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Research Article

SOME STUDIES ON INVESTIGATING THE CONCEPT OF OPTIMIZING VULCANIZATION PROCESS OF RUBBER FOR EFFECTIVE MECHANICAL PROPERTIES USING TAGUCHI METHOD: AN EXPERIMENTAL APPROACH

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ABSTRACT

The un-vulcanized natural rubber is sticky, deforms quickly on heating and its brittleness increases on cooling. Under this state, it is not suitable for many applications. In the present work, the performance of vulcanization of rubber has been evaluated in order to improve mechanical properties of the rubber. The experiments were performed as per Taguchi's mixed level parameter design (L18). The vulcanization load, vulcanization temperature curing time and rubber ratio were considered as process parameters. The mechanical properties viz. rubber strength and hardness were selected as response parameters. The results reveal that the best settings for maximum tensile strength is Curing time 22 mins, NR:PBR is 60%:40%, vulcanization Temperature is 130oC and vulcanization load is 3.5 Tons. Similarly the best setting for max hardness is Curing time 26 mins, NR:PBR is 100%:0%, vulcanization Temperature is 130oC and vulcanization load is 3.5 Tons

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INTRODUCTION

The unvulcanized natural rubber is sticky, easily deforms when warm, and is brittle when cold. In this state it cannot be used to make articles with a good level of elasticity. The reason for inelastic deformation of unvulcanized rubber is due to its chemical structure: rubber is composed of long polymer chains. These chains can move independently relative to each other, which enables the material to change shape. Vulcanization is a chemical process that introduces curatives like sulphur in the natural rubber like polymers in order to increase its durability. These curatives form bridges (crosslinks) between individual polymer chains and prevent the polymer chains from moving independently. The vulcanized material is less sticky and has superior mechanical properties. The crosslinking introduced by vulcanization. As a result, when stress is applied the vulcanized rubber will deform, but upon release of the stress, the article will revert to its original shape. Depending upon the types of curative used for the formation of cross-linking, the vulcanization process may be classified as sulphur vulcanization, peroxides vulcanization, urethane crosslinkers vulcanization, metallic oxides vulcanization, duece vulcanization or acetoxysilane vulcanization. Out of the above,

sulphur vulcanization is generally used. Sulphur is a slow reaction agent. So various types of accelerators, antigradents, activators and inhibitors are used to increase the rate of reaction and to control the process.

Depending upon the sulphur/accelerators ratio, the sulphur vulcanization process can be classified in three categories viz. Conventional, Semi-Efficient and Efficient Vulcanization as shown in Table 1.

Table 1 Levels of accelerators and sulphur in EV, SEV and CV

Type of System	Sulphur (phr)	Accelerator (phr)	Sulphur/Accelerator ratio
EV	0.4-0.8	6.0-2.0	2.5-12
SEV	1.0-1.7	2.4-1.2	0.7-2.5
CV	2.0-3.5	1.2-0.4	0.1-0.6

In the conventional vulcanization systems, the sulphur level is high and correspondingly the accelerator level is low whereas in efficient vulcanization systems, low level of sulphur and a correspondingly high level of accelerators are employed in vulcanisates for which an extremely high heat and reversion resistance is required. In semi efficient vulcanization systems, the intermediate level of accelerator and sulphur are employed.

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The CV systems provide better flex and dynamic properties but worse thermal and reversion resistance. For optimum levels of mechanical and dynamic properties of vulcanisates with intermediate heat, reversion, flex and dynamic properties, SEV system is used. EV system gives products with sulphur interlinks of shorter length and the products have improved resilience but lower strength.

A raw elastomer in its natural condition is a high molecular weight liquid with low strength. Although its chains are tangled, they can readily untangle upon stressing, giving rise to fracture. Vulcanization or curing is the process in which the chains are chemically coupled together to form a network. The most widely used vulcanizing agent is sulphur. Beside this, the several additives are used to control the process viz. Vulcanization accelerators (guanidines, thiazoles, dithiocarbamates, xanthates and thiurams), Activators (zinc oxide and stearic acid), Vulcanization inhibitors, Chemical/Physical plasticizers, Antidegradants (amines, phenolics and phosphites) Reinforcing fillers (carbon black and silica) Softeners (mineral oils. Animal and vegetable oils) Extenders (clay, calcium carbonate and ground coa) Tackifiers rosin derivatives, coumarone-indene resins, aliphatic petroleum resins, alkyl-modified phenol-formaldehyde resins)

LITERATURE REVIEW

Sulphur vulcanization is one of the important classes of engineering materials discovered by Charles Goodyear in 1839 and independently by Thomas Hancock, who patented the process in the UK on November 21, 1843. A vigorous research effort continues today to develop a fundamental understanding of this complex chemical process. Many researchers have been reported their research work in this field. Loo *et al.* [1] investigated the influence of curing temperature on chemical crosslink density, the distribution of crosslink types, the extent of sulphidic main-chain modifications, and the zinc sulphide formation in natural rubber (NR) gum mix with a conventional N-cyclohexyl-2-benzothiazylsulphenamide (CBS) accelerated sulphur system. The results indicated that elevated cure temperatures produce a network with a lower crosslink density, in particular a lower polysulphidic crosslink density. Furthermore, the formation of intramolecular sulphidic groups and zinc sulphide increases with increasing temperatures. Mukhopadhyay *et al.* [2] investigated the effect of vulcanization temperature (150°–180°C) on the structure and technical properties of natural rubber vulcanizates with four different accelerator: sulphur ratios (A - 0.6:2.4, B-1.5:1.5; C - 2.4:0.6; D-3.0:0.0) at the respective optimum cure times. The influence of cure temperature on the chemical crosslink density was determined. Results show that elevated cure temperatures produce a network with lower crosslink density. The formation of intramolecular sulphidic groups and zinc sulphide increases with increasing cure temperatures. Ingham *et al.* [3] investigated the effect of soluble zinc mercapto-benzothiazolate (ZMBT) and the derived insoluble basic zinc salt in an efficient CBS vulcanization (EV). Results illustrated that maximum oxidative stability is developed at optimum cure time. Mukhopadhyay *et al.*[4] investigated the effect of an increase in vulcanization temperature from 150° to 180°C on the network structure and technical properties of gum natural rubber Results indicated that the combination with MDB (1.2

phr) TMTD (0.6 phr) showed the best synergism of the two accelerators. Kok [5] examined the effect of compounding ingredients on the reversion of accelerated sulphur vulcanization of natural rubber (NR). Accelerators which normally provide fast cures are found to cause more severe reversion than slower accelerators. MgCO₃, CaCO₃, silica and China clay all increase reversion but the severity depends on the accelerated system employed.

Jeon *et al.* [6] investigated the effect of adding silica to rubber compound on its adhesion to brass-plated steel cord. The result shows that silica reduced the curing rate and enhanced the physical properties of the rubber compound, especially after thermal aging, when 20 phr of silica was added. Jayasuriya *et al.* [7] evaluated the effect of trimethylol propane trimethacrylate (TMPTMA) and phenoxy ethyl acrylate (PEA) as radiation vulcanization accelerator on radiation vulcanization of natural rubber latex (RVNRL). The Results illustrated that the PEA is a more effective accelerator for RVNRL since it imparts approximately equivalent physical properties compared to that of n-butyl acrylate (*n*-BA), the most widely used accelerator. Jinhua *et al.* [8] investigated the sensitizing efficiency of several polyfunctional monomers to radiation vulcanization of ethylene-propylene rubber. The results show that triethyleneglycol dimethacrylate (TEGDMA) gave the best results. Ravichandran and Natchimuthu [9] evaluated the effect of leather and scrap rubber loading on vulcanization characteristics of natural rubber compounds. Results indicated that the presence of leather was found to reduce the scorch time and increase the maximum and minimum torque. Punnarak *et al.* [10] investigated the effect of blend ratio, methods of vulcanization, i.e. sulphur, peroxide, and mixed system and the addition of compatibilizer on mechanical, thermal, and rheological properties was investigated. The blend with highest impact strength was obtained from 50/50 RTR/HDPE vulcanized by sulphur. Increasing the RTR content to more than 50% resulted in a decrease in the impact strength of blend, most likely due to the increasing carbon black content. For tensile strength, the presence of rubber and carbon black, however, unavoidably caused a drop in this property. Comparing among three methods of vulcanization, sulphur system seems to be the most effective method. Rattanasom *et al.* [11] investigated the effect of a sulphur vulcanization system, i.e. conventional vulcanization and efficient vulcanization on the mechanical properties and heat aging resistance of natural rubber (NR)/ tyre tread reclaimed rubber (RR) blends. The results reveal that the blends in both curing systems show an increase in their hardness and modulus with increasing RR content while other mechanical properties are adversely affected. Osabohien *et al.* [12] investigated the cure characteristics and the physico-mechanical properties of natural rubber, standard Nigerian rubber, SNR10 filled with cherry seed shell (CSS) and standard carbon black CB (N330) were determined. The results showed that the scorch, cure times and the maximum torque gradually increased with increasing filler content for CSS-filled natural rubber moreover in case of CB-filled natural rubber, the scorch and the cure times decreased while the maximum torque increased with increasing filler content. Qiuying and Yulu [13] investigated the influence of the basic properties of carbon black on the vulcanization and mechanical properties of filled

natural rubber compounds. Results indicated that the effect of carbon black surface activity, which was changed by introducing one kind of hindered amine light stabilizer on rubber reinforcement, is emphasized. The literature review reveals that lot of work has been reported on optimization of various vulcanization processes of rubber by changing various additives such as reinforcing agents (carbon black, cherry seed shell, silica, leather, reclaimed rubber etc), accelerators and activators (ZnO, Stearic acid etc). The optimization of the process parameters of the rubber is adversely effected by the various additives used in the rubber processing and various vulcanization parameters. Thus the optimization of the process parameters of the rubber has a great potential for future research.

Experimentation

The experiments were performed on the vulcanization of rubber used in various applications. In the present research work, the four factors viz. curing time, rubber ratio, vulcanization temperature and vulcanization load were selected based upon the previous literature and pilot study as shown in table 1.

Table 2 Process Parameters and their levels

Factors	L-1	L-2	L-3	L-4	L-5	L-6
Curing Time (mins)	6	10	14	18	22	26
Rubber Ratio (NR:PBR)	100:1	60:40	20:80	-	-	-
Vulcanization Temperature (oC)	110	120	130	-	-	-
Vulcanization Load (Ton)	2.5	3	3.5	-	-	-

The experiments were performed according to Taguchi mixed level L18 OA. The Table 2 shows control log of experimentation as per Taguchi L18 OA. The strength was calculated using tensile testing machine and hardness was measured by the Shore (Durometer) test.

Table 2 Control log of experiments as per L18 OA

S. No.	Curing Time (mins)	Rubber Ratio (NR:PBR)	Vulcanization Temperature (oC)	Vulcanization Load (Ton)
1	6	100%:0%	110	15
2	6	60%:40%	120	20
3	6	20%:80%	130	30
4	10	100%:0%	110	20
5	10	60%:40%	120	30
6	10	20%:80%	130	15
7	14	100%:0%	120	15
8	14	60%:40%	130	20
9	14	20%:80%	110	30
10	18	100%:0%	130	30
11	18	60%:40%	110	15
12	18	20%:80%	120	20
13	22	100%:0%	120	30
14	22	60%:40%	130	15
15	22	20%:80%	110	20
16	26	100%:0%	130	20
17	26	60%:40%	110	30
18	26	20%:80%	120	15

Based upon Table 2, experiments were conducted and output has been recorded for tensile strength and hardness as shown in table 03-08.

RESULTS AND DISCUSSION

Table 3 shows variation of S/N ratio and tensile strength, different process parameters). The results may be explained on the basis of the fact that natural rubber has high tensile strength

(because tensile strength depends upon the elastomer’s ability to partly strain crystallize when stretched and the natural rubber is an elastomers with a very regular chain structure that strain crystallizes).

Table 3 Observation of final experimentation and S/N ratio for Tensile strength

S. No.	Curing Time (mins)	Rubber Ratio (NR:PBR)	Vulcanization Temperature (oC)	Vulcanization Load (Ton)	Tensile Strength MPa	S/N ratio
1	6	100%:0%	110	15	11.20	20.9844
2	6	60%:40%	120	20	12.50	21.9382
3	6	20%:80%	130	30	13.40	22.5421
4	10	100%:0%	110	20	12.50	21.9382
5	10	60%:40%	120	30	10.32	20.2736
6	10	20%:80%	130	15	11.43	21.1609
7	14	100%:0%	120	15	11.90	21.5109
8	14	60%:40%	130	20	15.30	23.6938
9	14	20%:80%	110	30	13.60	22.6708
10	18	100%:0%	130	30	13.40	22.5421
11	18	60%:40%	110	15	10.50	20.4238
12	18	20%:80%	120	20	13.20	22.4115
13	22	100%:0%	120	30	13.40	22.5421
14	22	60%:40%	130	15	11.50	21.2140
15	22	20%:80%	110	20	12.00	21.5836
16	26	100%:0%	130	20	15.30	23.6938
17	26	60%:40%	110	30	18.20	25.2014
18	26	20%:80%	120	15	13.40	22.5421

Table 4 Observation of final experimentation and S/N ratio for Hardness

S. No.	Curing Time (mins)	Rubber Ratio (NR:PBR)	Vulcanization Temperature (oC)	Vulcanization Load (Ton)	Tensile Strength MPa	S/N ratio
1	6	100%:0%	110	15	61	35.7066
2	6	60%:40%	120	20	64	36.1236
3	6	20%:80%	130	30	77	37.7298
4	10	100%:0%	110	20	69	36.7770
5	10	60%:40%	120	30	75	37.5012
6	10	20%:80%	130	15	61	35.7066
7	14	100%:0%	120	15	62	35.8478
8	14	60%:40%	130	20	71	37.0252
9	14	20%:80%	110	30	70	36.9020
10	18	100%:0%	130	30	75	37.5012
11	18	60%:40%	110	15	61	35.7066
12	18	20%:80%	120	20	70	36.9020
13	22	100%:0%	120	30	74	37.3846
14	22	60%:40%	130	15	65	36.2583
15	22	20%:80%	110	20	68	36.6502
16	26	100%:0%	130	20	72	37.1466
17	26	60%:40%	110	30	73	37.2665
18	26	20%:80%	120	15	62	35.8478

The temporary nature of strain crystallization allows natural rubber to regain its original shape once the stress is removed. An elastomer with naturally poor tensile strength can be enhanced through the addition of reinforcing agents such as silica and carbon black. The majority of active applications will require an elastomeric compound with tensile strength of at least 6.9 MPa. Elongation is inversely proportional to hardness, tensile strength, and modulus. It takes more force to stretch a hard material having high tensile strength and high modulus than to stretch a soft material with low tensile strength and low modulus

The table No 04 illustrated that vulcanization temperature is the most significant parameter for maximum tensile strength rubber ratio and curing time are insignificant. Furthermore, table 05 illustrated vulcanization temperature ranked as one followed by rubber ratio and curing time. The confirmatory

experiments were conducted and result of study shows 3.12% improvement in tensile strength at optimized settings suggested by Taguchi design. These results are valid for 95% confidence level.

CONCLUSION

The results indicated that vulcanization temperature is the most significant parameter for high tensile strength and vulcanization load for high hardness. On the basis of experimental observations made on vulcanization of rubber it has been concluded that the best settings for maximum tensile strength is Curing time 22 mins, NR:PBR is 60%:40%, vulcanization Temperature is 130oC and vulcanization load is 3.5 Tons. Similarly the best setting for max hardness is Curing time 26 mins, NR:PBR is 100%:0%, vulcanization Temperature is 130oC and vulcanization load is 3.5 Tons. The results are in line with the observations made by other investigators [13, 14]. The confirmatory experiments has been conducted and found 3.5% and 4.3% improvements in tensile strength and hardness respectively.

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