

## Study on the influence of devulcanization conditions on the reclaim-based vulcanizates operation characteristics

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The method of experimental design has been applied to study the dependences of the operational characteristics of reclaim-based vulcanizates on the devulcanization conditions of rubber crumbs from solid and superelastic tyres – temperature, treatment time and softener quantity. Mathematical models in the form of second order polynomials, representing the dependence of the most important operation characteristics of the reclaims and the vulcanizates based thereof vs. devulcanization conditions have been obtained. Optimizing a number of predetermined goal functions according to a chosen scan method allows determination of the optimum values of the working conditions. It has been established that there are no devulcanization conditions suitable to ensure the best values for all important operation characteristics studied. That is why the devulcanization process should be run under particular conditions, tailored according to obtaining of reclaim for specific technological purposes defined preliminary.

**Keywords:**reclaim characteristics, rubber crumb, experimental design, devulcanization conditions

### INTRODUCTION

Although finely ground, disintegrated vulcanizates cause quality problems related to worsened operation characteristics of the final rubber products. From a theoretical point of view, the reason is in the thermodynamic incompatibility between the fresh rubber mesh matrix and the particles of the disintegrated vulcanizates, occurring mainly at their interface. That is why the studies on the reclaim of disintegrated worn and dispensed rubber particles aim first of all creating of effective methods for improving their compatibility with the fresh rubber mesh matrix, as well as of possibilities for inter-diffusion or interaction at the phase boundary [1]. Reclaiming is one of the reliable methods for solving the problems mentioned, as more and more companies recognize rubber reclaim as an important source for rubber processing industry first of all because of the environmental and economic benefits of recycling [2].

The technological process of devulcanization consists of reclaiming worn tyres and dispensed or/and obsolete technical rubber particles and their turning into a product with prevailing plastic properties. It is also characterized by the opportunity to compound fresh rubber and ingredients, and then revulcanize the compound. The principle of the process is described in [3]: optimal destruction of the three-dimensional vulcanization net, rupture of the rubber-carbon black bonds and then transforming the elastic vulcanizate into a plastic recyclable product [4].

In comparison to the initial vulcanizate, the structural specifics of the reclaims are connected with the effect that a part of the rubber substance in the reclaim still keeps remnants of the non-destructed curing net, while the other part though lacking cross links differs from the crude rubber by its molecular chain structure resultant from devulcanization side reactions. In fact, both features have negative effect on the reclaim quality.

During last years, many different reclaiming methods have been developed and successfully implemented [5-21]. One of them is devulcanization by thermal swelling in the presence of different agents, predominantly oil softeners [7,10]. Evidently, it is necessary to find optimal conditions for the devulcanization process, so that to achieve a maximum destruction rate of the curing net on one hand and on the other, to achieve minimization of the side reactions causing changes in the reclaim macromolecules.

This work aims at deriving polynomial mathematical models by planning a multifactor experiment via optimal experimental design and regression analysis. The models will describe the relationships between the process conditions for devulcanization *via* thermal swelling in oils and the basic rheological, vulcanization and mechanical properties of the obtained vulcanizates and reclaims.

### EXPERIMENTAL

#### *Initial vulcanizates*

Initial vulcanizates for reclaims were obtained from protector crumbs based on: 70 % of styrene-butadiene rubber (BULEX 1500, made in Bulgaria);

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20 % of isoprene rubber (SKI-3, made in Russia) and 10 % of butadiene rubber (SKD, made in Russia). The crumbs of waste vulcanizates from solid and superelastic tyres (made by Zebra Ltd., Bulgaria) were obtained on a 05-782/P3 disk grinder with two disks having 800 mm outer diameter, at a rate of 900 rpm and power of 75 kW.

Some of the most important characteristics of the crumbs used for reclaiming are summarized in Table 1. As seen, the prevailing fraction size is about 250-500 μm, approximately.

The process of devulcanizing rubber crumbs by thermal swelling in highly aromatic oil (softener) as a swelling agent, was carried out in a horizontal autoclave of 6 m length with inner diameter of 1.6 m. The temperature conditions, duration of the treatment and the amount of softener (with regard to the amount of crumbs) were considered in the scope of the optimization parameters to be included in a model, obtained by industrial data.

**Table 1.** Characteristics of the raw protector rubber crumbs

Characteristics	Values
1. Particle size, mm	Share, %
>1.6 mm	2.2
1.25 mm – 1.6 mm	0.6
1.0 mm – 1.25 mm	4.3
0.63 mm – 1.0 mm	11.8
0.50 mm – 0.63 mm	10.4
0.25 mm – 0.50 mm	52.7
0.20 mm – 0.25 mm	7.2
0.16 mm – 0.20 mm	6.6
0.10 mm – 0.16 mm	3.7
0.09 mm – 0.10 mm	0.3
< 0.09 mm	0.2
2. Acetone extract, %	12
3. Ash content, %	6.5
4. Carbon black content, %	30.2
5. Rubber content, %	49
6. Loss on heating, %	0.49
7. Specific gravity, g/cm <sup>3</sup>	1.15

The swelling agent was a highly aromatic oil type PN-6 (made in Russia), having the following characteristics: viscosity 35-40 CST (at 100 °C); density 0.96-0.98 g/cm<sup>3</sup>; flash point 230 °C; solid point 36 °C; refractive index 1.53-1.54; sulphur content 2.71-3.08 %; aniline point 55-65 °C. The hydrocarbon composition of the oil was the following: paraffinic-naphthenic fraction 8-10 %, aromatic fraction (low, medium and heavy hydrocarbons) 82-90 %, resins 7.0-8.0 %.

#### *Optimal experimental design*

The experimental design was conducted using a B<sub>m</sub> type optimal compositional plan [22, 23], containing 14 experiments for obtaining a second

order mathematical model. The model factors (process variables), i.e. the devulcanization conditions of the experiment, as well as their levels in a normal and encoded form, were set up as follows:

X<sub>1</sub> - devulcanization temperature, °C

Low level – 185 °C, encoded as /-1/;

Main level – 200 °C, encoded as /0/;

High level - 215 °C, encoded as /+1/.

X<sub>2</sub> – time for treatment (devulcanization) of the crumbs, hours

Low level – 3 h, encoded as /-1/;

Main level – 5.5 h, encoded as /0/;

High level - 8 h, encoded as /+1/.

X<sub>3</sub> - amount of the softener with regard to the crumbs amount, %

Low level – 5 %, encoded as /-1/;

Main level – 10 %, encoded as /0/;

High level - 15 %, encoded as /+1/.

The following basic characteristics of the obtained reclaims and vulcanizates were chosen for goal functions (functions of desirability) of the performed experimental design:

Y<sub>1</sub>– *Mooney* viscosity; Y<sub>2</sub> – devulcanization degree; Y<sub>3</sub> – crosslink density of the reclaim-based vulcanizates; Y<sub>4</sub> – tensile strength of the reclaim-based vulcanizates; Y<sub>5</sub> – heat aging resistance with regard to tensile strength of the reclaim-based vulcanizates.

The applied optimal compositional plan contained 14 experiments and is described by the following design matrix (Table 2):

#### *Matrix of the experimental design in normal and encoded values*

The reclaims obtained under the treatment conditions in each out of all 14 experiments (Table 2) were used for preparing identical rubber compounds according to the Bulgarian National Standard 5149-80, all having the following composition (in phr): reclaim - 100, zinc oxide – 2.5, dibenzothiazolyl disulfide – 0.9 and sulphur – 1.5.

The compounding was carried out on an open rubber two-roll mill with L/D 320×160 and friction rate of 1.27. Nominal speed of 25rpm on the slower roll was set.

The experimental samples (200×200×2mm) prepared from the examined compounds underwent vulcanization by electric heating on a hydraulic press at 143 °C, and pressure of 12MPa with time duration of 15 min.

*Test treatment of the prepared compounds and vulcanizates*

The following characteristics of the prepared compounds and the vulcanizates based thereof were further investigated:

- Vulcanization characteristics at 143 °C – according to ISO 3417:2010;
- *Mooney* viscosity, ML (1+4) at 100°C of the reclaims and the compounds based thereon (ISO 289-1:2002);
- *Shore A* hardness – according to ISO 7619:2001;
- Tensile stress-strain test properties of the vulcanizates ( $M_{100}$  and  $M_{300}$ -stress load at 100% and 300 % elongation,  $\sigma$  – tensile strength,  $\epsilon_{rel}$ - relative elongation,  $\epsilon_{res}$ - residual elongation (according to ISO 37:2008). The experimental tests were carried out at a stretching rate of 500 mm/min;
- Resistance to heat aging of the vulcanizates (at 70 °C for 72 hours) according to ISO 188:1998. The negative sign of the aging coefficient shows the worsening of the respective mechanical properties, in %;

- The equilibrium rate of swelling in xylene of the reclaims was determined by a particular laboratory method;

- The devulcanization rates of the modified crumbs (i.e. the reclaims) were evaluated as the ratio between the equilibrium swelling rate of the devulcanized samples under different conditions and the same index of the initial untreated sample.

RESULTS AND DISCUSSION

Table 3 presents the experimentally obtained *Mooney* viscosity index of the reclaim samples devulcanized under different conditions, (according to the design matrix, Table 2). As seen, the conditions of the devulcanization process influence to a sufficiently high extent the *Mooney* viscosity values. So, this index varies under the prescribed test conditions in the range of 33÷52 units ML. The experimental results have good reproducibility, as shown by the parallel runs of experiment No. 9 (corresponding values: 36, 35, 35). That issue allows assuming that by changing the devulcanization conditions, for particular needs one can obtain reclaims with preset *Mooney* viscosity levels.

**Table 2.** Matrix of the experimental design in normal and encoded values:

Experiment №	X <sub>1</sub>		X <sub>2</sub>		X <sub>3</sub>	
	Devulcanization temperature		Devulcanization time		Amount of softener	
	normal	encoded	normal	encoded	normal	encoded
1	185	-1	3	-1	5	-1
2	215	+1	3	-1	5	-1
3	185	-1	8	+1	5	-1
4	215	+1	8	+1	5	-1
5	185	-1	3	-1	15	+1
6	215	+1	3	-1	15	+1
7	185	-1	8	+1	15	+1
8	215	+1	8	+1	15	+1
9	185	-1	5.5	0	10	0
10	215	+1	5.5	0	10	0
11	200	0	3	-1	10	0
12	200	0	8	+1	10	0
13	200	0	5.5	0	5	-1
14	200	0	5.5	0	15	+1

**Table 3.** *Mooney* viscosity index of the sampled reclaims according to the experimental design matrix

Experiment №	1	2	3	4	5	6	7	8	9	10	11	12	13	14
<i>Mooney</i> viscosity, units ML (1+4) 100 °C	42	52	33	47	40	48	33	36	35	42	44	34	38	34

**Table 4.** Rate of equilibrium swelling (R of ES) in xylene and devulcanization rate (DR) of samples from modified reclaims obtained according to the experimental design matrix.

Experiment №	1	2	3	4	5	6	7	8	9	10	11	12	13	14
R of ES %	331	273	329	280	313	301	324	289	330	292	298	310	292	295
DR	1.96	1.83	2.21	1.87	2.09	2.01	2.20	1.93	2.19	1.95	1.99	2.07	1.95	1.97

3 parallel runs of experiment № 9: R of ES values-328,330,331; DR values: 2.19, 2.20, 2.19.

Untreated sample R of ES: 150

**Table 5.** Mooney viscosity index of standard rubber compounds based on reclaims modified under different conditions according to the experimental design matrix.

Experiment №	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Mooney viscosity, units ML (1+4) 100 °C	34	30	27	29	33	30	26	29	29	35	37	28	32	30

The devulcanization rate of the reclaims obtained under different conditions has been determined with regard to their equilibrium swelling rate compared to the same index of an untreated sample. The investigations aim at establishing supervision and optimization of the devulcanization process, as well as the effect its conditions have on the obtained reclaim characteristics. The experimental results are given in Table 4. It is evident, that the reclaims resulting from experiments № 3 and №7 have the highest devulcanization rate. Also, their swelling equilibrium rate reaches the highest values, hence it is the closest to the index of a fresh elastomer. It is obvious, however, that the devulcanization is only fractional in all experimental cases. The process has been completed at the least extent in the case of experiments № 2 and № 4. Certain correlation between Mooney viscosity index and the devulcanization degree has also been observed. In brief, the lower the Mooney viscosity values, the higher devulcanization degree is obtained. Obviously, the devulcanization rate and the reclaim properties are strongly dependent on the process conditions. That is why the optimum devulcanization conditions should be specified in advance and first of all, the process should be run only under the prescribed controlled conditions to minimize the undesired side effects of worsening the qualities of the reclaims obtained.

Table 5 summarizes the values of Mooney viscosity index regarding the prepared standard rubber compounds based on reclaims devulcanized under different conditions. Obviously, Mooney viscosity values of the standard rubber compounds are generally lower than those of the respective reclaims. That meets the theoretical expectations as low-molecular ingredients (without reinforcing fillers) have been added to the reclaims and the system becomes less saturated in high molecular

products. Besides, an additional thermomechanical destruction of the rubber substance in the reclaims has taken place while being compounded with ingredients on the roller mill.

As seen from the table, Mooney viscosity values vary in the range of 26÷37 units. The highest values are obtained for compound № 11, and the lowest ones - for compound № 7. Having in mind that all other process conditions are fixed at the same levels, that means the variations observed are only due to the different reclaim properties, resulting from the devulcanization conditions. Reproducibility of the experimental results is good, as the parallel runs of experiment No. 9 show (corresponding values: 29, 30, 29).

The vulcanization characteristics of the prepared standard rubber compounds, based on reclaims, devulcanized under different conditions according to the experimental plan matrix are represented in Table 6. As shown, there is a significant difference in the vulcanization characteristics of the rubber compounds due to the devulcanization conditions.

The minimum torque ( $M_L$ ), which correlates significantly with the effective viscosity of the compound prior to vulcanization, possesses values between 8 and 15 dNm, while the maximum torque ( $M_H$ ), characterizing the vulcanizates hardness, varies in the range between 30-54 dNm.

The crosslink density of the curing net, which could be evaluated by  $\Delta M$  values, varies between 32 and 40 dNm. Compound № 8 demonstrates the highest value, while compounds № 2 and 6 have the lowest ones.

The vulcanization scorch time is relatively uniform and for most of the compounds it is between 3.9 and 5.8 minutes. Compound № 2 has the longest scorch time, while compound № 4 - the shortest one.

**Table 6.** Vulcanization characteristics of standard rubber compounds obtained from reclaim devulcanized under different conditions.

Characteristic Compound code	M <sub>L</sub> dNm	M <sub>H</sub> dNm	ΔM MH-ML dNm	t <sub>s</sub> min	t <sub>90</sub> min
1	12	46	34	5.8	12.0
2	15	54	39	4.4	11.1
3	9	41	32	5.0	11.1
4	11	49	38	3.9	12.1
5	10	48	38	4.4	10.9
6	13	53	40	4.3	10.8
7	9	43	34	4.8	12.0
8	8	30	22	4.4	8.9
9	9	44	35	4.8	12.0
10	11	46	35	4.4	10.3
11	12	47	36	4.6	10.9
12	9	44	35	4.6	11.6
13	10	46	36	4.4	10.9
14	9	47	38	4.0	10.1
3 parallel runs of experiment №9	9,9,9	44,44,5,45	35,35,36	4.8,4.7,4.8	11.8,12.0,11.9

**Table 7.** Mechanical properties of vulcanizates obtained from modified reclaims under different conditions

Experiment №	M <sub>100</sub> MPa	M <sub>300</sub> MPa	σ MPa	ε <sub>rel.</sub> %	ε <sub>res.</sub> %	Shore A hardness, rel. units	Aging coefficient Kσ, %
1	1.64	-	5.66	238	7	56	-12.7
2	1.69	-	5.54	220	6	58	-8.1
3	1.68	-	4.85	237	8	55	4.3
4	1.47	-	4.81	228	8	56	10.2
5	1.69	-	5.73	227	8	57	-5.2
6	1.69	-	5.83	222	6	57	-7.7
7	1.51	-	4.21	216	4	55	5.5
8	1.57	-	5.27	220	7	56	-5.3
9	1.66	-	4.92	222	5	58	-6.7
10	1.58	-	5.69	235	7	55	-0.7
11	1.72	-	5.52	228	5	60	-15.0
12	1.50	-	4.60	229	5	55	-5.6
13	1.58	-	4.95	235	5	58	-10.9
14	1.52	-	4.33	241	8	57	0.5
3 parallel runs of experiment № 9	1.65; 1.66; 1.65	-	4.87; 4.95; 4.92	220; 225; 220	5; 5; 5	59; 58; 59	-6.7; -6.9; -6.7

The optimum vulcanization time (t<sub>90</sub>) does not vary significantly. For most compounds it remains in the limits of 11-12 min, with an exception of that for compounds №№ 8, 10 and 14, which is about 9-10 min. The main mechanical properties of the vulcanizates obtained from reclaim devulcanization under different conditions according to the experimental design matrix are presented in Table 7. The values for M<sub>100</sub> are close for all experimental cases investigated. The difference between the minimum and maximum values is about 10%. The

compounds based on reclaims №№ 6, 5, 1 and 10 possess the highest tensile strength values (in the range between 5.6-5.8 MPa), while compound № 7 reaches the lowest one (4.2 MPa). The highest tensile strength value for sample №6 correlates significantly with the highest crosslink density of the curing net (Table 6). The relative elongation of the particular compounds is almost the same. The residual elongation in all the cases considered is negligible. The values of Shore A hardness index for

each vulcanizate are also close, varying in the range of 55÷60 relative units.

The thermal aging resistance of the vulcanizates obtained from devulcanized reclaims under various conditions is different. The reason is in the various conditions under which the crude crumbs have been devulcanized: the different rate of devulcanization process and the side reactions, which worsen the reclaim quality, respectively. Best aging resistance properties with regard to all parameters studied ( $M_{100}$ , tensile strength and relative elongation) demonstrate compounds № 4 and № 10.

Having carried out the experimental plan in the design matrix, the results achieved allow deriving the following regression models in the form of second order polynomials, representing the existing dependences of the goal functions  $Y_1$ – $Y_5$  on the devulcanization conditions, including the factors:

$X_1$ -devulcanization temperature, °C;

$X_2$ -devulcanization time, h,

and  $X_3$ -softener quantity, %:

1) Mooney viscosity  $Y_1$  for the reclaims obtained:

$$Y_1 = 36.90 + 3.50[X_1] - 5.60[X_2] - 1.40[X_3] + 1.60[X_1]^2 + 2.10[X_2]^2 - 0.90[X_3]^2 - [X_1][X_2]$$

2) Devulcanization rate  $Y_2$ :

$$Y_2 = 2.00 - 0.12[X_1] + 0.07[X_1]^2 + 0.02[X_2]^2 - 0.05[X_3]^2 - 0.03[X_1][X_3] + 0.04[X_2][X_3]$$

3) Crosslink density of the reclaims based vulcanizates  $Y_3$ :

$$Y_3 = 38.20 + 3.00[X_1] + 2.20[X_3] - 3.25[X_1]^2 - 2.75[X_2]^2 - 1.25[X_3]^2 - 5.25[X_1][X_2] - 6.25[X_1][X_3] - 6.00[X_2][X_3]$$

4) Tensile strength of the reclaims based vulcanizates  $Y_4$

$$Y_4 = 4.51 + 0.10[X_1]^2 + 0.54[X_2]^2 + 0.13[X_3]^2$$

5) Aging coefficient with regard to tensile strength of the reclaims based vulcanizates  $Y_5$ :

$$Y_5 = -8.58 + 0.33[X_1] + 5.85[X_2] + 0.34[X_3] + 5.18[X_1]^2 - 1.72[X_2]^2 + 2.83[X_3]^2 - 0.79[X_1][X_2] - 3.06[X_1][X_3] - 2.86[X_2][X_3]$$

Having tested the adequacy of the obtained mathematical models  $Y_1$ – $Y_5$  (optional goal functions) by the Fisher criterion (level of significance 0.02) and considering the affirmative results, the models have been subjected to an optimization procedure. The optimal coordinates of the global extrema to all goal functions have been determined implementing standard scan optimization methods. The values for the factors concerning devulcanization conditions have been obtained in the factorial space studied, respectively.

The final results obtained are the following:

1) For the relation Mooney viscosity vs. devulcanization conditions - the lowest Mooney viscosity ( $Y_1^{MAX}=32$  relative units) has been

obtained under the following devulcanization conditions:

- devulcanization temperature 183°C;

- devulcanization time 8 h;

- softener quantity 5 % of the crumbs amount.

Comparing the results from Tables 2 and 3 and those derived by the optimization procedure one can consider that in the case of industrial experiment № 3 from the design matrix, the applied devulcanization conditions for waste vulcanizates are very close to the extremum coordinates, found by the numerical optimization software utility.

2) For the relation reclaim devulcanization rate  $Y_2$  vs. devulcanization conditions – the highest devulcanization rate ( $Y_2=2.25$ ) is obtained at the following factor values:

- devulcanization temperature 175 °C;

- devulcanization time 8 h;

- softener quantity 8.5 % of the crumbs amount.

Reclaims № 3и № 7 demonstrate the highest devulcanization rate achieved experimentally: 2.21 and 2.20, respectively (Table 4). These values deviate by about 2-3% from those predicted by the derived model.

3) For the relation  $\Delta M$  (crosslink density of the reclaim-based vulcanizates) vs. reclaiming conditions in the form of highest vulcanizate crosslink density  $Y_3$ ,  $Y_3=41$  dNm has been obtained under the following devulcanization conditions:

- devulcanization temperature 215 °C;

- devulcanization time 21 min;

- softener quantity 5 % of the crumbs amount.

A practical experimental point with these coordinates does not exist in the generated experimental design matrix. The highest  $\Delta M$  value, achieved experimentally for a compound based on reclaims № 6, is  $\Delta M=40$  dNm (Table 6).

4) For the relation tensile strength of the reclaim-based vulcanizates vs. the reclaiming conditions  $Y_4$ , the highest value of the tensile strength of the reclaim-based vulcanizates  $Y_4=6.15$  MPa has been measured at the following factor values:

- devulcanization temperature 215 °C;

- devulcanization time 3h;

- softener quantity 15 % of the crumbs amount.

A practical experimental point with these coordinates does really exist in the experimental design matrix as experiment № 6. The experimentally obtained highest tensile strength value is 5.83 MPa (Table 7). It differs just by 5 % from the value predicted by the mathematical model.

5) For the relation aging coefficient (with regard to tensile strength of the reclaim-based vulcanizates) vs. reclaiming conditions  $Y_5$ , the highest value of the aging coefficient with regard to tensile strength of

the reclaim-based vulcanizates  $Y_5=10.9\%$  has been determined at the following factor values:

- devulcanization temperature 215 °C;
- devulcanization time 8 h;
- softener quantity 15 % of the crumbs amount.

There is such an experimental point in the experimental design matrix as experiment № 4. The aging coefficient value with regard to the tensile strength determined experimentally is 10.2 %. It differs just by 6 % from the one predicted by the derived mathematical model.

The analysis of the results obtained reveals that the optimal conditions for performing devulcanization of waste vulcanizates are considerably differing for each of the 5 goal functions, i.e. devulcanization conditions ensuring the most appropriate values for all important parameters simultaneously could not be found. That infers that the optimal devulcanization conditions should be chosen depending on the future reclaim application, keeping in mind the following issues:

- The lowest value of the *Mooney* viscosity index, the highest degree of devulcanization of the crumbs, respectively, is achieved at low temperature (175-185 °C) but for the longest treatment time (8 h) possible and at an average quantity of softener (8.5%). Evidently, the lower temperature and longer treatment time allow the softener to facilitate the highest rate of intermacromolecular diffusion and scission more bonds.

- In the case of longer treatment, undesired side destructive processes occur simultaneously with the devulcanization of waste vulcanizates. Therefore, if aiming at obtaining reclaim-based vulcanizates of maximum tensile strength, the reclaiming conditions should be intensified so that to minimize the undesired destructive processes yielding lower molecular mass and worsened mechanical parameters, respectively. The mathematical model prompts the same, namely: best mechanical properties have the vulcanizates based on reclaims devulcanized at higher temperature (215 °C) and maximum quantity of the softener – 15%. All these conditions lead to intensified vulcanization treatment since the conditions benefit the diffusion of the molecules of the softening agent. However, modification of the compound should proceed for the shortest time (3 h) in order to maximally eliminate the destruction processes. Assuming that first priority is to obtain reclaim-based vulcanizates with best mechanical properties, the reclaiming conditions should be set at the values shown above.

## CONCLUSIONS

1) The dependences of the exploitation characteristics of obtaining reclaim-based vulcanizates on the conditions of reclaiming crumbs from solid and superelastic tyres – temperature, treatment time and softener quantity, have been studied by the method of experimental design.

2) Mathematical models in the form of second order polynomials representing the relations between the most important operation characteristics of the reclaims and the vulcanizates based thereof vs. devulcanization conditions have been derived. The extremum values of the processing conditions have been determined by optimization procedures implementing standard scan search routines.

3) It has been established that there are no practical devulcanization conditions ensuring the best values for all most important exploitation characteristics studied. That is why devulcanization should be conducted under conditions of compromise, tailored according to the specific future application of the reclaim obtained:

- the lowest *Mooney* viscosity value of the reclaims has been achieved at 183 °C for the longest treatment time (8 h) and at a minimum quantity of softener (5%);

- the highest devulcanization rate of the crumbs has been achieved at 175 °C for the longest treatment time (8 h) and at average quantity of softener (8.5%);

- best mechanical properties possess the vulcanizates based on reclaims devulcanized at 215 °C, for the shortest treatment time (3 h) and at a maximum quantity of the softener – 15%, in order to maximally eliminate the destruction processes;

- the highest aging resistance coefficient with regard to the tensile strength is obtained for the vulcanizates based on reclaims, devulcanized at 215 °C, for the longest treatment time and at a minimum quantity of the softener – 5%.

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## ИЗСЛЕДВАНЕ ВЛИЯНИЕТО НА УСЛОВИЯТА НА ДЕВУЛКАНИЗАЦИЯ ВЪРХУ ЕКСПЛОАТАЦИОННИТЕ ХАРАКТЕРИСТИКИ НА ВУЛКАНИЗАТИ НА БАЗАТА НА РЕГЕНЕРАТ

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(Резюме)

Чрез прилагане на метода на планирания експеримент бяха изследвани зависимостите на експлоатационните характеристики на вулканизати на базата на регенерат от условията на девулканизация на каучуково брашно от плътни и супереластични гуми - температура, време на третиране и количество на омекчителя. Получени бяха математични модели под формата на полиноми от втора степен, представящи зависимостта на най-важните експлоатационни характеристики на регенерата и базираните на него вулканизати от условията на девулканизация. Оптимизирайки значителен брой предвалително определени целеви функции по метода на сканирането бяха определени оптималните стойности на условията на девулканизация. Установено беше, че няма условия на девулканизация, които да осигуряват най-добрите стойности едновременно на всички изследвани важни експлоатационни характеристики. Ето защо процесът на девулканизация трябва да бъде провеждан при условия, дефинирани предварително с оглед получаването на регенерат за специфични технологични цели.