

## DEVELOPMENT OF AN ECO-EFFICIENT PRODUCT/PROCESS FOR THE VULCANISING INDUSTRY

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### ABSTRACT

This paper presents the development of an eco-efficient product/process, which has improved mechanical properties from the introduction of natural fibres in the EPDM (Ethylene-Propylene-Diene-Terpolymer) rubber formulation. The optimisation analysis is made by a fractional factorial design <sup>2</sup><sup>11-7</sup>. Different formulations were evaluated using a multi-response desirability function, with the aim of finding efficient levels for the manufacturing time-cycle, improving the mechanical properties of the product, and reducing the raw material costs. The development of an eco-efficient product/process generates a sustainable alternative to conventional manufacturing.

### OPSOMMING

Die ontwikkeling van 'n omgewingsdoeltreffende produk of proses, wat verbeterde meganiese eienskappe het as gevolg van die toevoeging van natuurlike vesels in die EPDM ('n tipe polimeer) rubber formulering word voorgehou. Die optimiseringsanalise is gedoen deur 'n breukdeel faktoriaal ontwerp <sup>2</sup><sup>11-7</sup>. Verskillende formulerings is geëvalueer deur 'n multi-reaksie wenslikheidsfunksie met die doel om doeltreffende vlakke vir die vervaardigingstydskilus te vind en sodoende die meganiese eienskappe van die produk te verbeter en die roumateriaalkoste te verminder. Die ontwikkeling van 'n omgewingsdoeltreffende produk of proses genereer 'n volhoubare alternatief tot konvensionele vervaardiging.

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## 1 INTRODUCTION

Some decades ago, thermoplastic materials replaced metal in the automotive sector when they were accepted as engineering materials. Due to demand, thermoplastics have increased up to 20% in cost. This increased cost created the need for employing fillers in thermoplastic materials to generate compounds and reduce costs. Among the reinforcements recently used are natural fibres, which offer a lot of advantages; they are low in cost, have a low density, and they are renewable, abundant, and environmentally friendly. Natural fibres also have a high degree of flexibility during processing, and do not damage the equipment [1].

### 1.1 Natural fibres in the industry and conventional processes

While many researchers have focused on the use of natural fibres as an alternative material, government entities have been seeking to establish new policies for the use and handling of materials to ensure that there is as little negative impact as possible on the environment. The industrial sector, on the other hand, is searching for greater acceptance of green products and environmentally-friendly processes, which include the introduction of natural fibres. Table 1 presents examples of work involving natural fibres that has been conducted by researchers within different process groups.

Table 1: Studies with natural fibres

Process	Author	Fibre type	Obtained product	Application
Injection moulding	Amigó et al. (2008)	Cotton Flax Sisal Hemp Fique Kenaf [2]	HDPE compound recycled/cotton. HDPE compound recycled/flax. HDPE compound recycled/sisal. HDPE compound recycled/hemp. HDPE compound recycled/fique. HDPE compound recycled/kenaf.	Textile sector
	Ruksakulpiwat et al. (2009)	Vetiver grass [3]	NR/vetiver/PP compound. PP/EPDM/vetiver compound.	Industrial
	Shojaei and Fereydoon (2009)	Short glass fibre [4]	PA6/glass fibre/EPR-g-MA compound.	Industrial
	Gava et al. (2010)	Short silica fibre [5]	Nylon 6/EPDM-g-MA compound.	Variety
	Seong et al. (2011)	Carbon [6]	Carbon fibre/PA6 compound.	Automotive industry
	Kord (2011)	Wood flour [7]	PP/wood flour/PP-MA compound.	For watery media
	Zhao et al. (2011)	Rice straw [8]	PLA/rice straw compound.	Packing film for food
	Becerra et al. (2011)	Agave fibre (Tequilana Weber) [9]	LDPE/tequilana agave compound.	Industrial
	Chaochanchaikul et al. (2012)	Sawdust particles [10]	Saw dust particles/HDPE compound.	Construction

Table 1: Studies with natural fibres (continued)

Process	Author	Fibre type	Obtained product	Application
Compression moulding	Herrera and Valadéz (2005)	Henequen [11]	PE/henequen compound.	Industrial
	Sanjuan and Jasso (2009)	Agave fibre (Tequilana Weber)	PP virgin/tequilana agave/PP-MA compound. PP recycled/tequilana agave/PP-MA compound. PP re-processed/henequen/PP-MA. PP post-consumer/henequen/PP-MA compound.	Industrial
		Henequen [12]		
	Ismail and Shaari (2010)	Ash palm and halloysite [13]	Palm ash/halloysite nanotubes/EPDM compound.	Marketing
	Anuar and Zuraida (2011_)	Kenaf [14]	Thermoplastic NR/kenaf compound. PP/EPDM compound.	Automotive components
	Valente et al. (2011)	Wood flour	LDPE/wood compound. LDPE/wood/glass fibre compound. LDPE/wood/glass fibre/recycled glass fibre compound. PP/wood compound. PP/wood/glass fibre compound. PP/wood/glass fibre/recycled glass fibre compound.	Industrial
		Recycled glass fibre [15]		
	Viet et al. (2011)	Kenaf powder [16]	Kenaf powder/HDPE recycled/NR/PE-MA compound.	Marketing
	Zabihzadeh et al. (2011)	Rapeseed stems [17]	PP/rapeseed compound.	Panels
	Najafi and Khademi (2011)	Rice husks flour	HDPE recycled/rice husks flour compound. HDPE recycled/sawdust flour compound. HDPE recycled/sanded panel flour compound.	Panels
Sawdust and panels flour [18]				
Singha and Rana (2012)	Americana agave [19]	PS/Americana agave/MMA compound.	Wire coating	
Safwan et al. (2013)	Palm kernel Shell [20]	Palm kernel shell/PP/palm nanosilica. Almond shells/PP compound.	Automotive	
Extrusion	Mahdavi et al. (2010)	Date palm [21]	Date palm/HDPE compound.	Commercial use
	Acharya et al. (2011)	Sugar cane bagasse [22]	Sugar cane bagasse/epoxy compound.	Several applications
Rotational moulding	López et al. (2012)	Agave fibre (Tequilana Weber) [23]	LMDPE/ tequilana agave compound.	Industrial
Mixing	Lárusson et al. (2013)	Polyvinyl alcohol fibres [24]	Reinforced with cement and fibre compound.	Concrete panels
Fibre wash	Alawar et al. (2009)	Date palm tree fibres [1]	Date palm tree fibre.	Textile

## 1.2 Research problem and objectives

The aim of this research was to investigate and develop a new alternative for the disposal of tequila agro-industries' waste in Mexico [23]. The great amount of waste generated after extracting tequila juice is causing environmental problems [25, 27]. This waste can be used as a rubber reinforcement to improve the mechanical properties of the product, reduce time in the manufacturing cycle, and decrease costs of raw materials.

The objective of this work is to develop a sustainable vulcanised EPDM (Ethylene-propylene-diene-terpolymer) rubber product by introducing natural agave fibres in the formulations, using a non-conventional manufacturing process with a multi-response desirability function where mechanical properties are improved, costs of raw materials are decreased, and the manufacturing time is evaluated.

## 2 METHODOLOGY

2.1 The strategy for designing experiments using the response is summarised as follows:

1. Determination of factors, levels and response variables.

A brainstorming exercise was carried out among a group of experts to identify all the factors affecting the process and product. A cause-effect diagram was then used to determine the factors and levels according to the technological knowledge of the process, combining practical experience and written reports. The response variables were determined according to the client's quality specifications and the manufacturer's interest in the process's operational conditions [28, 30].

2. Determination of the fractional factorial design considering costs and time [28, 30].

3. Statistical data analysis was carried out through a technical variance analysis (ANOVA), normally used in experiment design [28, 30].

4. Obtaining a regression equation to interpret the process behaviour according to Castaño and Domínguez (2010) [28] and Gutiérrez and De La Vara (2008) [30].

$$Y_j = \beta_0 + \sum_{i=1}^k \beta_i X_i + \varepsilon \quad (1)$$

5. Process optimisation through the multi-response desirability function [28, 30].

A general focus of the multi-response function is that after the regression equations are found and properly adjusted, it can proceed through the individual desirability function  $d_j$  to give desirable values to each of the response variables. The  $d_j$  value is found in the interval  $0 \leq d_j \leq 1$ . If  $d_j$  is 1, then the value of the variable is the most desirable, however if  $d_j$  is 0, the variable is not desirable [28]. The value for  $d_j$  is given by equation (2).

$$d_j = \begin{cases} \left( \frac{\hat{y}_j - LIE_j}{T_j - LIE_j} \right)^s, & \text{si } LIE_j \leq \hat{y}_j \leq T_j \\ \left( \frac{\hat{y}_j - LSE_j}{LSE_j - T_j} \right)^t, & \text{si } T_j \leq \hat{y}_j \leq LSE_j \\ 0 & \text{si } \hat{y}_j \geq LSE_j \text{ o } \hat{y}_j \leq LIE_j \end{cases} \quad (2)$$

where  $\hat{y}_j$  is the predicted value for the response variable;  $LIE_j$  is the inferior specification limit;  $LSE_j$  is the superior specification limit;  $T_j$  is the most desirable value;  $s$  and  $t$  are exponents to select the required form of desirability in each response. Global desirability ( $D$ ) is considering all the response variables through their desirability functions; a method proposed by Derringer and Suich [31], which is to maximise  $D$ , known as the geometrical measure of  $d_1, d_2, \dots, d_m$  [28, 30].

$$D = (d_1^{w_1} d_2^{w_2} \dots d_m^{w_m})^{1/\sum w_i} \quad (3)$$

where  $W_i$  weights are steady, allowing the relative importance of each response variable to be balanced. The higher the weight given to a variable in relation to the remaining variables, the greater the weight in the optimisation.

In other words, if all variables are equally important,  $W_i$  is equal to 1. Note that the exponent  $s$  and  $t$  can be introduced as part of the  $W_i$  weights [28, 30].

For the procedure applying the desirability function, regression equations were constructed separately from each response, and were then determined by weighting the responses respectively to minimise, maximise, and maintain.

Afterwards, the multi-response optimisation was carried out, considering the importance of each response, comparing it with the others, and seeking a solution in which all the responses had an acceptable level. The D desirability function is measured on a scale of 0 to 1. The closer the value of the D global desirability function to 1, the more acceptable the product [28, 30].

### 3 EXPERIMENTAL

The following equipment and materials were used in the experiment:

#### 3.1 Equipment

Brabender internal mixer (DDRV-502). Brabender external mixer (PM-3000). Hydraulic press (Carver 38954DI1A00). Imperial stove V. Sartorius analytic scale (AG BP 4100). Rheometer oscillating disc (ODR 2000). Steel mould (150 mm x 150 mm x 3 mm). Standard die cut tear strength (ASTM-D624). Standard die cut elongation at break (ASTM-D412). Die cutting pneumatic (ATOM SE-20-C-C). Durometer type Shore A PT#T588-59 (ASTM-D2240). Instron machine 365.

#### 3.2 Materials

EPDM IP 4725P (Dow Nordel®), viscosity Mooney ML1+4 a 125°C, 70% of ethylene. Molecular medium weight distribution. Thermal carbon black (N990®Degussa). Stearic acid (Vstearin®SA29). Zinc oxide (xzinal 821 active). Paraffinic oil (324). Benzothiazyl disulfide (MBTS). Tiuram disulfide ultra accelerators (TMTD). Compatibilising agent EPDM-MA (Fusabond N MF-4160 Dupont®). Sulphur in powder [3, 32, 33] and agave fibre (Tequilana Weber) [9, 12, 23].

### 4 METHOD OF MANUFACTURING

Agave fibre (Tequilana Weber) was dried in a stove for 24 hours at 80°C to remove any moisture [11, 34, 35].

The production of tequila begins with the planting of agave (the Weber Tequilana plant, which is native to Mexico). The plant must mature between 7 and 10 years. Once mature, the leaves of the plant are removed, leaving the pineapple, which weighs about 150 kg.

After extracting the juice from the agave for the tequila production process, the industry generates a large amount of solid waste. For example, 8 kg of bagasse are generated for every litre of tequila. One ton of bagasse has approximately 70 kg of fibre and 350 kg of pulp as reusable sub-products.

#### 4.1 Preparation of raw material

An analytical scale was used to weigh all the components used in the different formulations for each experimental treatment. Carbon black (40, 80 PCH), paraffinic oil (15, 40 PCH), stearic acid (0.5, 2 PCH), zinc oxide (3, 5 PCH), EPDM-MA (2, 7 PCH), MBTS (0.7, 2 PCH), TMTD (0.1, 0.6 PCH), agave fibre (Tequilana Weber in Mexico) (10, 40 PCH) and vulcanising agent (1, 2.5 PCH) [32, 36, 39].

## 4.2 Programming of operational parameters

Operational parameters were set for the internal mixer (120°C, 50 rpm, 10 minutes) [16, 20], external mixer (120°C, 20 rpm) and hydraulic press (160°C, 7 and 10.5 minutes) [11, 13, 19, 38, 41]. The mould was allowed to preheat inside the press.

## 4.3 Manufacturing of rubber/fibre product

The process, which started in the internal mixer, mixed the components for 10 minutes. The EPDM rubber melted in one minute. Next, stearic acid, ZnO, EPDM-MA, MBTS and TMTD, was added and mixed for three minutes. Afterwards, black carbon and paraffinic oil were added and mixed for one minute. Immediately after this, fibre was added and mixed for two minutes, and lastly sulphur was added and mixed for three minutes. The mixture was removed, and 20 g was weighed and laminated in the external mixer. The rest of the mixture was poured into the preheated mould, producing 3 mm thick sheets [38, 39, 42, 44]. The description of the non-conventional process of fabricating vulcanised rubber for the rubber/fibre product is shown in Figure 1.

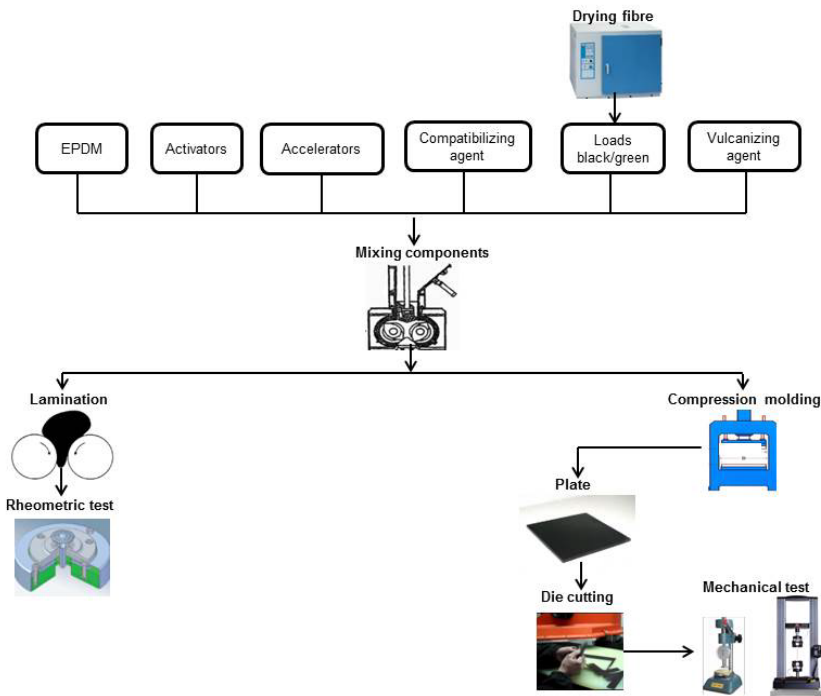


Figure 1: Diagram of the non-conventional process of fabricating vulcanised rubber with natural fibre

## 4.4 Rubber/fibre product testing

Rheometric tests were made (curves of vulcanisation) in an oscillating disc rheometer to obtain the manufacturing cycle time (MCT) [33, 43] and raw material cost (CM). Elongation at break (EB) tests were made in an Instron machine 365 (ASTM-D412) with a velocity of 500 mm/min [41, 43]. The hardness (HSA) tests were made with durometer type Shore A (ASTM-D2240) [41, 43]. Tear strength (TR) tests were made (ASTM-D624) with a velocity of 400 mm/min in an Instron machine 365.

## 5 EXPERIMENTAL DESIGN

The two types of experimental design used in this investigation are a fractional design  $2_{III}^{11-7}$  = 16 experiment [28], and a non-replicated design of resolution III. In these resolution III designs, the principal effects are not alias among them can be consulted [28], but there are

principal effects that are alias of double interaction [28, 30]. The variables were established using a codification high value 1 and low value -1 according to the Yates nomenclature [29, 30]. The quantitative factors, levels and five-response variables are presented in Table 2. The objective is to determine whether the change in the levels of factors has a statistically significant effect on the response variables of the rubber/fibre product and non-conventional vulcanising process.

**Table 2: Experimental factors and levels**

Factors	Code	Levels		Measurement Units
		Low -1	High 1	
Carbon black	A	40	80	PCH <sup>a</sup>
Paraffinic oil	B	15	40	PCH
Stearic acid	C	0.5	2	PCH
Zinc oxide	D	3	5	PCH
EPDM-MA	E	2	7	PCH
MBTS	F	0.7	2	PCH
TMTD	G	0.1	0.6	PCH
Fibre	H	10	40	PCH
Sulphur	I	1	2.5	PCH
Vulcanisation time	J	7	10.5	minutes
Mixing temperature	K	105	120	°C

<sup>a</sup> Rubber hundred parts

## 6 RESULTS AND DISCUSSIONS

The results of the experiments are shown in Table 3. The experimental treatments were made in a random order, and the experimental data was analysed using Statgraphics and Minitab.

### 6.1 Variances analysis MCT, CM, EB, HAS, and TR

With these results, an ANOVA analysis of each response variable was made separately to see what factors played an important role in the process and product (see Table 4). The procedure for the variance analysis can be consulted [28, 30]; some effects have P-values less than 0.05, indicating that they are statistically significant.

### 6.2 Multi-response optimisation

The desirability function was proposed by Harrington [45], and improved by Derringer and Suich [31]. The multi-response optimisation determines the best factor and experimental levels arrangement in order to fulfil the requirements of the desired response variables at an optimal point [28, 30].

The desirability function method is applied to the regression equations (4), (5), (7) and (8), based on the product quality specifications and the manufacturer's interest where MCT and CM are minimised; EB and TR are maximised and HAS is maintained in 70 Shore A. The regression equation (6) was not included in the estimate of the desirability function, because it was not significant [28].

The results from the optimisation that applied the multi-response desirability function are shown in Figure 2, where two non-significant factors (stearic acid and mixing temperature) were removed. Note that there are factors that have non-significant effects in some responses; however, they were included because they can have significant results in the other responses.

**Table 3: Results experimental design 2<sup>11-7</sup><sub>III</sub>**

Exp.	Coded Factors											Response Variables				
	A	B	C	D	E	F	G	H	I	J	K	MCT <sup>a</sup>	CM <sup>b</sup>	EB <sup>c</sup>	HSA <sup>d</sup>	TR <sup>e</sup>
1	1	-1	-1	1	1	1	-1	-1	1	-1	-1	9.3	15.1	676.4	79.6	40.4
2	-1	1	1	1	-1	1	-1	-1	-1	-1	-1	10.6	14.0	892.5	67.6	31.3
3	-1	1	1	-1	-1	-1	1	1	1	-1	-1	9.7	12.8	844.7	80.6	33.3
4	-1	-1	-1	1	-1	1	1	1	-1	1	1	9.2	14.6	611.3	71.3	40.0
5	1	-1	1	1	-1	-1	1	-1	-1	-1	1	7.7	14.0	648.1	69.6	47.1
6	-1	-1	1	1	1	-1	-1	1	1	1	-1	7.3	15.6	407.1	74.3	33.2
7	1	-1	1	-1	-1	1	-1	1	1	-1	1	10.2	13.0	644.1	79.00	43.1
8	-1	-1	-1	-1	-1	-1	-1	-1	1	1	1	9.7	15.9	833.7	71.3	41.5
9	-1	1	-1	-1	1	1	-1	1	-1	-1	1	10.4	13.9	770.4	75.6	35.9
10	1	1	-1	-1	-1	1	1	-1	1	1	-1	8.1	15.0	673.5	62.0	34.7
11	1	-1	-1	-1	1	-1	1	1	-1	-1	-1	7.7	14.0	149.0	81.0	46.2
12	-1	1	-1	1	1	-1	1	-1	1	-1	1	8.5	15.1	649.1	71.0	28.8
13	1	1	-1	1	-1	-1	-1	1	-1	1	-1	9.8	14.0	783.5	64.3	31.8
14	1	1	1	-1	1	-1	-1	-1	-1	1	1	10.3	15.8	953.1	70.0	35.2
15	1	1	1	1	1	1	1	1	1	1	1	8.1	14.9	598.3	60.3	31.3
16	-1	-1	1	-1	1	1	1	-1	-1	1	-1	9.6	17.2	689.8	66.3	40.5

- <sup>a</sup> Manufacturing cycle time measurement unit: minutes
- <sup>b</sup> Cost of raw materials measurement unit: Mexican pesos
- <sup>c</sup> Elongation at break measurement unit: %
- <sup>d</sup> Hardness measurement unit: Shore A
- <sup>e</sup> Tear strength measurement unit: KN/m

**Table 4: Results from the ANOVA analysis**

ANOVA	Significant factors	Discussion	Regression equations derivate from(1)
TR	A B D I	A is a reinforcement that allows partial immobilisation in rubber chains, increasing tearing strength. B is used to give flexibility to the product. D is an activator that accelerates the reaction with sulphur. I is the cross-linking agent used to make the vulcanising reaction.	$TR=37.18+1.59A-4.36B-1.65D-0.70E+0.006F+0.59G-0.29H-1.36I-1.12J$ (8)
MCT	G	Organic composite that releases sulphur when it reaches vulcanisation temperatures, forming cross-linkages. G increases the vulcanising time for the rubber/fibre product, which decreases the manufacturing cycle time in the new non-conventional process of fabricating vulcanised rubber for the rubber/fibre product.	$MCT=9.17-0.23A+0.28B-0.31D-0.25E+0.3F-0.54G-0.1H-0.27I-0.11J$ (4)





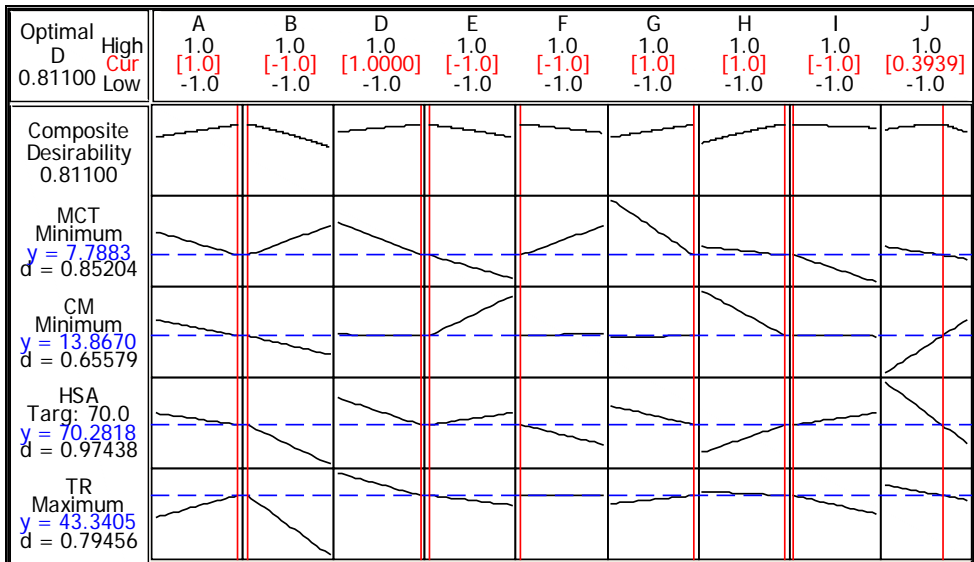


Figure 2: The global desirability function to evaluate the effect of each factor

It is important to describe the other components shown in the optimisation. The blue lines determine the  $d_j$  individual desirability value of the responses. A  $d_j$  value close to 1 means that the desirability degree in the response is acceptable; thus it is concluded that the four responses have an acceptable value. The D global desirability has a highly acceptable value, which means that the whole product and process are acceptable, reaching an optimal global response.

The red lines are a control, showing the point that indicates the selected level of the factor that meets the optimisation, and corresponds to the value that is marked between red brackets [ ] in the line referring to the factors.

In this way, the following objectives are met:

- Developing a sustainable product for vulcanised EPDM rubber with the introduction of natural fibres in the formulation;
- Implementing a non-conventional process of rubber vulcanisation where the manufacturing cycle time MCT and raw material costs CM are minimised;
- Maintaining the hardness HSA in industrial specifications; and
- Maximising the elongation to break EB and tear strength TR, through the multi-response desirability function.

Therefore the best combination of coded factors and levels is: A [1], B [-1], D [1], E [-1], F [-1], G [1], H [1], I [-1] and J [0.4].

## 7 CONCLUSION

The optimisation of a non-conventional process of vulcanised EPDM rubber was carried out in this research with the multi-response desirability function, centred on a fractional factorial design  $2_{III}^{11-7}$  with five responses. The experimental strategy allowed only a few tests to be done while generating substantial knowledge of the non-conventional process of vulcanised rubber and ideal performance.

The objective was to determine the best configuration of factors and their levels in order to:

- minimise the manufacturing cycle time (MCT);
- minimise the raw material cost (CM);
- maintain the hardness at 70 in Shore A;
- maximise the elongation to breaking point as well as to tearing for a sustainable rubber/fibre product; and
- to implement an eco-efficient and non-conventional process of rubber vulcanisation.

The multi-response desirability function is a highly effective technique in the optimisation of the process and quality of products. From the eleven factors studied, only nine analysed factors have a statistically-significant effect on the responses of the rubber/fibre product and the newly-developed process. The experiment evaluated charges, oil, activators, accelerants, compatibilising agent, fibre, vulcanising agent, vulcanising time, and mixing temperature.

The responses were individually analysed to find the best ANOVA to obtain the adjusted regression equations. These regression equations satisfactorily describe the new process data. Therefore the best combination of decoded factors and levels is: A (80 PCH), B (15 PCH), D (5 PCH), E (2 PCH), F (0.7 PCH), G (0.6 PCH), H (40 PCH), I (1 PCH) and J (7 minutes).

This arrangement allows us to conclude that the new process optimised with the multi-response desirability function maintains the sustainable product with the required characteristics to meet the desired eco-efficiency and be environmentally friendly. Finally, the multi-response optimisation is an alternative that provides solutions to several problems of a product in a process. This allows process engineers to be more involved in the process and to identify more of the factors that make an impact on the product and the process.

## NOMENCLATURE

HDPE	High density polyethylene
NR	Natural rubber
PP	Polypropylene
PE	Polyethylene
PA6	Polyamide 6
PS	Polystyrene
PLA	Polylactic acid
LDPE	Low density polyethylene
EPDM	Ethylene-propylene-diene-terpolymer
EPR-g-MA	Maleate ethylene propylene rubber
EPDM-g-MA	Maleic anhydride grafted EPDM
PP-MA	Polypropylene graft maleic anhydride
PE-MA	Maleic anhydride grafted polyethylene
MMA	Methyl-methacrylate
LMDPE	Linear medium density polyethylene

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