Oil Res EB | 0.011 | 0.034 | 0.51 | 0.088 | 0.096 | 0.106 | 0.011 | 0.543 |
| Oil Swell | 0.275 | 0.793 | 0.132 | 0.297 | 0.472 | 0.917 | 0.032 | 0.769 |

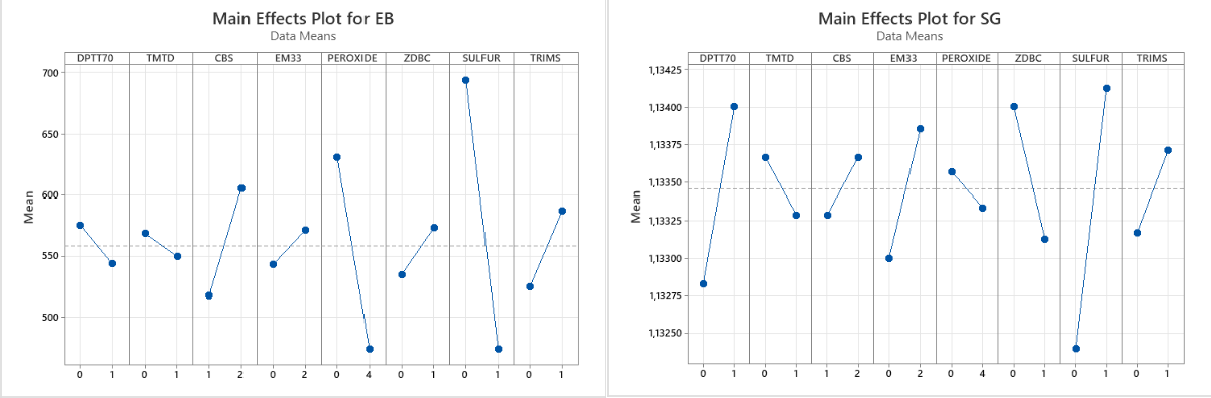
The significance of the quadratic model obtained in Table 2 for all process parameters which have a P value of less than 0.05 has a significant influence on the response parameters [22]. In this study, the process parameters that were significant to the process parameters were only found in the mechanical properties with the dominant parameters, namely DPTT70 and sulfur, while the parameters with other curing agents did not have a significant effect. Therefore, the regression method cannot be used to analyze the effect of curing agents on oil durability and accelerated aging which will then be analyzed using a main effects plot diagram.

*Effect of Curing Agent on Mechanical Properties*



**(a)**

**(b)**



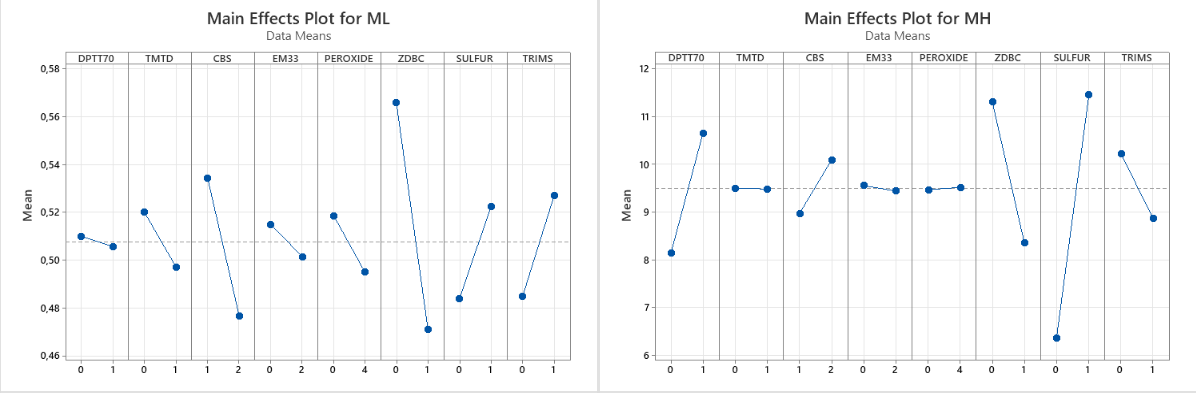
**(c)**

**(d)**

**FIGURE 5.** Main Effects Plot Curing Agent on Mechanical Properties *(a) Hardness; (b) Tensile; (c) Elongation; (d) Specific Gravity*

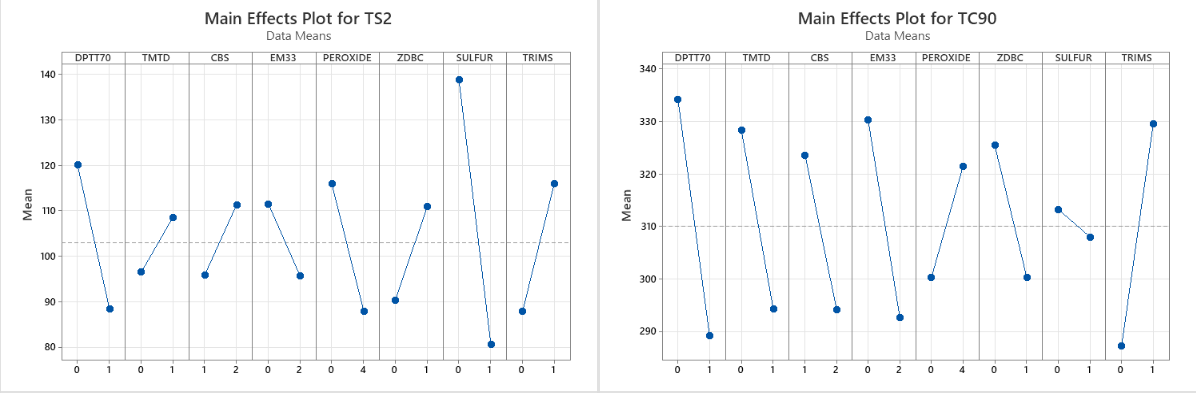
In Figure 5 it is shown that curing agents have a significant influence on the mechanical properties of EPDM, especially on variations in sulfur and peroxide. These two agents act as crosslinking agents in vulcanization, combining carbon-carbon linkages and sulfide bridges to modify the physico-mechanical properties of the vulcanizate. The combination of these vulcanization systems influences the vulcanization characteristics, crosslink density, and physical-mechanical properties of the material. Vulcanizates using a combination of sulfur and peroxide show higher tensile strength and elongation compared to those using only one of these systems [4].

*Effect of Curing Agent on Rheometry*



**(a)**

**(b)**



**(c)**

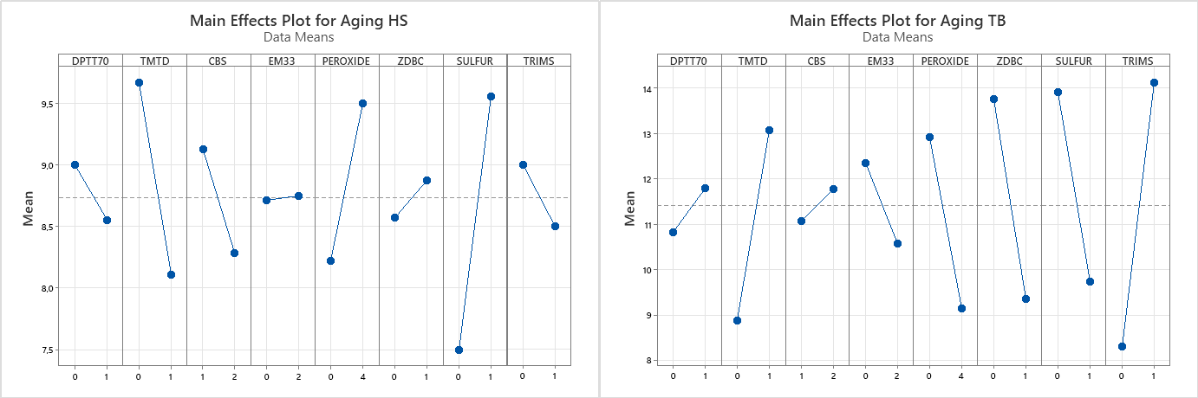
**(d)**

**FIGURE 6.** Main Effects Plot Curing Agent on Rheometry *(a) Mooney Low; (b) Mooney High; (c) Scorch Time; (d) Cure Time 90%*

In Figure 6, the rheometer test results show that the ML and MH parameters are most influenced by the ZDBC and Sulfur Accelerator. According to Mayasari HE et al. [9], MH is the maximum torque which indicates the stiffness modulus of vulcanized rubber, while ML is the minimum torque which is related to the viscosity and processability of the compound. The maximum torque (S'MH) represents the cross-linking of the vulcanization process, where higher MH values provide good dimensional stability and thermal resistance. A low ML value makes the compound injection process easier because the viscosity is low.

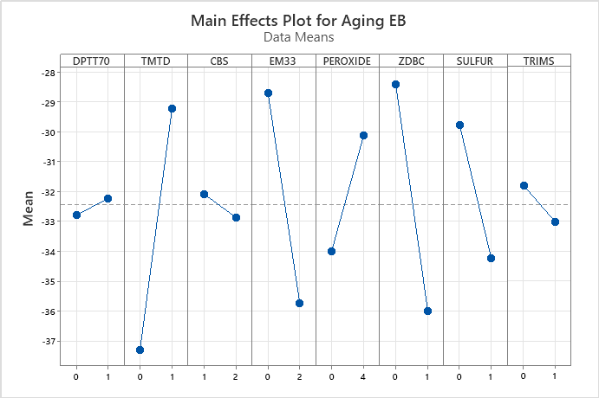
Scorch Time (Ts2) is influenced by the sulfur concentration, while Cure Time 90% (Tc90) is influenced by the composition of the accelerator used. Scorch time is the initial time of vulcanization, and a low scorch time increases production efficiency, although it should be noted that high temperatures can cause scorching or polymer degradation. Tc90 determines the most optimal vulcanization time, determined when the maximum torque (MH) is reached, before the reversion or overcure phenomenon occurs [24].

*Effect of Curing Agent on Accelerated Aging*



**(a)**

**(b)**

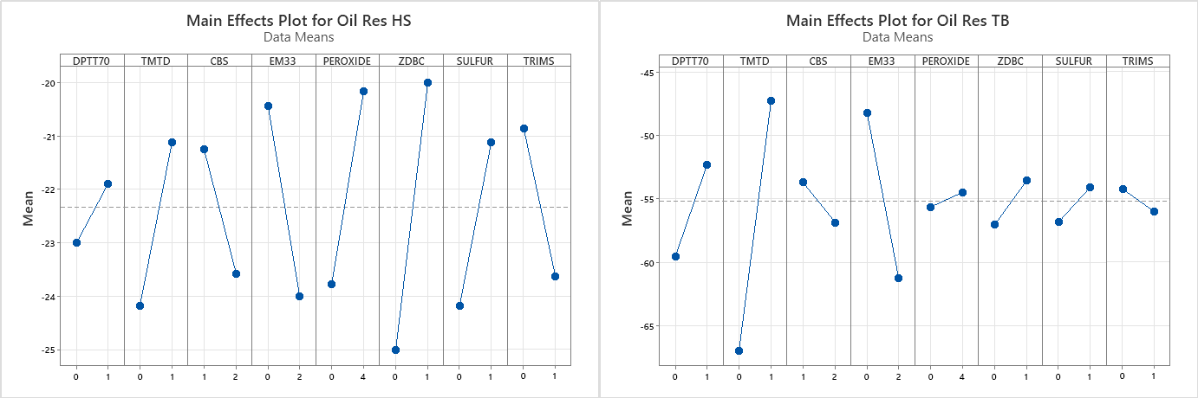


**(c)**

**FIGURE 7.** Main Effects Plot Curing Agent on *Accelerated Aging (a) Hardness Change; (b) Tensile Change; (c) Elongation Change*

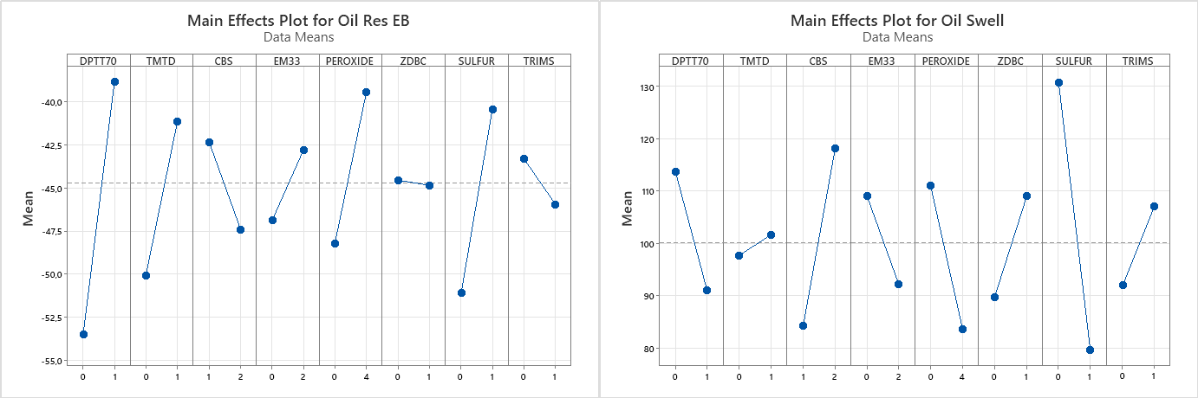
In Figure 7, accelerator is the composition that has the most influence on the heat resistance of the compound because of its stronger cross-link structure. Maciejewska M. et al. [12] stated that the cross-link structure mainly depends on the sulfur content and the sulfur/accelerator ratio in the vulcanization system. Vulcanization systems are classified into conventional (CV), semi-efficient (semi-EV), and efficient (EV) based on this ratio. CV systems have high sulfur content and low accelerators, resulting in long polysulfide cross-links that improve mechanical and dynamic properties but worsen thermal stability and thermo-oxidative aging resistance. The semi-EV system has a balanced sulfur and accelerator content, providing optimal mechanical properties and thermal stability. EV systems have low sulfur content and high accelerators, resulting in short scorch times, high vulcanization rates, and higher cross-link density compared to CV.

*Effect of Curing Agent on Oil Resistance*



**(a)**

**(b)**



**(c)**

**(d)**

**FIGURE 8.** Main Effects Plot Curing Agent on Oil Resistance *(a) Hardness Change; (b) Tensile Change; (c) Elongation*

*Change; (d) Oil Swell*

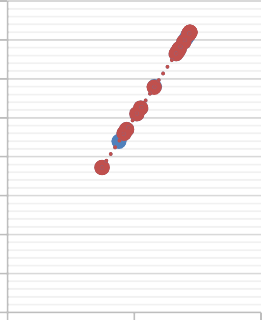
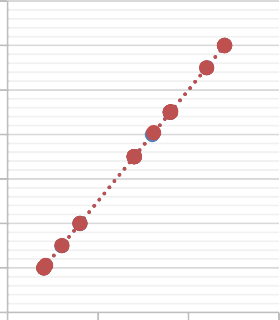
In Figure 8, the results show that the effect of curing agents on oil resistance is mainly influenced by the oil swell value. EPDM soaked in Oil IRM 903 expands up to 150% of its original shape, causing a decrease in the mechanical ability of the compound. Swelling in elastomers occurs due to pressure between the rubber matrix and the solvent, which causes the matrix to expand or shrink. Soaking in IRM 903 increases the rubber mass due to solvent diffusion into the rubber matrix. The lower the swelling percentage, the higher the resistance to solvents. This is associated with stronger cross-linking, reducing the free volume in the rubber matrix and making it more difficult for solvents to penetrate the matrix [9].

According to Darko C. (2022), swelling in polymers is influenced by the cross-link density; Lower density allows the absorption of more solvent molecules, while higher density limits solvent absorption. Therefore, TMTD, as a Thiurams type accelerator used in semi-EV systems, produces polysulfidic bonds that are resistant to swelling [13].

## Discussion of Artificial Neural Network Results

In this research, the fractional factorial experimental design was carried out using Minitab 15 software. Experiments carried out based on the experimental design model will analyze the influence between process parameters and response parameters. In addition, the experimental results obtained are used as input in the ANN prediction simulation. The experimental results are presented in the figure below.

*Evaluation of ANN Prediction Data*



200

100

Predicted (Kgf/cm²)

0

R² = 1

Experiment

ANN

160

140

120

100

80

60

40

20

0

Tensile Strength

**(b)**

50 55 60 65

Predicted (Shore A)

R² = 1

Experiment

ANN

64

62

60

58

56

54

52

50

Hardness

**(a)**

Experimental (Shore A)

Experimental (Kgf/cm²)

**FIGURE 9.** Comparison between actual experimental result value and predicted value of *(a) Hardnes; (b) Tensile; (c)*



**(c)** Elongation at Break

1000

800

600

400

200

0

R² = 1

Experiment

ANN

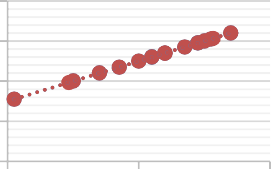
0 100 200 300 400 500 600 700 800 900 1000

Predicted (%)

Experimental (%)

*Elongation*

The artificial neural network method can predict the mechanical capabilities of materials, which in this research are Hardness, Tensile Strength, and Elongation at Break. The accuracy of this method is shown by the MSE value of the hardness parameter which is close to 0 with the comparison between the experimental value and the predicted results shown in Figure 9. Generally, among Rubber Technologists, hardness parameters can be predicted using a rule- of-thumb system where each addition of PHR carbon will increase the hardness value of a rubber compound by considering the particle size of the carbon itself. In the Tensile Strength and Elongation at Break parameters, it was found that there were errors in several variables, namely MSE Tensile Strength of 182.04 in Variable 12 and MSE of 1943.63;385.79 in Elongation of variable 6;13. This shows that the nonlinear behavior of elongation at break is higher than other outputs. Therefore, ANN requires more experience in learning complex nonlinear relationships for elongation at break than for other outputs.



**(a)**

0.8

0.6

0.4

0.2

0

Mooney Low (ML)

R² = 1

Experiment

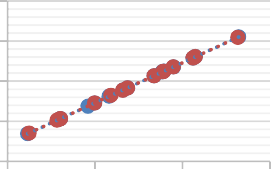
ANN

0.3

0.5

Predicted (dNm)

0.7



**(b)**

20

15

10

5

0

Mooney High (MH)

R² = 1

Experiment

ANN

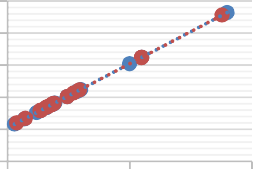
2.3 7.3 12.3 17.3

Predicted (dNm)

Experimental (dNm)

Experimental (dNm)

**FIGURE 10.** Comparison between actual experimental result value and predicted value of *(a) Mooney Low; (b) Mooney*



**(c)**

Scorch Time (TS2)

250

200

150

100

50

0

R² = 1

Experiment

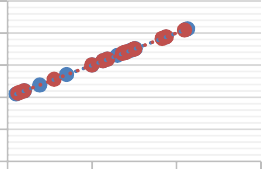
ANN

52.3

152.3

Predicted (sec)

252.3



**(d)**

Cure Time 90% (TC90)

500

400

300

200

100

0

R² = 1

Experiment

ANN

200 300 400 500

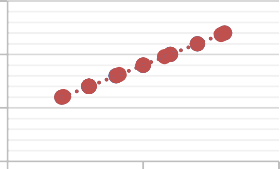
Predicted (sec)

Experimental (sec)

Experimental (sec)

*High; (c) Scorch Time; (d) Cure Time 90%*

The predicted rheometry values presented in Figure 10. contain several points of inaccuracy between experiment and prediction because developing a predictive ANN model can be a challenge due to the nonlinear nature of rubber vulcanization. A suitable ANN architecture is the most challenging part of building a model and it is hoped that an ANN model with a sufficient number of hidden layers can provide adequate results. Rheometry values obtained from ANN predictions which have good accuracy with MSE values close to 0 (zero) and form a linear line (R2 = 1) such as on ML result as shown in Figure 10a.



**(a)** Ageing Test Hardness Change

15

10

5

R² = 1

Experiment

0

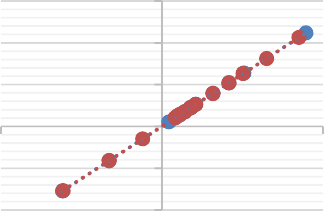
ANN

4

9

Predicted (Point)

14



**(b)**

Ageing Test Tensile Change

60

40

-50

20

0

-20 0

-40

Predicted (%)

R² = 1

Experiment

50

ANN

Experimental (Point)

Experimental (%)

**FIGURE 11.** Comparison between actual experimental result value and predicted value of *(a) Ageing HS Change; (b)*



Predicted (%)

-60

R² = 1

Experiment

ANN

-20

-40

20

10

0

-10

-20

-30

-40

-50

-60

0

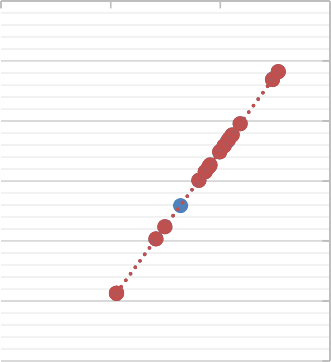
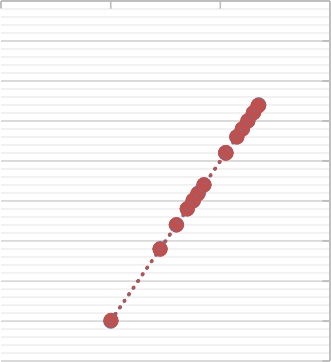
Ageing Test Elongation Change

20

**(c)**

Experimental (%)

*Ageing TB Change; (c) Ageing EB Change*



**(a)**

Oil Test Hardness Change

0

**(b)**

Oil Test Tensile Change

0

-60 -40 -20

-5 0

-10

-15

-20

-25

-30

-35

-40

-45

-150

-100

-50

0

-20

-40

R² = 1

Experiment

ANN

-60

-80

R² = 1

Experiment

ANN

-100

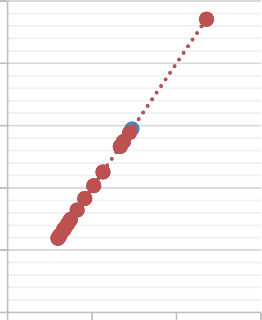
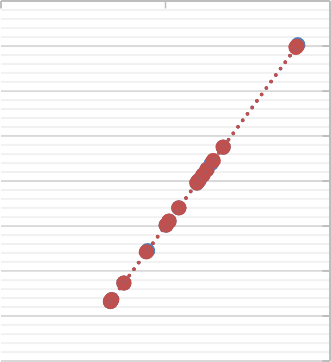
-120

Predicted (Point) Predicted (%)

Experimental (Point)

Experimental (%)

**FIGURE 12.** Comparison between actual experimental result value and predicted value of *(a) Oil HS Change; (b) Oil TB Change; (c) Oil EB Change; (d) Volume Swell*



Predicted (%)

100 200 300

0

50

0

Experiment

ANN

**(d)** Oil Test Volume Swell

250

200

150

R² = 1

100

Predicted (%)

-80

R² = 1

Experiment

ANN

-30

-40

-50

-60

-70

-10 0

-20

-100 -50

**(c)** Oil Test Elongation Change

0

Experimental (%)

Experimental (%)

The aging value obtained from the EB aging test presented in Figure 11. has good MSE accuracy of all aging tests parameters. Ageing of rubber was mainly correlated with temperature effect which may degrade rubber polymer structure gradually. In this case the uncontrolled condition of crosslink density built by curing system were breakdown over time during ageing testing evaluation, even in some variable this ANN model can’t accurately predict the tensile and elongation change. It can be fixed by giving more input data variable to give more information on this ANN model to reduce unstable data variation.

The ANN modeling simulation results presented in Figure 12. show accurate prediction results for each oil resistance test parameter. Oil resistance in elastomers is mainly based on the principle of swelling due to absorption of a solvent or fluid, which in this study used IRM 903 oil. Swelling occurs in 3 stages: solvent absorption occurs on the polymer surface, the polymer penetrates the polymer, and the polymer structure expands when the solvent trapped in the pores penetrates the polymer chain network.

# CONCLUSION

From the results of the research that has been carried out, it can be concluded that in the fractional factorial experimental design method, the best oil resistance was obtained in the 1st Run-Order where, in this variable, the curing agent was used at the maximum level. This shows that oil resistance is influenced by the crosslink density of the vulcanizate. Then in the ANN method EPDM oil resistance can be predicted accurately using the feed forward backpropagation method with 10 neurons, and the RMSE and MAE parameters (which indicate the residual error between the observed value and the predicted value) are very small, while R 2 (represents the proportion of variability in the prediction results) very close to the maximum value, namely 1 for all response parameters. This means that the ANN model has been well trained, and is expected to have satisfactory predictive performance.

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