

Low Temperature Vulcanisation: Technique for CR-BIIR Blend for Encapsulation of Oceanic Sensors

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ABSTRACT

Blend of chloroprene rubber (CR) and bromobutyl rubber (BIIR) is used for encapsulation of piezo sensors used in sea water. Conventional encapsulation method of these sensors involving high temperature vulcanisation (HTV) often leads to deterioration of piezo properties due to thermal degradation. This paper reports a low temperature vulcanisation (LTV) technique carried out at 90 °C for CR-BIIR blend using chlorinated polyethylene (CPE) as compatibiliser and LTV system consisting of modified di-o-tolyl guanidine and thiocarbonyl as accelerators and ZnO as curing agent. The conventionally used scavenger MgO was eliminated and only ZnO was used to boost the cure reaction. Properties specific to sonar sensors, namely, acoustic transparency, electrical resistivity, water absorption and physico-mechanical properties were evaluated besides evaluation of morphology. The results are found to compare better than the conventional blend. Accelerated thermal ageing at 70 °C for 7 days yielded 97 % retention of tensile strength. The technique was implemented in a PZT hydrophone sensor and was successfully underwater tested.

Keywords: Chloroprene rubber; Bromobutyl rubber; Sonar sensor; Low temperature vulcanisation

NOMENCLATURE

μm	Micrometre
E'	Storage modulus
E''	Loss modulus
T_g	Glass transition temperature
$\text{Tan } \delta$	Damping factor
μPa	Micro Pascal

1. INTRODUCTION

Vulcanisation of rubber is conventionally carried out at higher temperatures of 150 °C and above. However, in underwater sensor encapsulation process, the vulcanisation at higher temperature has detrimental effect on sensor material due to thermal degradation. This is more prevalent in the fabrication of acoustic sensors using polyvinylidene fluoride (PVDF) film and thin walled piezo ceramics. In addition to high energy consumption, the high temperature vulcanisation (HTV) affects the quality and longevity of rubber products¹. HTV is reported to affect the sensitivity and reliability of the sensor². As the cure temperature increases, the efficiency of the cross linking reaction is reduced and results in lower crosslink density³. Low temperature vulcanisation (LTV) offers rubber products with optimum properties, fine finish and appearance^{4,5}. Palaty, Shiny and Joseph, Rani¹ reported the advantages of LTV.

Rubbers which have shortcomings in one or more properties can be compensated and an optimum performance

can be obtained by blending of different rubbers⁶. Thus, a blend of different rubbers can offer a set of properties, which are not possible with either of the individual polymers⁷. Blend of chloroprene rubber (CR) and bromobutyl rubber (BIIR) is an attractive blend owing to the inherent characteristics of the individual rubber. CR is a polymer with a wide spectrum of properties like resistance to oxidative ageing and flex cracking, excellent ozone resistance, oil resistance and low damping characteristics⁸. BIIR is resistant to weather and chemicals, has low gas and moisture permeability, high energy absorption and is co-vulcanisable with other diene rubbers⁸⁻¹⁰.

There are many functions that rubber is expected to exhibit when it is used as an encapsulant material in oceanic electro-acoustic sensors. Encapsulation provides protection against harsh sea water environment by acting as an insulating material and function as an acoustic compliant material by allowing the acoustic waves to pass through it without significant attenuation¹¹. Chlorinated polyethylene (CPE) has been reported as a compatibiliser for CR and BIIR blend in conventional HTV¹².

It is advantageous, if both the individual rubbers of a blend are vulcanised by a single cross linking agent¹³. Metal oxides are commonly used for curing CR and considered as an essential part of curing system^{7,14,15}. Halogenated butyl rubber responds to cure system used for CR as well as butyl rubber¹⁴. CR is conventionally cured with ZnO/MgO combination for various reasons¹⁵.

Although CR-BIIR blend is researched extensively, LTV system for this blend specific to underwater oceanic application

is not reported much. Therefore, the objective of the proposed study is to develop an encapsulant material using CR–BIIR blend with 80:20 ratio using a LTV process at 90 °C. The 80:20 blend ratio of CR and BIIR was optimised in an earlier study¹⁶. Here, a novel cure system consists of modified guanidine and thiocarbonyl accelerators and ZnO curing agent was used. The conventionally used scavenger MgO is eliminated as MgO deactivates ZnCl₂, the cross linking catalyst formed during the curing process. Properties of CR, BIIR and its blend are studied, specific to sonar applications, namely, acoustic, electrical resistivity, water absorption and physico-mechanical properties. The technique was implemented in a PZT hydrophone sensor and was successfully tested in an underwater environment.

2. EXPERIMENTAL

2.1 Materials and Methods

The composition of the CR–BIIR blend in the ratio 80:20, without the curing system used is the same as used in the previous study by the same authors¹⁸. The materials used in the compositions are CR (Neoprene W, M/s. DuPont Dow Elastomers, USA), BIIR (Polysar X2, M/s. Polysar Ltd. USA), CPE, which is used as compatibiliser (CM 0136, M/s. DuPont Dow Elastomers, USA), Stearic acid (M/s. Godrej Soaps Pvt. Ltd., Mumbai), which acts as activator along with zinc oxide, Vulkanox HS and Vulkanox 4020 (M/s. Bayer India), both are used as antidegradants, Carbon black and Talc, the commercial grade are the materials used as fillers for the blend. The curing agent used is commercial grade zinc oxide. The accelerators used are Zinc diethyldithio carbamate (ZDEC) (M/s. ICI India Ltd.) thiocarbonyl (TC) and modified di-*o*-tolylguanidine (MDOTG) (supplied by M/s. Golde Chem, Mumbai, India).

2.2 Preparation of Rubber Blend

A master batch of CR–BIIR blend in 80:20 ratio was prepared in a standard laboratory size two roll mixing mill using the formulation as given in Table 1. The mill temperature was maintained at 50 °C ±5 °C. Subsequently, CR–BIIR blends with different combination of curing system were prepared and designated as Z-0, Z-1, Z-2, Z-3 and Z-4 as per the details given in Table 1.

2.3 Cure Characteristics

The cure characteristics of the blends were evaluated using Moving Die Rheometer (Model MDR 2000) M/s Alpha Technologies Services Inc., USA, as per ASTM D 5289¹⁷. The parameters evaluated are minimum torque (ML), maximum torque (MH), scorch time (t_{s2}) and the optimum cure time (t_{90}). The maximum rheometer torque (MH–ML) is also estimated which indicates the ultimate extent of cure. Details of cure systems and their kinetics were reported by Mohanadas¹⁸, *et al.* The cure parameters were calculated on the basis of a 60 min run time as reported by Das¹⁹, *et al.*

2.4 Vulcanisate Properties

Tensile slabs of size 150 mm × 150 mm × 2 mm, were moulded at 90 °C for the respective cure time. CR–BIIR blend compound with cure system Z-3 was used for

Table 1. CR–BIIR blend with different cure system combination

CR–BIIR blend	Cure system	Phr
Z-0	ZnO	3.0
Z-1	ZnO	3.0
	MDOTG	1.0
Z-2	ZnO	3.0
	MDOTG	1.0
	ZDEC	1.0
Z-3	ZnO	3.0
	MDOTG	1.0
	TC	1.0
Z-4	ZnO	3.0
	MDOTG	1.0
	ZDEC	0.5
	TC	0.5

the detailed study. The dumbbell and angle test specimens were punched out from the slabs and subjected to tensile and tear tests using a Zwick Model 1476 UTM. Specific gravity was determined in an Electronic Densimeter, Mirage, MD 200S. Hardness was determined using a Wallace digital Shore-A Durometer, Cogenix, H 17A. Water absorption characteristics were determined as per ASTM D471 in 3.5 % NaCl solution at 30 °C for 24 h. Electrical properties were determined as per ASTM D257. Acoustic echo reduction test was conducted by Pulse Tube method. Dynamic mechanical properties were carried out using DMA (Q 800 Model, TA Instruments, USA) from –80 °C to 30 °C, 3 °C/min ramp, 1 Hz, single cantilever, 15 μm amplitude. The phase morphology of specimens was studied in a JEOL-EO model JSM 6390 Scanning Electron Microscope (SEM).

3. RESULTS AND DISCUSSION

3.1 Cure Characterisation of LTV System

The cure parameters of CR–BIIR blends with different cure systems determined at 90 °C are as given in Table 2.

The vulcanisation of CR in the presence of ZnO typically occurs above 150 °C²⁰. The cure parameters of compound Z-0 with 3 Phr of ZnO elucidate the inactivity of ZnO at 90 °C. Modified guanidine accelerator in blend Z-1, increases the net torque (MH–ML) value from 1.84 dNm to 7.32 dNm. The activity of binary accelerator systems using zinc diethyl dithio carbamate (ZDEC) (Z-2) and TC (Z-3) with guanidine is reflected in the reduction of respective t_{s2} and t_{90} . The synergistic effect of ZDEC/TC system (Z-4) is effusive in the results. The elimination of MgO from the cure system stimulates the curing action due to the auto catalytic action of ZnCl₂ formed *in-situ* during the cure reaction²¹. Desai²², *et al.* reported the

Table 2. Cure characteristics of CR–BIIR blends with different cure systems

Compound (Table 2)	Temp. (°C)	ML (dNm)	MH (dNm)	MH–ML (dNm)	t_{s2} (min)	t_{90} (min)
Z-0	90	2.73	4.57	1.84	-	55.08
Z-1	90	2.63	9.95	7.32	15.03	48.69
Z-2	90	2.49	9.55	7.06	12.12	46.00
Z-3	90	2.50	9.86	7.36	12.30	47.41
Z-4	90	2.83	10.25	7.42	11.20	45.55

slowdown of curing when MgO is jointly used with ZnO. Ethylene thiourea generates carcinogenic nitrosamine during cure reaction and hence, the elimination of ethylene thiourea is as an additional point in favour of the curing system²³.

3.2 Phase Morphology of Vulcanizates

The morphology and the phase dimensions determine the overall mechanical and physical properties of the blend. The morphological analysis carried out for neat and filled compounds of CR, BIIR and their blends with and without compatibiliser using SEM is as shown in Figs. 1(a)-1(h).

It can be observed that the surface morphology of neat compounds (Figs. 1 (a) to 1(d)) is smooth, whereas the surface morphology of compounds with fillers (Figs. 1(e) to 1(h)) is not smooth. The possible reason for this rough phase morphology is that the rubber compounds with the fillers undergo high shearing during mixing process. The formation of additional platelet structures observed in Fig. 1(g) may be due to the poor interface adhesion between CR and BIIR. Whereas, in the micrograph of compatibilised blend shown in Fig. 1(h), most of the platelet domains merged resulting single phase morphology due to the improvement in interfacial adhesion by the compatibiliser. This indicates that compatibiliser is an important material that allows blending of two immiscible polymers creating a homogeneous mixture. Generally, the neat BIIR comprises of larger particle size as compared to the lower particle size in case of the neat CR. These are reasonably visible in Figs. 1(a) and 1(b). The effect of compatibiliser is seen as dispersion of phases into the regions of interfaces of these rubbers and allows mixing, which are reasonably visible in Figs. 1(d) and 1(h). The compatibiliser used in the present study is a polar CPE. It concentrates on the interfaces of CR and BIIR and enhances the polar-polar interactions resulting an improved interfacial adhesion and hence, compatibility. This behaviour is reported by Singh²⁴, *et al.* for similar blends that uses HTV. Pandey²⁵, *et al.* observed in their study of

blend that the extra halogen present in the compatibiliser CPE enhanced the interfacial adhesion between CR and BIIR. Compatibiliser controls the sizes of the domains in a multi-phase blend by lowering the interfacial tension between the phases or reduces the agglomeration of domains by steric stabilisation. An important effect of the use of compatibiliser is the increase in the adhesion between the phases and consequently improving the mechanical strength of the blend. Beyond the use of compatibiliser, the addition of fillers like nanoparticles are expected to increase the viscosity of matrix, thereby help improving the compatibility of blends, which are reasonably visible in Figs. 1(e) and 1(g). Hence, from SEM studies, it is learnt that the compatibilised blend is more suitable for the encapsulation of the oceanic sensor than the uncompatibilised blend.

3.3 Evaluation of Blends using Dynamic Mechanical Analysis

The results of dynamic mechanical analysis (DMA) carried out on individual rubber compounds and blends are as given in Table 3.

Table 3. Viscoelastic properties of neat compositions at -80 °C

Compound	Storage modulus E' (MPa)	Loss modulus E'' (MPa)	Tg (°C)
CR	6000	77.62	-22.79
BIIR	4231	317.2	-27.86
80/20	6741	174.4	-20.45
C80/20	6650	176.4	-20.41

A higher storage modulus of ~650 MPa to 750 MPa was observed for the blend of CR-BIIR than CR alone. This indicates a better miscibility and interaction at the interfaces of CR and BIIR. The polar CPE concentrates at the interface of CR and BIIR making the blend compatible due to polar-polar interactions. This behaviour is also reported by Singh²⁴, *et al.*

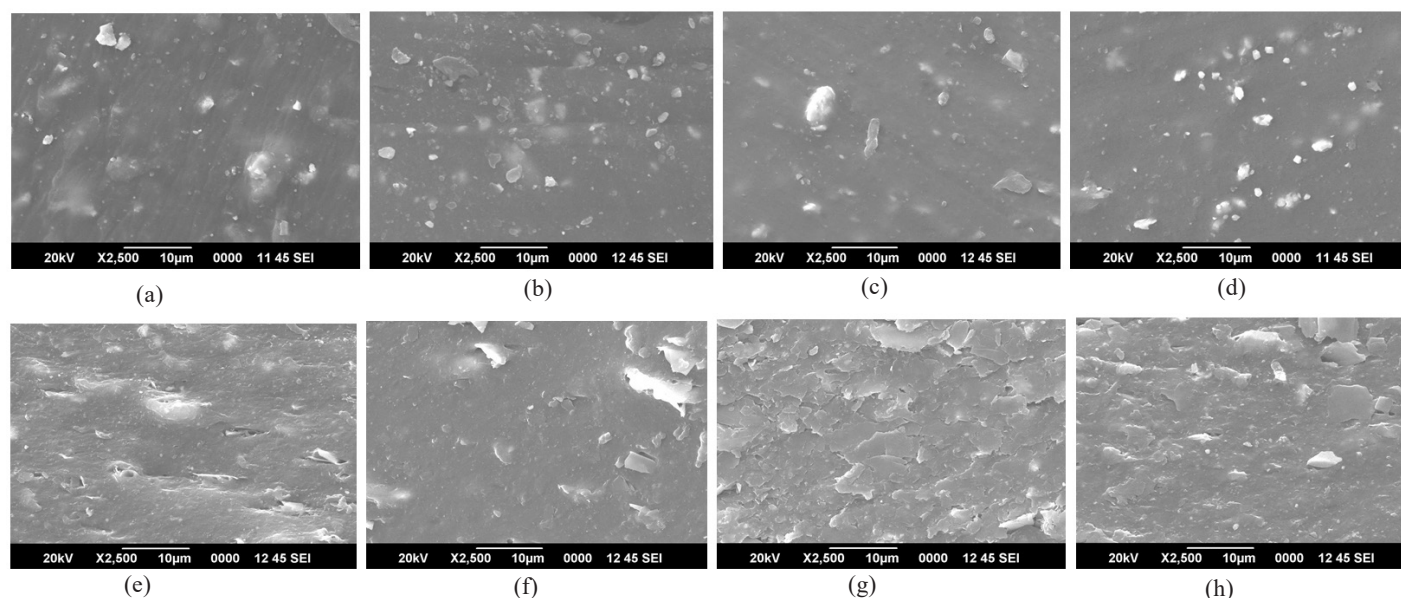


Figure 1. SEM micrographs: (a) Neat CR (b) Neat BIIR (c) Neat CR-BIIR blend (d) Neat CR-BIIR blend with compatibiliser (e) CR with fillers and other ingredients (f) BIIR with fillers and other ingredients (g) CR-BIIR blend with fillers and other ingredients, and (h) CR-BIIR blend with fillers, other ingredients and compatibiliser.

in HTV technique. The slightly lower value of E' at -80 °C obtained for compatibilised blend (C80/20) than the uncompatibilised blend (80/20) may be due to the presence of the third polymer, the CPE. As expected, a higher loss modulus was observed for BIIR than CR. This is because of the delayed elastic response to deformation exhibited by butyl rubbers due to the presence of two methyl side groups in the polymer chain. However, the loss modulus is not much affected by the addition of compatibiliser. The variation in storage modulus with temperature is as shown in Fig. 2. The observations drawn from the data provided in Table 4 are also noticed from Fig. 2 over a temperature range of -80 °C to 30 °C.

The tensile and tear strengths computed for CR, BIIR and their blends are as given in Table 4. Five samples in each were evaluated and the obtained standard deviation is also given.

Table 4. Tensile and tear strengths of CR, BIIR and their blends

Compound	Tensile strength (MPa)	Tear strength (N/cm)
CR	16.93 ± 0.15	552 ± 9.43
BIIR	8.71 ± 0.35	247 ± 10.41
80/20	16.06 ± 0.21	466 ± 10.06
C 80/20	16.41 ± 0.31	479 ± 9.43

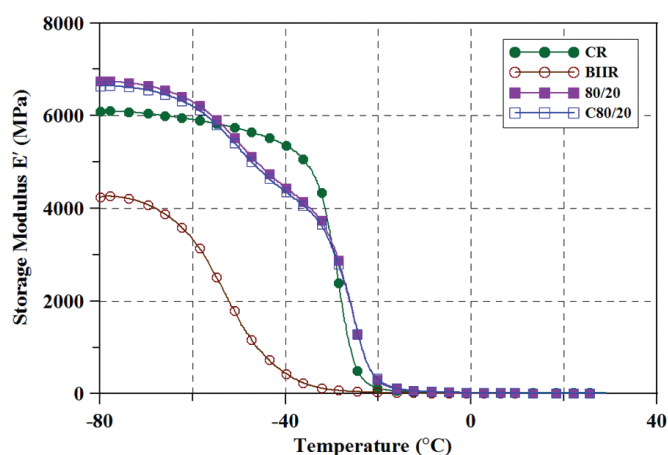


Figure 2. Effect of temperature on storage modulus.

The decrease in tensile and tear strengths of blends than that of CR is due to the dilution effect of BIIR. However, the blends retained its tensile and tear strengths close to its master rubber CR. Due to the effect of compatibilisation, there is an increase in tensile and tear strengths. Although, a very significant improvement in tear and tensile strength was not observed, achieving these improvements using a LTV method is considered to be an improvement through this study. If the best compatibiliser is selected to make a blend of two immiscible rubber materials, then the expected outcome in its tensile and tear strengths would be the best and it is beyond the scope of the present paper. Mangaraj²⁶ observed that incompatible blends of elastomers resulted into lower mechanical properties.

The loss factor, $\tan \delta$, obtained from DMA analysis of CR, BIIR and their blends with and without compatibiliser is as shown in Fig. 3. It is observed that the $\tan \delta$ peak of blend with and without compatibiliser has shifted towards the higher

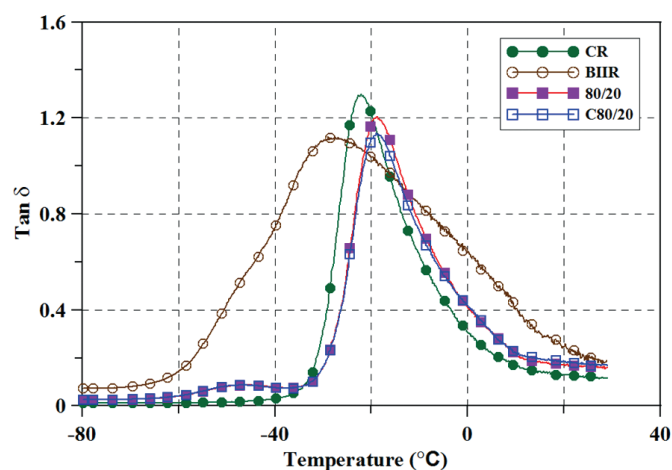


Figure 3. Variation of $\tan \delta$ with temperature.

temperatures. The $\tan \delta$ peak due to the soft BIIR moiety appear as shoulder to the major peak of CR, in both the compatibilised and non-compatibilised blends, indicating better miscibility of the blends²⁷.

3.4 Evaluation of Acoustic Property

The acoustic evaluation of the individual vulcanisate and the blends was carried out using the pulse tube measurement technique by impulse method in a water filled impedance tube²⁸. Results of echo reduction tests conducted with these samples are as shown in Fig. 4.

For an acoustic material, the impedance, which is the product of velocity of sound in the medium and the density of the medium, should match sea water, so that incident sound waves can enter the material without being reflected²⁹. The BIIR being an energy absorber shows a higher echo reduction, as expected. For an excellent acoustically transparent material, the echo reduction values should be generally close to 0 dB³⁰. It is observed from Fig. 4 that the compatibilised blend (C80/20) exhibits a low echo reduction among all the other samples. This improvement in the acoustic property is due to the merging of the interfaces of CR and BIIR by the action of compatibiliser. The echo reduction is comparable to all the attempted samples. A better consistent behaviour is noticed for the C80/20 sample at higher frequencies.

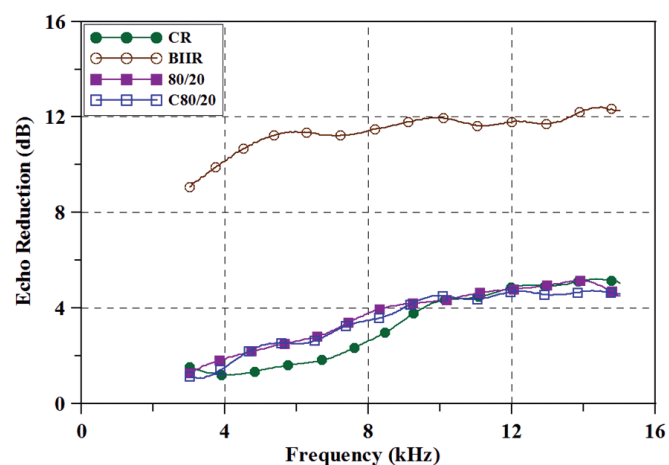


Figure 4. Echo reduction of blend and individual rubbers.

Figure 5 shows the comparison of acoustic performance of LTV and conventional HTV system. The echo reduction increases almost linearly with increase in frequency. However, LTV system shows a lower echo reduction and is almost stabilised from 9 kHz onward.

A marginal gain in echo reduction of the order of 1 dB is obtained over the conventional HTV method. Although this gain is marginal, it is very significant in underwater acoustic sensing especially to detect silent vessels made using stealth technology, as the encapsulant made out of the present work allowed additional passage of about more than 6 % of acoustic intensity to the sensor.

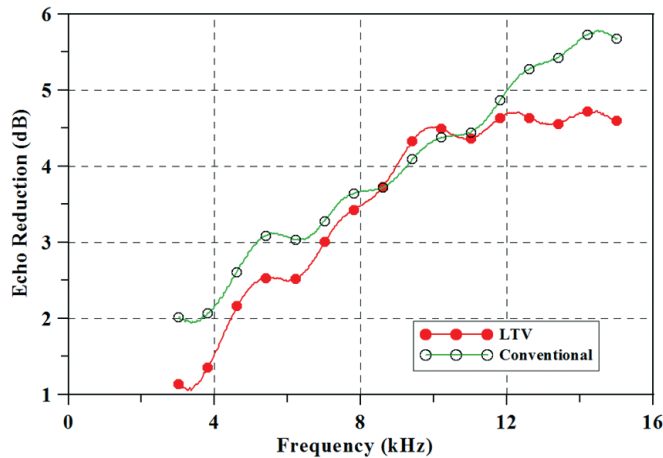


Figure 5. Comparison of echo reduction in optimised sample C80/20 with conventional HTV system.

3.5 Evaluation of Physical and Mechanical Properties

The physical and mechanical properties of the blend C80/20 with LTV system were determined as per the relevant ASTM standards and as given in Table 5, and are compared with the conventional HTV system. The LTV system offers higher tensile, tear strengths and volume resistivity and the other parameters like hardness and modulus are comparable.

Table 5. Physical and mechanical properties of the compatibilised CR-BIIR blend

Property	Test standard	Values	
		LTV	HTV
Specific gravity	Densimeter	1.368	1.414
Hardness (Shore A)	ASTM D 2240	62	62
Salt water absorption (% wt. gain)	ASTM D 471	0.34	0.21
M 100 (MPa)	ASTM D 412	3.38	3.76
M200 (MPa)	ASTM D 412	5.89	6.58
Tensile strength (MPa)	ASTM D 412	16.41	9.53
Elongation at break (%)	ASTM D 412	575	274
Tear strength (N/cm)	ASTM D 624	479	378
Volume resistivity@ 1000V (Ω cm)	ASTM D 257	2.67×10^{11}	1.43×10^{11}

3.6 Accelerated Thermal Ageing

The retention of modulus at 300 per cent elongation, tensile strength and elongation at break after accelerated ageing of dumbbell specimens at 70 °C for 7 day in an air oven were computed. The test results obtained for LTV and HTV systems are as given in Table 6. LTV offers better retention of properties than HTV suggesting that LTV blend is an ideal material for encapsulation of oceanic sensors especially transducers used in sonar systems.

Table 6. Percentage retention of tensile properties of dumbbell specimen

Property	Retention of property (per cent)	
	LTV system	Conventional system
M300	123.10	94.98
TS	97.08	90.82
EB	95.49	93.68

3.7 Implementation to PZT Hydrophone Sensor and underwater Characterisation

A commercial PZT sensor in its crystal form was encapsulated by molding at 90 °C with the new LTV blend for realizing a hydrophone. The frequency response of hydrophone was evaluated in an underwater test facility. The details of the experiments are given elsewhere by Mohanadas¹⁶, *et al.* The hydrophone exhibits a flat frequency response up to 4 kHz with a sensitivity of -197 dB ref $1V/\mu$ Pa within an acceptable deviation of ± 1 dB. It is found that the experimental result is found to fairly match with the specifications of PZT.

4. CONCLUSIONS

A novel LTV technique for CR-BIIR blend for encapsulation of oceanic sensors is successfully demonstrated. The conventionally used scavenger MgO was eliminated and only the ZnO curing agent was used to boost the cure reaction. The technique uses modified guanidine and thiocarbonyl as cure accelerators. The functional properties specific to underwater oceanic sensors viz. acoustic transparency, tensile, tear strengths, aging studies for longevity to survive in the hostile ocean environment are studied. Results are verified with that of the traditional HTV system and found that the proposed LTV system has the advantages over the conventional HTV system. Case study carried out on PZT sensors suggests that the proposed LTV technique has potential utility in other underwater sensors especially optical sensors³¹.

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In the present study, he formulated the concept, conducted the preparation of rubber compounds, characterisation data acquisition and analysis.

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In the current study, he has provided all guidance in improving the conceptualisation, data analysis and product characterisation, critical reviewing of manuscript and final approval.