Sulphur-vulcanised Epoxidised Natural Rubber (ENR)-Carbon Black Blends with Reproducible Electrical Conductivity Behaviour

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Sulphur-vulcanised epoxidised natural rubber [ENR]-carbon black printex XE2B blends with high electrical conductivities (up to 10^{-1} S.cm^{-1}), exhibiting good tensile properties and high damping were successfully prepared by using an internal mixer. Effect of carbon black's surface area and structure on electrical behaviour was studied for the vulcanised blends by testing them through a series of straining cycles (i.e. a total of 900 times of strain loading and unloading process). Carbon black printex XE2B particles with 'hollowed out' shape and higher surface area were observed by using a transmission electron microscope. The carbon black printex XE2B characteristics contributed to the very low percolation threshold (i.e. 3.0 wt % of printex XE2B loading) and also the reproducible electrical conductivity of its blend under straining process (≥95% retention of original unstrained value). With good mechanical properties and reproducible electrical behaviour, the sulphur-vulcanised ENR-carbon black printex XE2B blends (with printex XE2B loadings, 5.0–40.0 wt %) have potential to be used as a raw material for manufacturing flexible smart sensor devices.

Carbon blacks are regularly used to modify the physical behaviours of an elastomer by simply introducing them into the elastomer's matrix. Changes of static and dynamic moduli[1], strength[2–3], abrasion resistance[4] and electrical conductivity[5–7] are usually expected for a carbon black filled elastomer. Grades of carbon black, which can enhance the physical strength of an elastomeric host significantly, are also called reinforcing fillers.

Percolation threshold[8–11] is a volume fraction of conductive filler at which it can be assumed that a continuous interconnecting conductive filler network is created in its elastomeric host matrix. Below this volume fraction, the electrical resistivity is relatively high and above this threshold, the elastomeric compound behaves like an electrical conductor. In the region of the percolation threshold, the conductive filler particles will relocate and also reorientate under the application of a tensile strain and subsequently result in changes of the elastomeric compound's electrical behaviour. It is also well known that the electrical resistance of an elastomeric

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compound will decrease as the proportion of its conductive filler increased[5–7].

Both natural rubber and synthetic rubber blends based on a common reinforcing grade-carbon black (*i.e.* N330) exhibited non-reproducible electrical behaviour after only one straining process as reported by Yamaguchi[6]. Sau[12] worked on the blends of ethylene-propylene-diene rubber (EPDM), nitrile rubber (NBR) and silicone rubber respectively with common reinforcing grade carbon black as conductive filler. It was reported the conductivities of all blends were decreased by increasing the degree of test pieces compression. All these observations were attributed to the permanent destruction of the interconnecting networks as built up by the common reinforcing grade carbon black particles during their reorientation/relocation process[6,12].

This study investigated the possibility of preparing a type of vulcanised epoxidised natural rubber (ENR)-carbon black based material with reproducible electrical conductivity behaviour. A special grade of conductive carbon black (*i.e.* printex XE2B) was introduced into the ENR host matrix (at weight fractions above the percolation threshold) in order to achieve the targeted application. It was decided to produce the ENR-carbon black blends by using a temperature-controllable internal mixer (HAAKE PolyLab OS, RheoDrive 16). Electrical conductivities

and some basic physical properties (*i.e.* hardness, tensile properties, tear strength, compression set and resilience) were also determined for all the vulcanised blends.

EXPERIMENTAL

Chemicals and Raw Materials

ENR, a sustainable material (grade ENR 50 with 50 mole% of epoxide level, manufactured by the Malaysian Rubber Board) was chosen as the only elastomeric host matrix due to its good mechanical properties and high damping behaviour[13]. High damping is an advantage in shock absorption, which can reduce unnecessary noises if used as the material for high-sensitive sensor devices manufacturing. Specialty conductive grade of carbon black printex XE2B, manufactured by Evonik Degussa GmbH was used as the only conductive filler in this work. A comparison of characteristics of both conventional reinforcing grade carbon black *i.e.* N330 and specialty conductive grade carbon black *i.e.* printex XE2B is summarised in *Table 1*. The printex XE2B is manufactured under different processes (*i.e.* controlled partial oxidation of heavy oil or gasification process) from the conventional reinforcing grade carbon black (*i.e.* combustion of oil in a furnace)[14]. As a result, the surface area of printex XE2B is much greater than other common reinforcing grade carbon blacks[14].

TABLE 1. COMPARISON OF CHARACTERISTICS OF COMMON REINFORCING GRADE CARBON BLACK (N330) WITH SPECIALTY CONDUCTIVE GRADE CARBON BLACK (PRINTEX XE2B)

Character	N330	Printex XE2B
Surface area/NSA[a] (m²/g)	82[14]	>900[14]
Mean primary aggregate size (nm)	105[14]	100[14]
Mean carbon black particle (nm)	32[14]	30[14]

[a] NSA: nitrogen surface area (*ASTM D 3037-88*)

The conventional sulphur vulcanisation system was chosen here. Sulphur and benzothiazole disulphide (Perkacit MBTS, sourced from Bayer Malaysia Sdn. Bhd.) were used as the curing agent and accelerator respectively. Zinc oxide and stearic acid both sourced from Luxchem Sdn. Bhd. were also added as the vulcanisation system activators.

Preparation of ENR-Carbon Black Blends and Addition of Vulcanising Chemicals

An internal mixer (HAAKE PolyLab OS, RheoDrive 16) was first used to prepare ENR-carbon black blend masterbatches with different compositions of ENR wt %: carbon black wt % (97.5:2.5, 95.0:5.0, 92.5:7.5, 90.0:10.0, 80.0:20.0, 70.0:30.0 and 60.0:40.0). Blend formulation used for this study is shown in *Table 2*. A fill factor of 0.80 for the internal mixer (with free total volume, 320 mL) was chosen in order to perform the mixing. The starting temperature of each mixing was 100ºC and the mixer rotor speed was set at 100 r.p.m. *Table 3* summarises the stages of each mixing.

2.0 p.p.h.r. of sulphur and 1.0 p.p.h.r. of Perkacit MBTS were added later manually to each masterbatch on a pre-warmed 50ºC two-roll mill with gap distance adjusted to ~3.0 mm. Each masterbatch was removed from the two-roll mill in sheet form after about five min of mixing.

Preparation of Sulphur-vulcanised ENR-Carbon Black Test Pieces

Physical and electrical properties of each sulphur containing ENR-carbon black blend sheet as prepared by the two-roll mill was analysed. Appropriate amounts of each blend were cut from the sheet and fed into a 2 mm thick rectangle (130 mm × 100 mm) mould.

TABLE 2. ENR-CARBON BLACK PRINTEX XE2B BLEND FORMULATION

Raw material/chemical	Parts per hundred rubber (p.p.h.r.)
ENR (ENR 50)	60.0-97.5
Carbon black (Printex XE2B)	2.5-40.0
Zinc oxide	5.0
Stearic acid	2.0
Sulphur	2.0
Perkacit MBTS	1.0

TABLE 3. STAGES OF MIXING FOR ENR-CARBON BLACK PRINTEX XE2B BLEND MASTERBATCH BY USING AN INTERNAL MIXER (HAAKE POLYLAB OS, RHEODRIVE 16)

Stage of mixing	Timing
Addition of raw ENR	0th minute
Addition of carbon black	1st minute
Addition of zinc oxide and stearic acid	2nd minute
Sweeping	3rd minute
Dumping	6th minute
	(Total time= 6 minutes)

The mould together with the blend was sent for hot pressing and curing (at temperature 150ºC, pressure 60 p.s.i. and duration of curing level 90%, t_{c90} as determined by a Monsanto Moving Die Rheometer, MDR 2000E (*Table 4*).

Test pieces for the tensile properties (tensile strength, elongation at break and modulus) and Dunlop rebound resilience were prepared according to standards *BS ISO 37*[15] and *BS 903-A8*[16] respectively. Test pieces in disc shape (with thickness 6.3 mm and diameter 13.0 mm) that were needed for compression set (*BS 903-A6*[17]) and hardness measurement (*BS 903-A26*[18]) were prepared by feeding the disc-shaped mould with layers of blended material direct from the two-roll mill. Test pieces were also prepared without carbon black added to the vulcanised ENR blend for control purposes.

Physical Property Tests

Basic physical properties (*i.e.* IRHD hardness, tensile properties, rebound resilience and compression set) tests were performed according to BS ISO standards[15–18]. Six test pieces of each vulcanised blend were used to repeat each test in order to define the

final result as the mean value of a total of six measurements.

Morphological Study (Transmission Electron Microscopy)

Simple transmission electron microscopy (TEM) study (with a Philips CM12 microscope at accelerating voltage, 80 kV) was carried out for all sulphur-vulcanised ENR-carbon black printex XE2B blends. All observed images were captured digitally. Some ultra-thin samples (*ca*. 150 nm) of each blend were obtained for this purpose by sectioning with a microtome.

Electrical Conductivity Determination (Effect of Straining Process)

Electrical conductivities for all unstrained samples were calculated first based on the 2-probe method and *Equation 1* in order to determine the percolation threshold. The effect of strain up to 100.0% elongation in term of sample's length on selected sulphur-vulcanised ENR-carbon black printex XE2B blends (only those above the percolation threshold) was also investigated. A Keithley 6517A electrometer with built in-voltage

TABLE 4. CURING DURATION (t_{C90}) OF SULPHUR VULCANISING AGENT CONTAINING ENR AND ENR-CARBON BLACK PRINTEX XE2B BLENDS AS DETERMINED BY A RHEOMETER

Blend (ENR wt % : carbon black wt %)	Curing duration (t_{c90}), ± 0.5 minute
100.0 : 0.0	26.1
97.5 : 2.5	25.8
95.0 : 5.0	25.2
92.5 : 7.5	25.2
90.0 : 10.0	24.8
80.0 : 20.0	23.5
70.0 : 30.0	22.3
60.0 : 40.0	21.2

source up to 1 kV was used in this work. For each blend, six test pieces in strip form with dimensions 80 mm × 20 mm × 1 mm were prepared using a hot press (150ºC and duration according to *Table 4*) in order to obtain a mean value. Each of the test pieces was strained by using an in house designed jig system. Three separated cycles of straining process were carried out for all blends. Each cycle of straining process consisted 300 times of strain loading and unloading motion (*i.e.* with sample stretched to 100% elongation and then, returned back to 0% elongation). The mean electrical conductivity value obtained from each cycle was calculated respectively. The electrical "resistance" measurement set-up in this study is shown in *Figure 1* (based on the 2-probe method and *Equation 1*[19–20]).

$$\sigma = \left(\frac{1}{R}\right)\left(\frac{L}{A}\right) \qquad \dots 1$$

where σ is the electrical conductivity, R is the mean value of apparent resistance (voltage/current ratio) for the pressed sample, L is the measured electrode spacing distance and A is the cross-sectional area of pressed sample between the current-carrying electrodes.

Both opposite faces and edges of each test piece were coated with a large area of quick-drying silver paint (Acheson Electrodag 915) in order to minimise the effect of contact resistance. A fine copper wire of length 5 cm was attached to the middle of each side of these silver paint coated areas for connection to the electrometer. All the contacting surfaces between the jig and the test piece were properly insulated by a layer of insulating PTFE sheet (~1.0 mm thick). All readings were made at 10 s after each elongation step in order to standardise the effect of stress relaxation. The new length (L_i) and thickness (d_i) values of each test piece were also measured after each elongation step in order to calculate the new values of cross-sectional area (A_i).
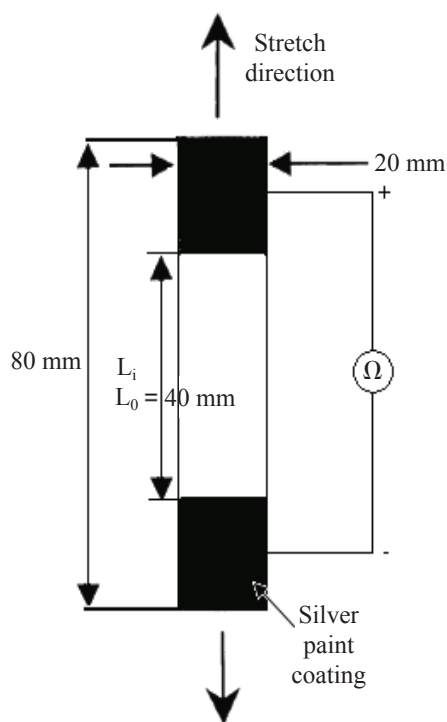

Figure 1. Electrical resistance measurement setup for the study of effect of straining process on the sulphur-vulcanised ENR-carbon black printex XE2B blends.

RESULTS AND DISCUSSION

Physical Property Tests

IRHD Hardness Measurement. Results of the IRHD measurement are summarised in *Figure 2*. The degree value of hardness (IRHD) of blend increased with proportion of added carbon black printex XE2B. This was due to the hard and rigid nature of carbon black printex XE2B[7,14] and increased stiffness of the blend as the content of conductive filler increased.

Tensile Properties Measurement. Results of the tensile strength, elongation percentage
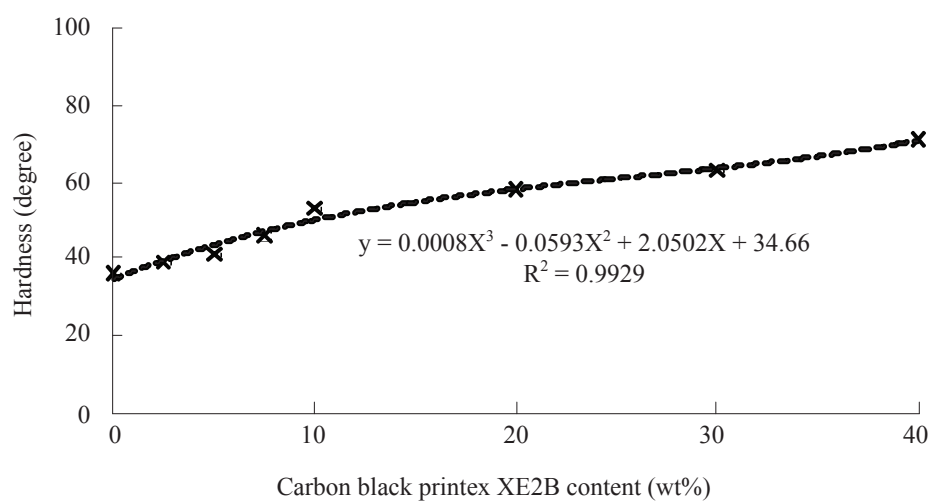
$$y = 0.0008X^3 - 0.0593X^2 + 2.0502X + 34.66$$
$$R^2 = 0.9929$$

Figure 2. IRHD of the sulphur-vulcanised ENR and ENR-carbon black printex XE2B blends.

$$y = 5E\text{-}06X^5 - 0.0005X^4 + 0.0174X^3 + 0.3123X^2 + 3.129X + 8.4506$$
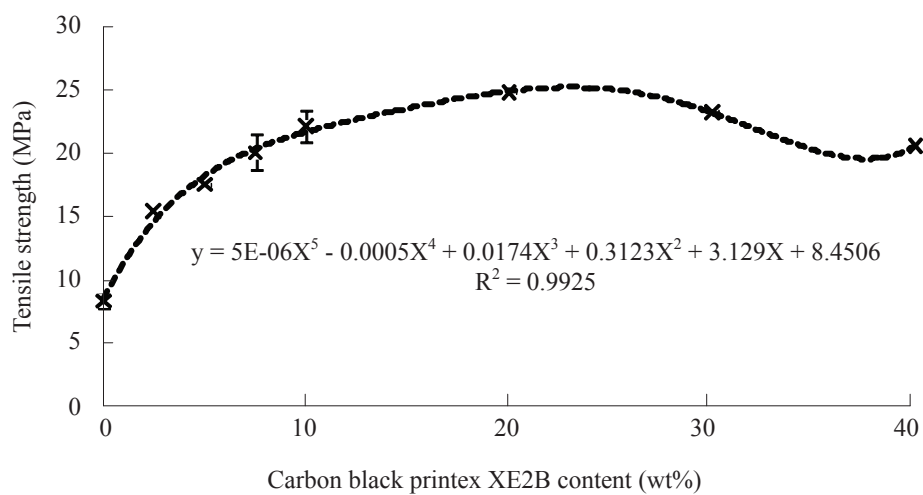$$R^2 = 0.9925$$

Figure 3. Tensile strength of sulphur-vulcanised ENR and ENR-carbon black printex XE2B blends.

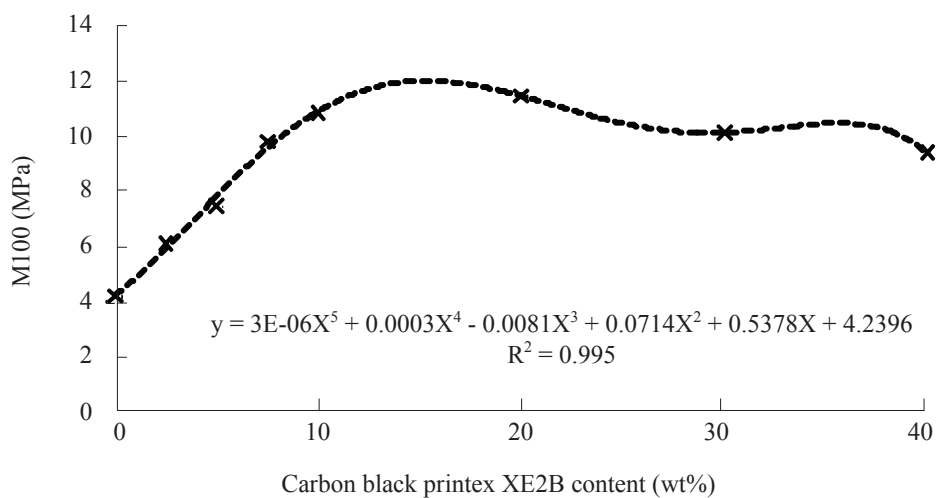*Figure 4. Elongation percentage at break (EB %) of sulphur-vulcanised ENR and ENR-carbon black printex XE2B blends.*

Figure 5. M100 of sulphur-vulcanised ENR and ENR-carbon black printex XE2B blends.

at break (EB %) modulus at 100% (M100) and modulus at 300% (M300) are shown in *Figures 3, 4, 5* and *6* respectively. Carbon black printex XE2B acted as a reinforcing agent and hence, both tensile strength and EB % of blends increased with the proportion of carbon black (2.5-20.0 wt %). The tensile strength and EB % decreased for blends with ≥ 30.0 wt % of carbon black (due to the increase of phase-separated or agglomerated carbon black particle regions). Donnet[3] reported carbon blacks with higher structure could act as the reinforcing fillers (up to an optimised addition level) as a result of the increased volume of elastomer-filler interface connection. Results of M100 and M300 also showed the blend's good elastomeric feature in general.

Compression Set and Dunlop Rebound Resilience Measurements. Results of the compression set and Dunlop rebound resilience measurements are summarised in *Figures 7* and *8* respectively. As expected, the compression set values increased with the content of carbon black printex XE2B. At the same time, the Dunlop rebound resilience values decreased with the content of carbon black printex XE2B. In other words, damping properties of the ENR-carbon black printex XE2B blends could be enhanced with their decreasing rebound resilience values. Both of these phenomenon happened due to the carbon black printex XE2B's rigid structure and non-elastomeric behaviour.

Morphological Study (Transmission Electron Microscopy)

Simple transmission electron microscopy (TEM) study was carried for all sulphur-vulcanised ENR-carbon black printex XE2B blends. However, only two examples of representative TEM micrographs (*Figure 9*) were presented here, *i.e.* blend with 2.5 wt % of printex XE2B just below percolation threshold

and blend with 5.0 wt % of printex XE2B just above percolation threshold. Both TEM micrographs suggest that each individual carbon black printex XE2B particle has 'hollowed out' shape[7] which is significantly different from the conventional reinforcing grade carbon blacks[6]. This also explains why each individual carbon black printex XE2B particle has higher surface area per unit mass if compared to the particle of conventional reinforcing grade-carbon black (*e.g.* N330). In this case, the effective filler volume fraction is also much higher than the simple volume of added filler[7]. Jha[7] also reported that each 'hollowed out' shaped-printex XE2B particle could also trap larger volume of unmovable rubber in their conducting shell. As a result, even small volume of added printex XE2B will be able to a create dramatic effect on electrical properties.

Electrical Conductivity Determination (Effect of Straining Process)

Electrical conductivities for all unstrained samples were identified first in order to determine the percolation threshold (*Figure 10*). The electrical conductivities of all the blends increased with the proportion of conductive filler, as observed in several studies of electrically conductive rubber blends[5,6,8,9,19,20]. The blends reached an electrical conductivity in the order of 10^{-1} S.cm^{-1} with only 10.0 wt % of carbon black printex XE2B loading. The estimated electrical conductivity percolation threshold for the ENR-carbon black printex XE2B in this study is about 3.0 wt %. A very low threshold value was seen here due to the higher surface area per unit mass for each individual carbon black printex XE2B particle (*Figure 9*) if compared to the conventional carbon black[6].

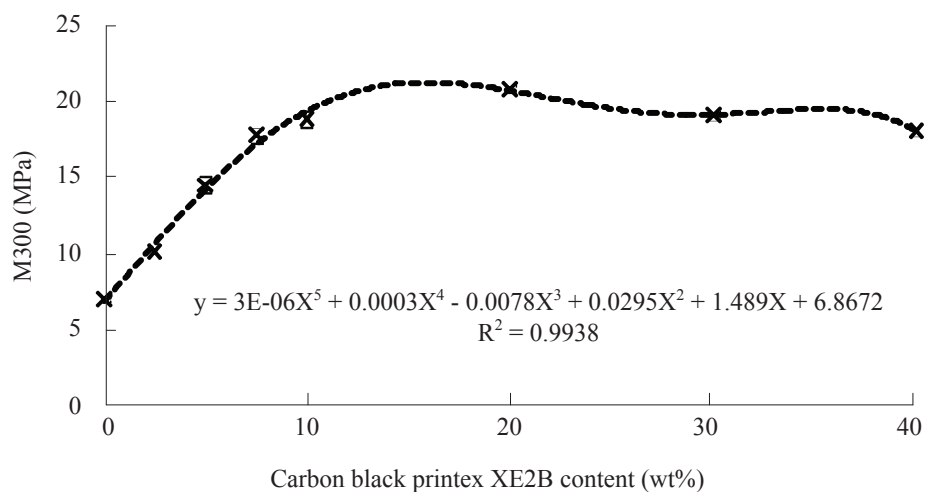The charts of log electrical conductivity versus elongation % for selected sulphur-

$$y = 3E\text{-}06X^5 + 0.0003X^4 - 0.0078X^3 + 0.0295X^2 + 1.489X + 6.8672$$
$$R^2 = 0.9938$$

Figure 6. M300 of sulphur-vulcanised ENR and ENR-carbon black printex XE2B Blends.

$$y = -8E\text{-}06X^5 + 0.0008X^4 - 0.0271X^3 + 0.3486X^2 - 0.501X + 13.959$$
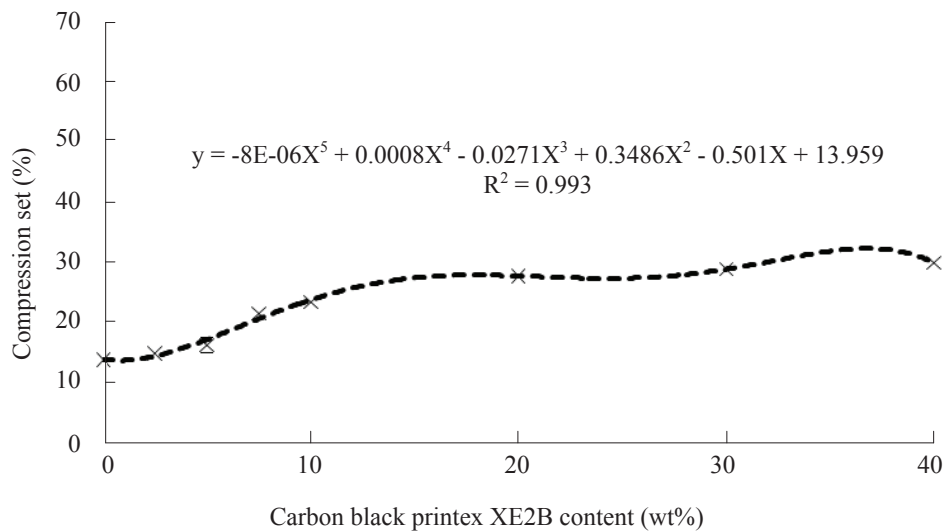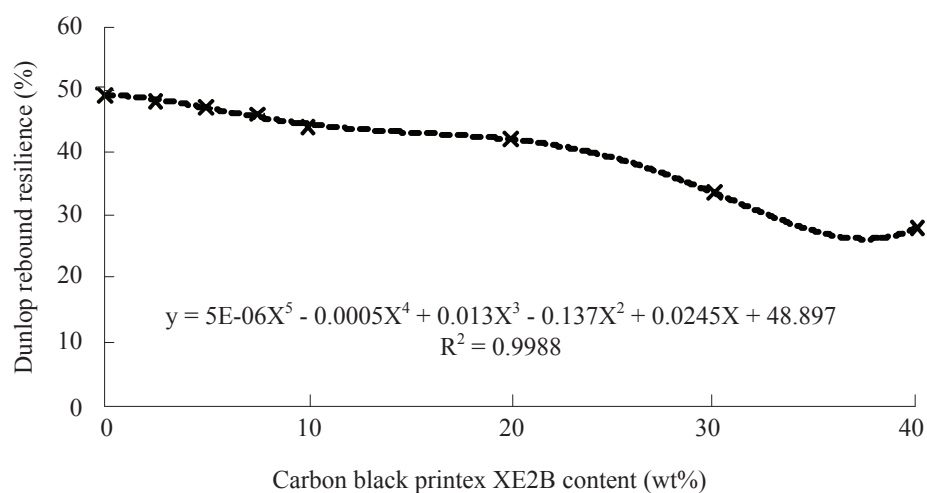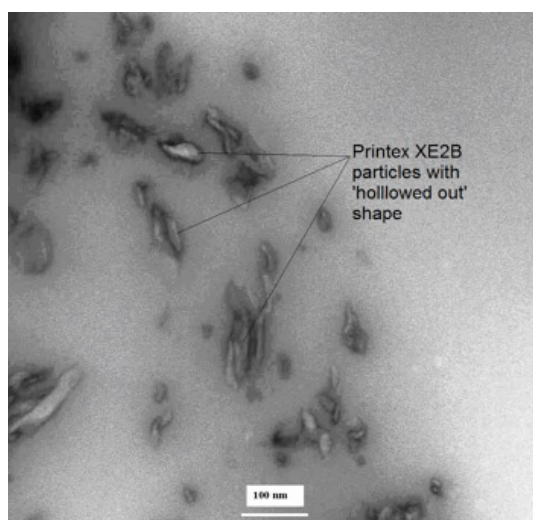$$R^2 = 0.993$$

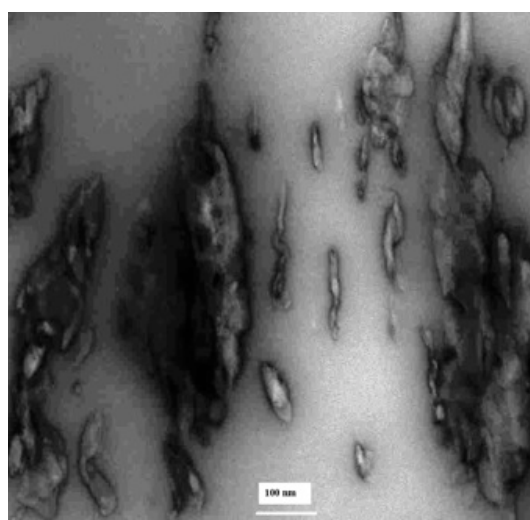*Figure 7. Compression set (%) of sulphur-vulcanised ENR and ENR-carbon black printex XE2B blends.*

$$y = 5E\text{-}06X^5 - 0.0005X^4 + 0.013X^3 - 0.137X^2 + 0.0245X + 48.897$$
$$R^2 = 0.9988$$

*Figure 8. Dunlop rebound resilience (%) of sulphur-vulcanised ENR and
ENR-carbon black printex XE2B Blends.*

(a)   (b)

*Figure 9. Transmission electron micrographs (20,000 ×) of the
Sulphur-vulcanised ENR-carbon black printex XE2B blends. Blends contain,
(a) 2.5 wt % of carbon black printex XE2B and (b) 5.0 wt % of carbon black printex XE2B.*
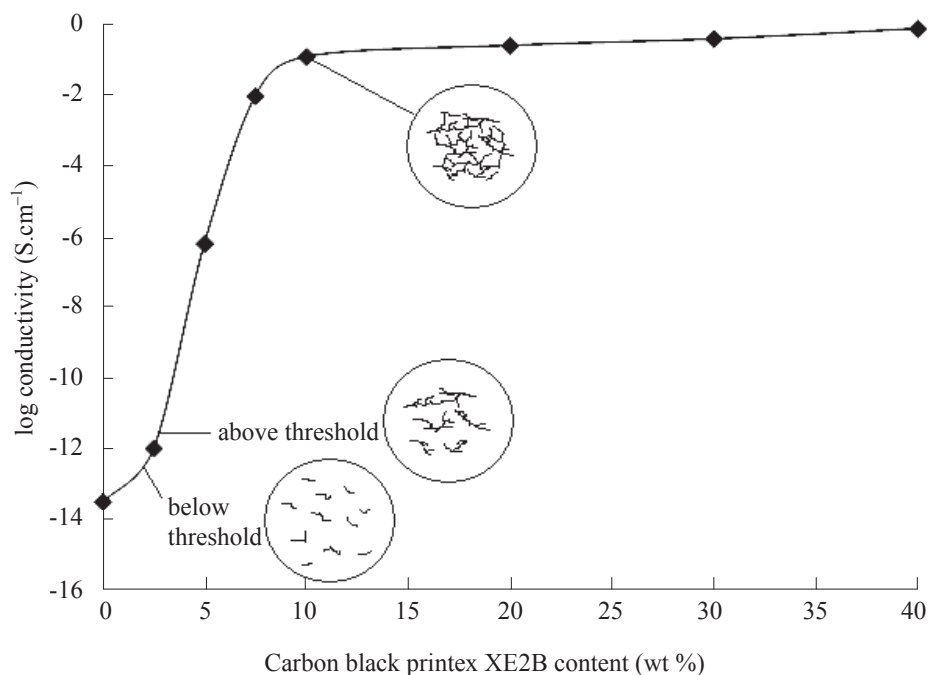
Figure 10. Electrical conductivities for the sulphur-vulcanised ENR-carbon black printex XE2B blends as a function of carbon black printex XE2B content (in wt %).

vulcanised ENR-carbon black Printex XE2B blends (those above the percolation threshold, *i.e*. 5.0 wt %, 10.0 wt %, 20.0 wt %, 30.0 wt % and 40.0 wt %) are all shown in *Figure 11*. Three separated cycles of straining process were carried out for all blends. Each cycle of straining process consisted of 300 times of strain loading and unloading motions. The mean electrical conductivity value obtained from each cycle was calculated and presented in *Figure 11*. The first, second and third cycles of straining process for all vulcanised blends exhibited almost similar reproducible electrical behaviour, *i.e*. the conductivity increased almost linearly with strain loading and also able to recover very closely to the original unstrained value during the unloading process. At each straining cycle, the conductivity increased at least by 1 order of magnitude at an elongation of 100% (in terms of sample's length).

The electrical behaviour of all blends prepared in this study was in contrast with those of the blends prepared with the conventional reinforcing grade carbon black (*i.e.* N330). According to literature[6–7], the electrical conductivity of test piece prepared from the rubber-carbon black N330 blend decreased permanently even during the first straining process (*i.e*. with one strain loading and one strain unloading). This was attributed to the high levels of phase separation between the two constituent materials and also the permanent destruction of conductive carbon black N330 networks within the rubber host matrix. The carbon black N330 particles were reported[6] to be in normal spherical
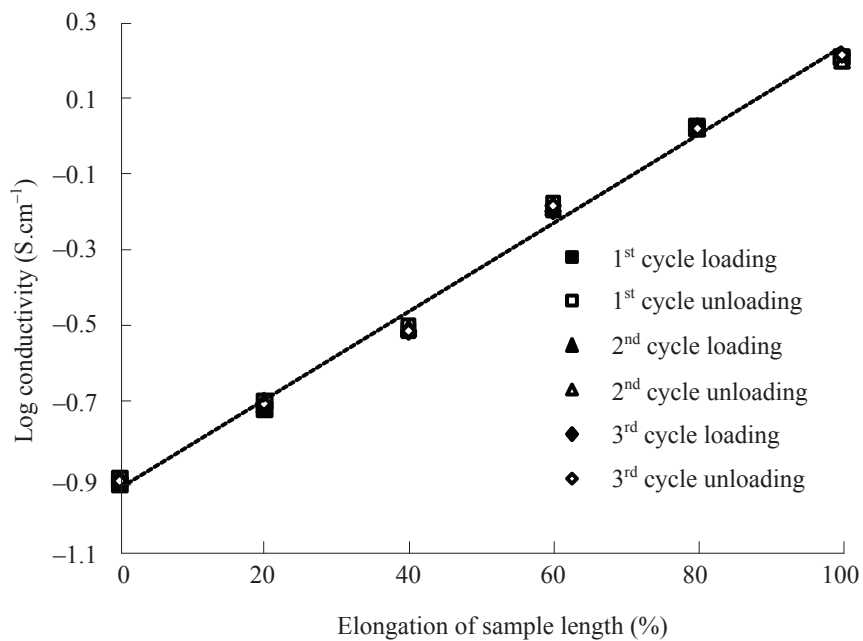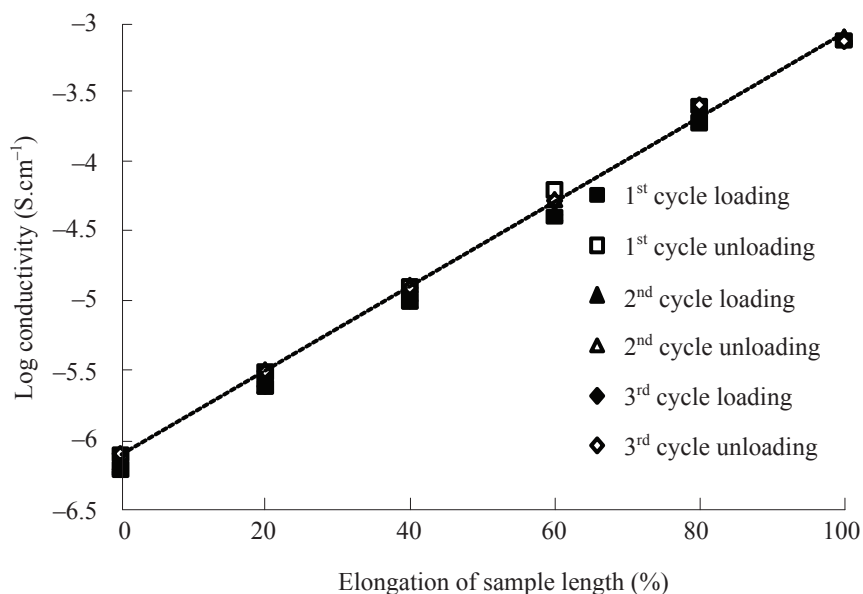
Figure 11. Charts of log electrical conductivity versus elongation % for the sulphur-vulcanised ENR-carbon black printex XE2B blends; (a) blend with 5.0 wt % carbon black printex XE2B, (b) blend with 10.0 wt % carbon black printex XE2B.
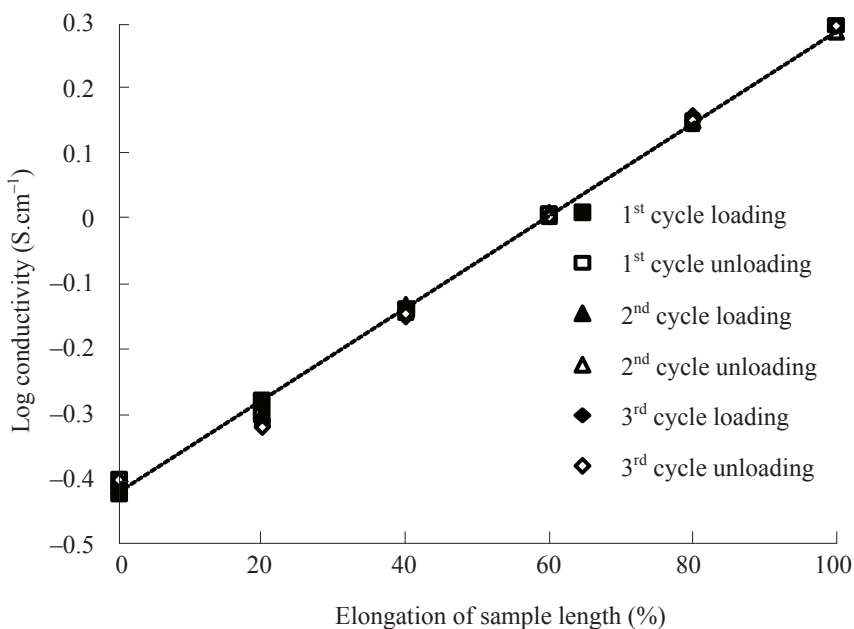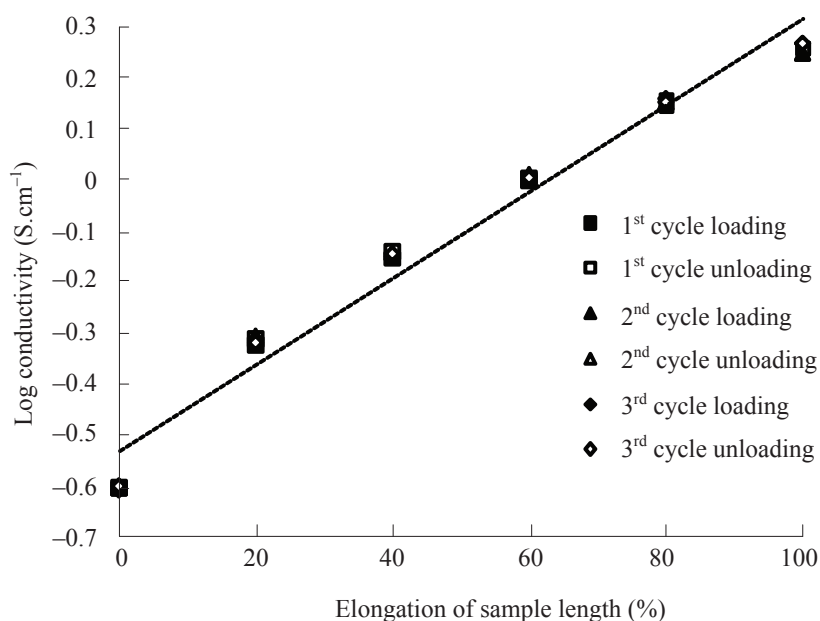
Figure 11. Charts of log electrical conductivity versus elongation % for the sulphur-vulcanised ENR-carbon black printex XE2B blends; (c) blend with 20.0 wt % carbon black printex XE2B, (d) blend with 30.0 wt % carbon black printex XE2B.
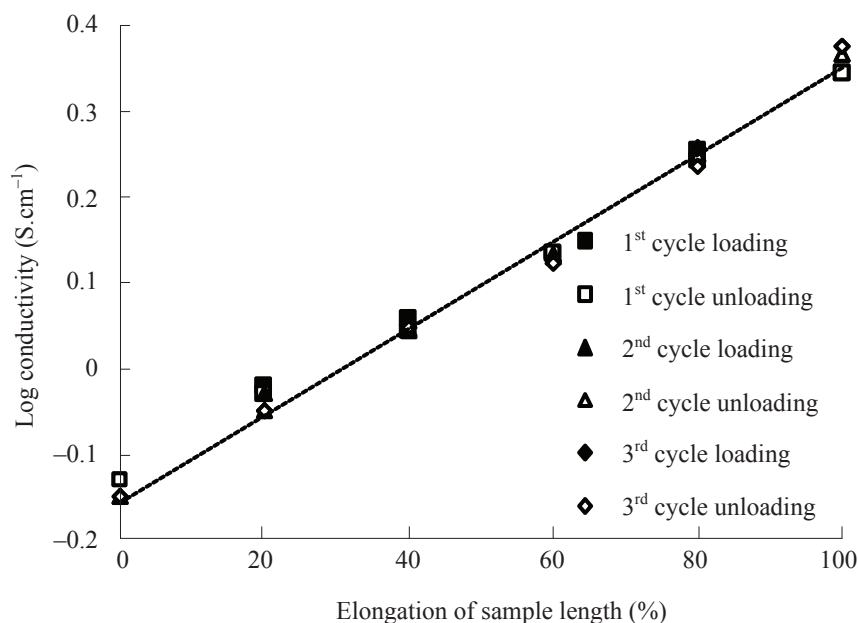
Figure 11. Charts of log electrical conductivity versus elongation % for the sulphur-vulcanised ENR-carbon black printex XE2B blends; (e) blend with 40.0 wt % carbon black printex XE2B.

shape (lower surface area with reference to *Table 1*) rather than the 'hollowed out' shape as observed for printex XE2B used in this study. Particles of carbon black printex XE2B with higher surface areas are likely able to undergo relocation and reorientation without losing their inter-connected conductive paths during the straining cycles. At the same time, they are also able to trap more rubber into their 'conducting shells' (as discussed in morphological study) and this may explain why the conductivity increased during the strain loading process of each cycle.

CONCLUSION

Sulphur-vulcanised ENR-carbon black printex XE2B blends with electrical conductivities up to 10^{-1} S.cm^{-1} were successfully prepared by using an internal mixer. All the fillers-added

vulcanised blends also possessed classic ENR physical properties, such as good tensile properties and high damping. The effect of filler's surface area and structure was most obviously seen when the blends were going through a series of straining processes (with monitored electrical behaviour). From the morphological study, it is learnt that the carbon black printex XE2B particles are in a 'hollowed out' shape and have much higher surface areas compared to the conventional reinforcing grade ones (*e.g.* N330). As a result, the effective printex XE2B volume fraction is also much higher than the simple volume of added printex XE2B. All these carbon black printex XE2B features did contribute to the very low percolation threshold for unstrained samples (*i.e.* 3.0 wt % of printex XE2B loading) and also the reproducible electrical behaviour (≥95% retention of original unstrained value) for samples under tensile strain. It is strongly

believed that sulphur-vulcanised ENR-carbon black printex XE2B blends (with printex XE2B contents, 5.0–40.0 wt %) have good potential to be used as the raw material for manufacturing flexible smart sensor devices[21] (*e.g.* pressure and load sensors) due to their good physical properties and reproducible electrical behaviour.

ACKNOWLEDGEMENTS

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