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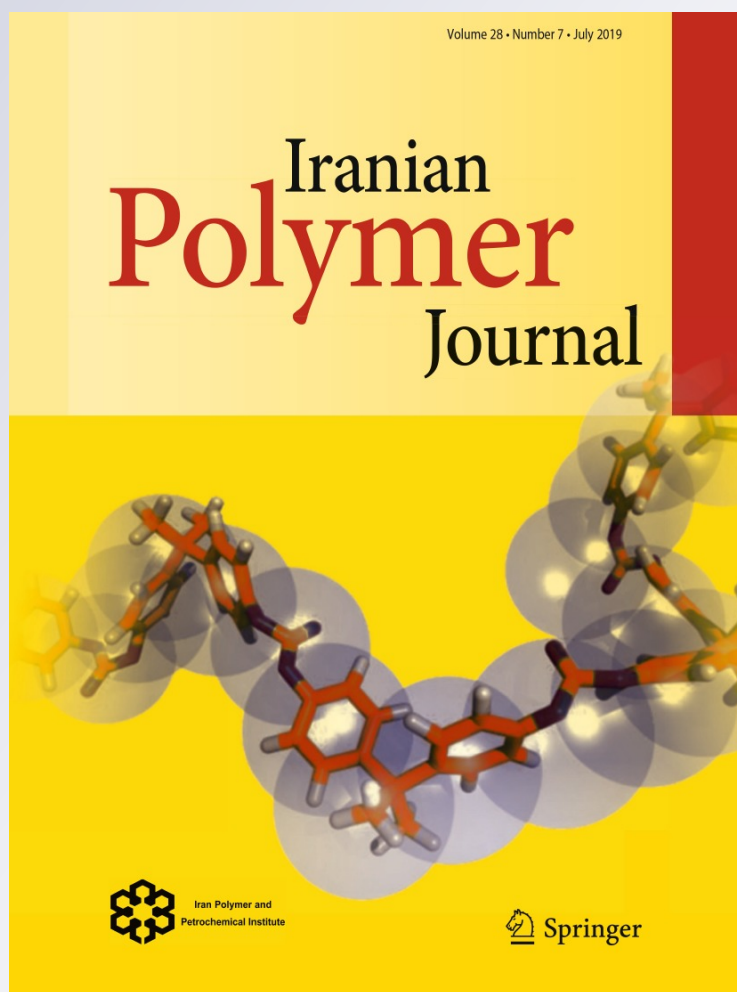
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Improvement in volume resistivity and morphology of a blend of polyolefin elastomer with linear low-density polyethylene

Mehri Dana¹ · Gholam Hossein Zohuri^{1,2} · Saeid Asadi³

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Abstract

A silane moisture-cured polyolefin elastomer/linear low-density polyethylene (LLDPE) blend was prepared through a two-step silane-grafting method (Sioplas Process) in an industrial scale twin-screw extruder. The silane-grafted compound was used to make wire and cable coatings. In this work, the effect of some interactive parameters on quality of the products prepared by the above method has been studied, while so far, there have been less experimental investigations. The volume resistivity of cross-linked compound was changed from 2.96×10^{14} to 7.41×10^{14} Ω cm with increasing LLDPE component by maximum 10 wt%. Surface morphology of the product was corrected with reduction in benzoyl peroxide (BPO) concentration from 0.2 wt% to 0.13 wt%. BPO at this level acted as an initiator in grafting reaction of vinyl trimethoxysilane. The curing condition and specimen preparation method by injection molding and/or extrusion were factors which influenced the hot-set test results at 200 °C. The results of tensile and elongation studies showed a maximum value of 9 MPa and 397% for the tests, after 6 h curing. With increases in curing time at a specified temperature, the gel content of the cross-linked compound was increased and reached its maximum value. The maximum gel content values were found to be approximately 60%, 80%, and 82% at temperatures of 25, 60, and 85 °C, respectively. The hardness, density, and tear strength of the samples did not vary significantly with the curing temperature.

Keywords POE/LLDPE blend · Sioplas Process · Volume resistivity · Surface defects · Curing process · Gel content

Introduction

Thermoplastic elastomers (TPEs) are among the most widely used plastics in today's market. TPEs are known as high-performance polymer materials and used in different ways in many industries [1]. They are generally employed as insulating materials in electrical and electronic applications. Thermoplastic elastomers often are block copolymers or blends of polyethylene (PE), polytetrafluoroethylene (PTFE), polyester, polyvinyl chloride (PVC), polyethylene-2,6-naphthalene dicarboxylate (PEN), polyamides,

polypropylene, and many others [2]. TPEs are also used in many applications, such as indoor/outdoor electrical cable insulation, optical fiber sheaths, welding cable insulation, high voltage automobile cables insulation, coil forms and bobbins for transformers, parts for motors, aerospace electrical parts, flame-retardant insulation for cables and housing for composite high voltage outdoor insulators, substation and transformer bushings, in molded products and encapsulation of coils and microelectronic chips, etc. [1].

However, a 20–30 year service life was expected for cross-linked polyethylene when it was first used as coating the medium voltage cable in the 1960s. However, the service life of some of these early cables was far less than expected. The defects on the surface of cables coated by cross-linked polyethylene strongly disturbed the operation of the cables, and many cables became unusable after 10–15 years. Previously, engineers and scientists did not know what was wrong, but now, it is found that insulating voids and contaminants, along with ionic impurities in semiconductors, and other shortcomings in design and production are responsible for voltage stresses on cables. These, coupled with the influence

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of moisture on the entire structure, lead to a phenomenon known as water tree. The growth of the tree through these microscopic cavities over time will ultimately lead to the destruction of the insulation. Today, there are insulators of cross-linked low-density polyethylene (XLPE) that prevent the growth of the water tree [3, 4].

Peroxide plays two roles: one as a curing initiator and another as an initiator of the silane-grafting reaction. These two roles compete with each other; therefore, it is necessary that the silane and peroxide concentrations be optimal, to ensure that the silane-grafting reaction is dominant reaction rather than curing reaction [5].

A widely explored approach to further reduce the electrical interference of insulation materials such as XLPE is based on the concept of nanostructured dielectrics, which was introduced in 1994 by Lewis [6]. Other examples of the application of this approach are: improvement in dielectric constant and thermal conductivity of polystyrene-*b*-polybutadiene-*b*-polystyrene triblock copolymer [7] and oxidized styrene-butadiene-styrene copolymer [8] using novel exfoliated graphite nanoplates and also the silane-surface-modified graphene/poly(vinylidene fluoride) fibrous membranes [9]. A suitable secondary component is added to the polymer matrix, which either modifies the nanostructure or introduces new functional moieties that are located on the surface of the filler material. A trend is the use of metal oxide nanoparticles composed of, e.g., SiO₂, Al₂O₃, TiO₂, MgO, or ZnO [10–12]. Such nanoparticles are thought to introduce charge traps in polymer matrix that reduce the mobility of charge carriers such as electrons, holes, and/or polar species, which can exist in a polyethylene insulation [13, 14]. An alternative approach that currently receives less attention is the use of polymer blends, which would offer considerable processing advantages. Crystallization of a higher melting polymer fraction may lead to the formation of nano-sized domains similar to metal oxide nanoparticles which could act as charge traps. However, most previous studies have failed to observe a substantial drop in DC conductivity upon blending of various polyolefins [13, 15].

At low temperatures, the rate-determining step of the cross-linking reactions is water diffusion, rather than the hydrolysis and the subsequent condensation reactions of the silyltrimethoxy groups. However, at high temperatures and high degree of silane grafting, the chemical reactions dominate in the cross-linking process [16, 17].

The grafting silane onto the polymer chains is the most critical stage of the process. This reaction takes place in compounding equipments such as co-rotating twin-screw extruder or a Co-kneader. It is being used as a chemical reactor in addition to its normal function. Base polymer, fillers, and processing aid are conveyed through gravimetric feeders inside the extruder feed zone heated to a temperature of about 150 °C. The rpm and feeding rate are optimized

at not very high values. Liquid silane/peroxide mixture is fed by a high precision diaphragm pump into the polymer melt between the second and third zones of extruder. Other compounds such as stabilizer, carbon black, scorch retardant, etc. are fed into the polymer melt through a side feeder. A limitation of the silane curing method is the tendency to premature cross-linking (scorching) of the material which can lead to product defects, though it can be solved by scorch retardant additives [16].

In this work, the cross-linking of metallocene-based polyethylene-octene elastomer (POE) and linear low-density polyethylene (LLDPE) blend was carried out using the Sio-plas Process, which involves a two-step silane cross-linking process. The silane-grafting reactions were carried out using an industrial scale twin-screw extruder. The silane-grafted compound was shaped as wire and cable coatings. The effect of some important parameters on product quality has been observed, while so far less has been studied experimentally. These parameters include: (1) a polymer with higher crystallinity with respect to volume resistivity of the product; (2) excess peroxide in relation to product quality; (3) the important parameters in hot-set test at 200 °C (the key test in wire and cable industry); and (4) the effect of silane curing process on product properties. The volume resistivity was improved with the LLDPE component increased by 10 wt%. Morphology of the surface of the wire and cable prepared was improved by reducing the benzoyl peroxide content. The effect of curing time, water temperature, and curing process on the properties of cross-linked compound was studied, and in addition, the curing conditions and type of specimen, prepared using injection molding and extruder, were examined in relation to the results obtained by hot-set test at 200 °C.

Experimental

Materials

POE and LLDPE were purchased from SK Global Chemical Co., Ltd, (South Korea) and Amir Kabir Petrochemical Co., (Iran), respectively. Pentaerythritol tetrakis (3-(3,5-di-tert-butyl-4-hydroxyphenyl) propionate), Irganox 1010 antioxidant, was supplied by BASF Co., Germany. Vinyl trimethoxysilane (VTMS) was purchased from RUI Chemical (China). Dibutyltin dilaurate (DBTDL), the catalyst, was supplied by Merck (South Korea). Dicumyl peroxide and benzoyl peroxide, the initiators, were supplied by Coin Akzo Nobel Co., (Netherlands) and Arkema (France), respectively.

Sample preparation

The silane grafting of POE/LLDPE blend was performed in an industrial scale twin-screw extruder with 12 heating/

cooling zones (Model CTE 65, COPERION Co. Germany). A temperature profile of 170–210 °C from the feed zone to the die zone and a constant feed rate were applied. The reaction was completed within 5 min. The concentration of the peroxide was varied in the range of 0.13–0.2 wt%, while a constant silane concentration of 5 wt% was used. In the next step, a blend of the silane-grafted compound (~95 wt%) and the catalyst masterbatch (~5 wt%) was shaped as wire and cable coatings. A catalyst master batch consisting of catalyst (0.025 wt%), fraction of polyolefin elastomer (3.75 wt%) and antioxidant (0.3 wt%) was prepared using a roller.

For extrusion coating of a cable using the prepared silane-grafted compound, an extruder with 7 heating/cooling zones was used at a production speed of 10 m/min under a temperature profile of 170–220 °C from the feed zone to the die zone. The specimens (sheets) with different thicknesses were cured under the same conditions; for this purpose, each (specimen) sheet was cured in a water bath operated at 85 °C for 4–6 h per 1 mm sheet thickness.

Measurements

Various properties of the specimens were measured according to the standard methods, as listed in Table 1.

Results and discussion

Morphological development

Table 2 shows the actions taken to improve surface quality of the wire and cable manufactured and other properties of the produced compound. Figure 1 provides the SEM images of the prepared samples for evaluating their surface quality. Although the indirect mixing (mixing in a feeding hopper) was more effective, eliminating the LLDPE from the formulation and replacement of BPO with DCP (Perkadox BC-40B-pd, 40%) were also experienced and found that they were efficient in making modified surfaces. As shown

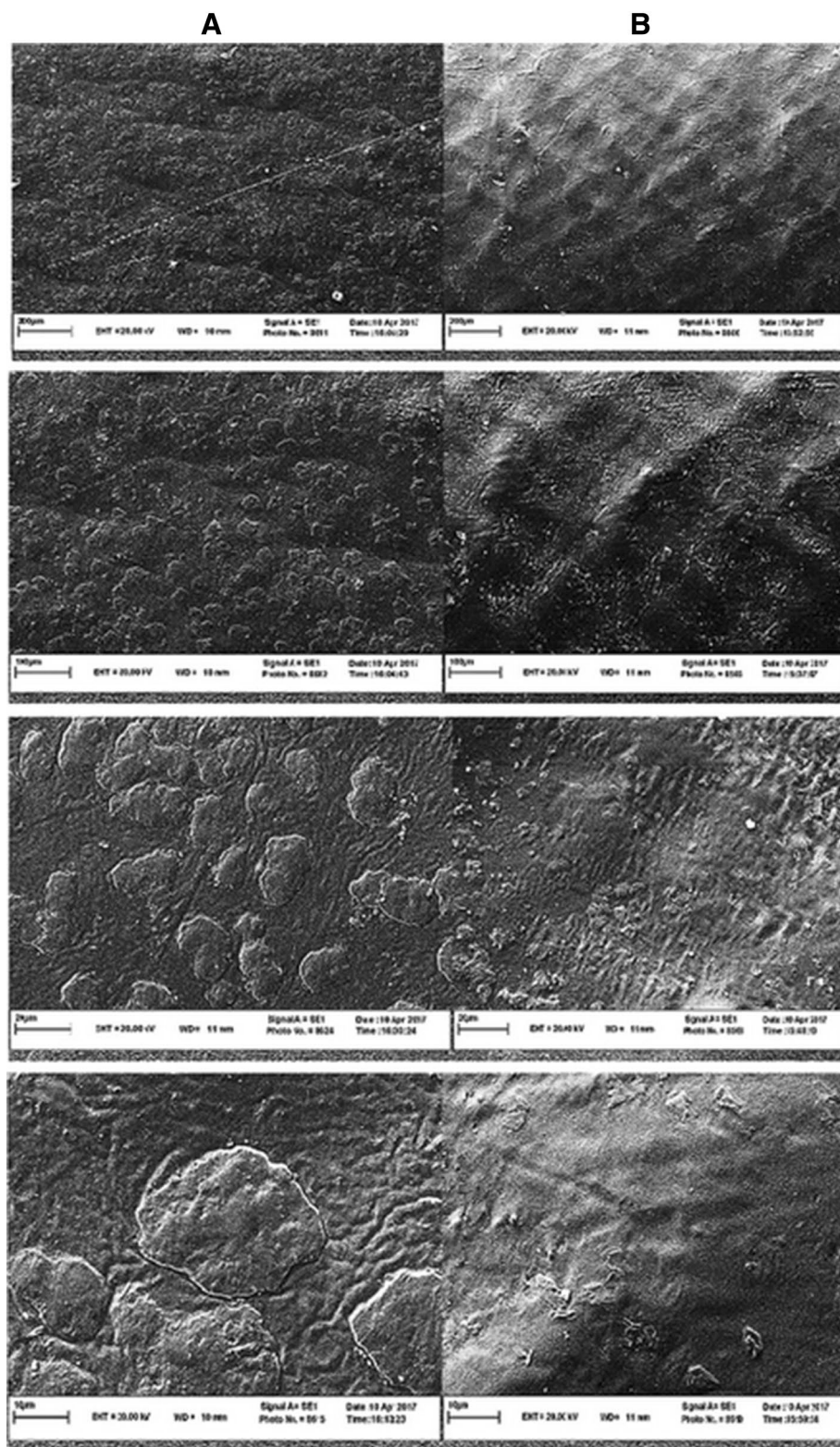
Table 1 Standard test methods used

Property	Standard test	Test conditions
Hardness, Shore A	ISO 868: 2003	1 s
Tensile strength-at-break and elongation-at-break	ISO 37: 2011 ISO 527: 2012	500 mm min ⁻¹ , 2-kN load cell, MPa
Density	ISO 1183: 2012	–
Melt flow index	ISO 1133: 2005	230 °C/5 kg, g/10 min, uncuring
Hot-set test	EN 60811-507: 2013	15 min, 200 °C, 20 N/cm ² , %, 8 h cured at 85 °C
Gel content	ASTM D 2765-95 a: 2016	Refluxing xylene containing 1% of antioxidant for 6 h, %
Volume resistivity	IEC 60502-1: 2004	–
Compression set	ASTM D 395: 2012	22 h at 70 °C 25% initial thickness, %
Tear strength	D415149: 2012	kg f/cm

Table 2 Procedure for improving the surface morphology of the product

Description					Properties
VTMS + BPO → wetting	VTMS + BPO → liquid pump	VTMS + BPO → liquid pump	VTMS + DCP → liquid pump	VTMS + BPO → wetting	
0.13 wt% BPO	0.2 wt% BPO → 0.13 wt%	100 POE + 0.13 wt% BPO	0.13 wt% BPO → 0.24 wt% DCP	0.2 wt% BPO	
73.6	1.4	2.5	70.4	75	Gel content %
7.1	18.2	20.9	15.5	3.22	MFI (230 °C, 5 kg)
0.881	0.914	0.899	0.909	0.911	Density
315.2	573.3	1090.0	391.6	483.0	Elongation-at-break
7.45	7.76	7.78	7.75	7.80	Tensile strength ISO 37: 2011
80.1	82.0	74.3	85.3	83.2	Hardness (shore A, 1S)
Suitable	inappropriate	relatively inappropriate	inappropriate	inappropriate	Surface quality

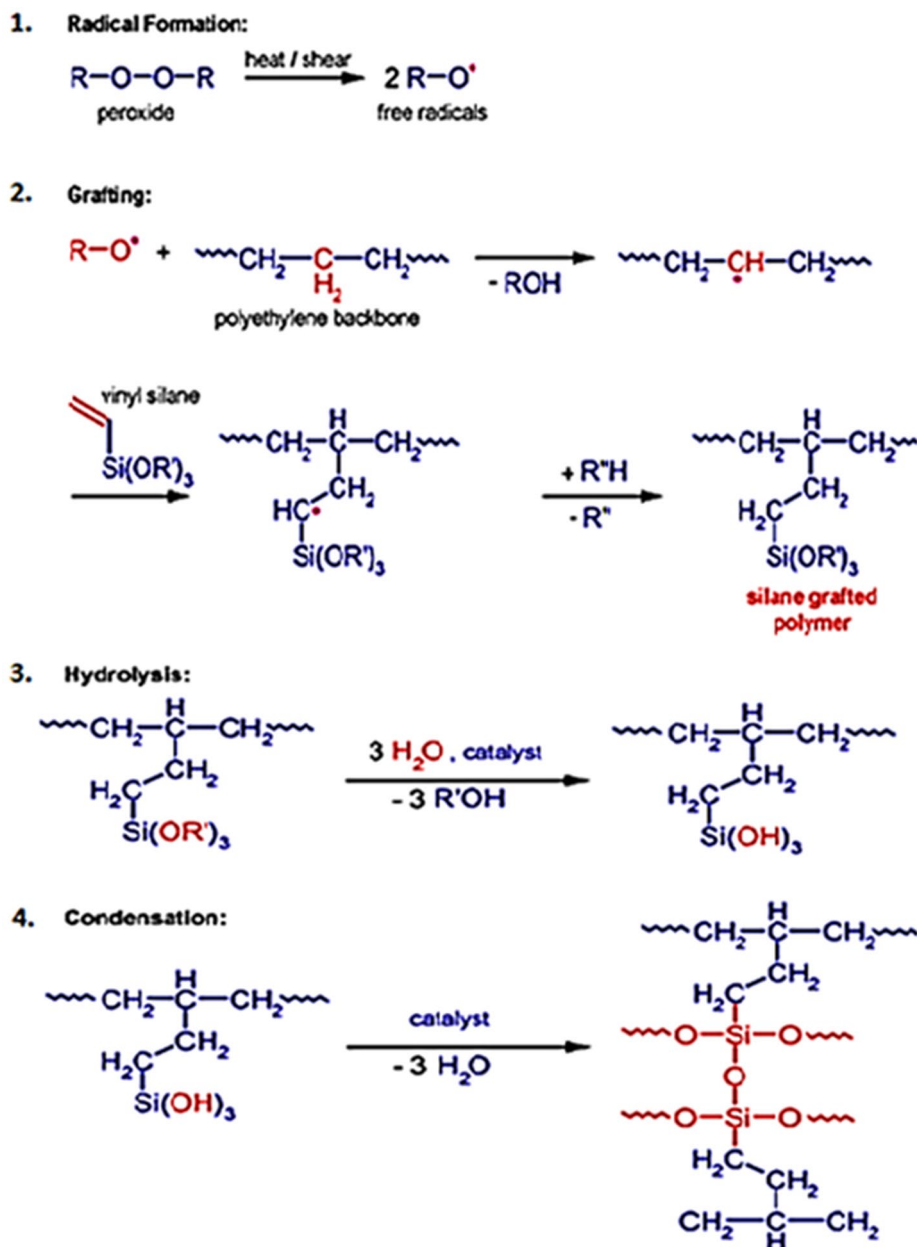
Fig. 1 SEM images of cable surface, **a** sample with good surface quality and **b** sample with poor surface quality. (1) Magnification $\times 250$, (2) magnification $\times 500$, (3) magnification $\times 2500$, and (4) magnification $\times 5000$



in the SEM images, only a decrease in BPO percentage was effective in improving the smoothness of the surface in the final product. The reason for this can be explained as follows, peroxide plays two roles: one as a curing initiator and

another as an initiator of the silane-grafting reaction. These two roles compete with each other; therefore, it is necessary that the silane and peroxide concentrations be optimal, to ensure that the silane-grafting reaction is dominant over the

Fig. 2 Mechanism of silane-grafting (1 and 2) and moisture-cross-linking (3 and 4) process



curing reaction [5]. During the process, even in the absence of the silane, peroxide is a suitable choice for making carbon-carbon bonds and cross-linking the polymer chains [18]. Grafting and cross-linking silane reactions are shown in Fig. 2. High concentrations of the peroxide result in the formation of cross links during the silane-grafting reaction and, therefore, the formation of gel spots, causing a rough surface of the product [16]. Therefore, it seems that the percentage of peroxide needs to be reduced. In both the Sioplas and Monosil methods, there is the risk of uneven distribution of the silane and hence the probability of local bonding which results in the formation of small gel spots. The distribution of cross-linked polyethylene chains is a direct result of effective dispersion of peroxide [19]. Direct

mixing of the silane with peroxide, using a suitable liquid pump in the extruder's hopper, was carried out simultaneously with reduction in peroxide content which resolved the problem of the final product and a smooth surface was produced.

Improvement of volume resistivity

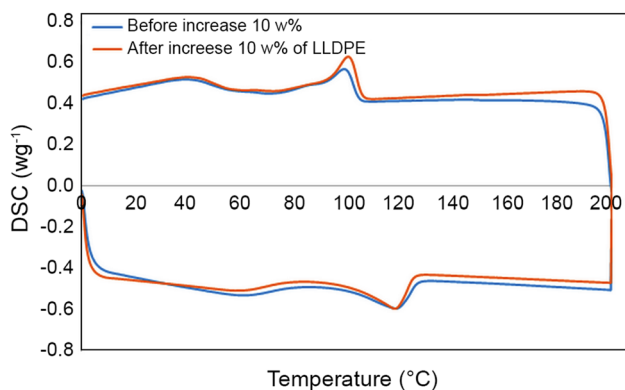
The results of the volumetric and superficial electrical resistance of the coating of the cross-linked compounds produced and reference samples are presented in Table 3. With increasing the LLDPE content up to 10 wt%, the volume resistivity is increased from 2.96×10^{14} to 7.41×10^{14} Ω cm. This behavior may be possibly due to an increase

Table 3 Comparison of volumetric and insulating electrical resistance of the cross-linked compounds produced with reference samples

Insulating resistance at 20 °C	Volume resistivity at 20 °C	Product name
Min: 80 (M Ω /Km)	Min: 5×10^{14} (Ω cm)	Admission limits
1350	1.5×10^{15}	Rubber rivals
> 5000	$> 3.7 \times 10^{15}$	XLPE
400	2.96×10^{14}	Cable coating cross-linked compound (not verified)
2000	7.41×10^{14}	Cable coating cross-linked compound (improved electrical resistance)

Table 4 Effect of LLDPE content (10 wt %) on the properties of the cross-linked compound

Properties	Test method	Before adding 10 wt % LLDPE	After adding 10 wt % LLDPE
Hardness	ISO 868, Shore A, 15 S	80.3	81.3
Tensile strength-at-break	ISO 527: 2012	6.33	6.83
Elongation-at-break	ISO 527: 2012	433.6	386.1
Gel content	ASTM D 2765-95 a: 2016	73.6	75.6
MFI (230 °C/5 kg), uncured	ISO 1133: 2005	6.58	6.55
Compression set	ASTM D 395: 2012	70.6	69.2
Tear strength	D415149: 2012	23	24.3
Density	ISO 1183: 2012	0.886	0.887
Tensile modulus	ISO 527: 2012		
20%		2.50	2.78
50%		3.41	3.58
100%		4.37	4.50
200%		5.15	5.23
300%		5.59	5.95
400%		6.18	–
Total crystallinity (%)		4.5	5.1

**Fig. 3** Differential scanning calorimetry (DSC) thermograms for the cross-linked compounds before and after adding 10 wt% LLDPE

in crystallinity of the polymer (Table 4, Fig. 3). The polymer crystal regions act as nano-reinforcement and fill the charge gap, thereby increasing the electrical resistance [13–15]. A slight improvement in some properties such as

hardness, tensile strength-at-break, gel content, compression set, tear strength, and density was observed with addition of LLDPE (10 wt%) into the cross-linked compounds, as expected (Table 4). Naturally, however, the two properties, elongation-at-break and MFI (230 °C/5 kg), were relatively reduced.

Hot-set test at 200 °C

Effect of curing conditions

It is required to achieve the hot-set property as the assessment method to confirm the insulation material of the electric wire maintaining its strength without melting at high temperature according to EN 60811-507: 2013 Standard. In accordance with this standard, the test was conducted, such that the dumbbell-shaped sample remained unchanged at high-temperature environment of 200 °C for 15 min with a definite static load weight of 20 N/cm². To pass the test (EN 60811-507, Part 507), the elongation between the gage

Table 5 Effect of curing conditions on the hot-set test results at 200 °C

Curing temperature (°C)	Curing time (h)	Results	
		Extension < 175%	Set < 15%
85	8 h	31.05	– 24.85
		28.85	– 26.50
		26.05	– 29.45
100	Shelf life at 200 °C (min)		
	2 h	3:26	
	4 h	4:28	
	6 h	3:56	

Table 6 Hot-set test results for specimens with different shapes

Specimen	Gel content %	Hot-set test at 200 °C	
		Extension < 175%	Set < 15%
Cable	74.0	154	2.25
	78.2	146	0.25
Dumbbell-shaped	75.6	29.6	– 23.9
	75.0	29.6	– 27.2

points must be $\leq 175\%$ after 15 min, and accordingly, the permanent elongation between the gage points after removing of the load is to be $\leq 15\%$ [20].

The results of the hot-set test at 200 °C at different curing conditions are shown in Table 5. As shown, the morphology of the specimens is affected by the curing conditions. For

the samples cured at 100 °C, the hot-set test was not passed because of possible degradation of the polymer. For the samples cured at 85 °C, however, the hot-set test was passed.°

Effect of processing type

The hot-set test at 200 °C was performed on the specimens prepared by both injection molding and extrusion processes (Table 6). The differences in the obtained results can be attributed to the different methods used in preparation of the test specimens. The pressure in extrusion process typically is 5000 Psi (35 MPa) which is less than that in injection molding process [21]. Due to high pressure of the injection method, the test specimens prepared by injection molding process are more compressed than those acquired by extrusion process. It may be stated that more compressed specimens have higher stress resistance.

Study of curing process

Effect of curing conditions on the properties of silane-grafted compound is shown in Table 7. The results indicate that the hardness, density, and tear strength of the cross-linked compound have not changed significantly under different curing conditions. The reductions of 12% and 16% were, respectively, observed for compression set and tension set. This is due to reduction in stress as a result of cross-linking process. Heat aging test was carried out at 70 °C for 168 h on a cross-linked compound both before and after curing. It was found that the results were improved by curing. Linear polymers are usually degraded more than the

Table 7 Effect of curing process on the properties of the silane-grafted and cross-linked compounds

Properties	Test method	Before curing	After curing
Hardness	ISO 868, Shore A, 1 S	87.5	86.3
Tensile strength-at-break	ISO 37, MPa	5.9	7.4
Elongation-at-break	ISO 37, %	132	287
Tensile strength-at-break	ISO 527, MPa	6.6	7.5
Elongation-at-break	ISO 527, %	1206	1023
Compression set	D45 113, (22 h, 70 °C)	64.4	52.3
Tension set	D45 1132, (22 h, 70 °C)	66.8	50.8
Tear strength	D 415149, kgf/cm	25.7	24.9
Aging Δ tensile	ASTM D 573	7.3	1.3
Aging Δ Elongation	7 day, 70 °C, %	23.5	47.9
Aging Δ Hardness		0.8	1.1
Density	ISO 1183	0.892	0.892
Tensile modulus			
20%		1.80	2.40
50%	ISO 527, MPa	2.56	3.23
100%		3.12	3.88
200%		3.67	4.68
300%		3.93	5.08

branched and cross-linked polymers, probably due to the absence of entanglements and cross links. As shown in Table 6, the increase in tensile strength after curing measured by the ISO 37: 2011 and ISO 527: 2012 methods is not the same. The rate of water propagation to reach the positions of silanol groups, which are susceptible to curing, is faster at lower thickness [22]. Therefore, this difference in the tensile strength increase can be attributed to the difference in the thickness of dumbbell-shaped specimens recommended by ISO 37: 2011 (≈ 2 mm) and ISO 527: 2012 (≈ 4 mm). Obviously, the increase in tensile modulus is due to the increase in tensile strength resulted from the curing process in a given present age of elongation-at-break (Table 7).

Effect of curing temperature and time on cross-linking

The effect of temperature and time of curing on gel content, as a criterion of degree of curing, is presented in Fig. 4. Each value of gel content is an average of three measurements. At low temperatures, the water penetration process is the rate-determining step, but at high temperatures, the hydrolysis reaction is the rate-determining step, resulting in faster formation of cross links. However, at high temperatures, a high degree of silane grafted on two types of polymers used in compounds is involved in the cross-linking chemical reactions [22].

An increase in curing time (from 15 min to 8 h) at a specified temperature leads to an increase in the gel content which reaches a certain value. The gel contents of 60%, 80%, and 82% were observed at 25, 60, and 85 °C, in the stated order. This result shows that the number of cross links formed in the gel structure is reached a maximum value, so the highest value of the gel content is obtained. In addition, the cross-linking reaction obeys a first-order kinetics of concentrations

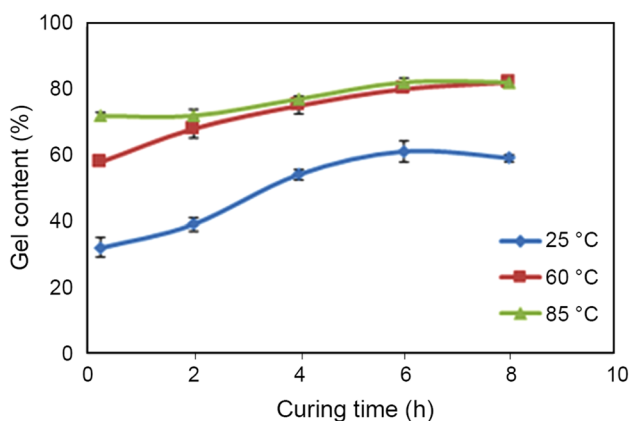


Fig. 4 Effect of temperature and curing time on the amount of gel content

of the catalyst and moisture [16, 22]. As shown in Fig. 4, the increase in temperature and time does not affect the highest gel content. In general, the amount of time needed to reach complete curing depends on the thickness, percentage and type of catalyst masterbatch, temperature, and humidity [22].

Effect of curing time on mechanical properties of the cross-linked compound

The tensile strength and elongation-at-break versus curing time are shown in Fig. 5. As shown, the highest amount of the tensile strength and the elongation are obtained after 6 h of the curing time. According to Fig. 4, the gel content increases with curing time. The loss in the mechanical properties is probably due to the polymer degradation occurred after curing completed. It may also be possible after reaching the highest amount of properties; by continuing the curing conditions, it can improve the direction of the polymer chains, thereby reducing the tensile strength [23].

Conclusion

Cross-linking of POE/LLDPE blend was carried out using the two-step Sioplas Process in an industrial scale twin-screw extruder. The blend of the silane-grafted compound (~ 95 wt%) and the catalyst masterbatch (~ 5 wt%) was used to make coatings for wire and cable. The volume resistivity of cross-linked compound was increased from 2.96×10^{14} to $7.41 \times 10^{14} \Omega \text{ cm}$ with increasing the LLDPE content (10 wt%). A decrease in BPO content by 0.07% showed an improvement in the quality of the cable surface which was revealed in SEM images. The curing conditions and the type of specimen were the factors influenced the hot-set test results at 200 °C. With increasing the curing time at a specified temperature, the gel content was increased and reached a maximum value. The maximum values were found to be approximately 60%, 80%, and 82% at temperatures 25,

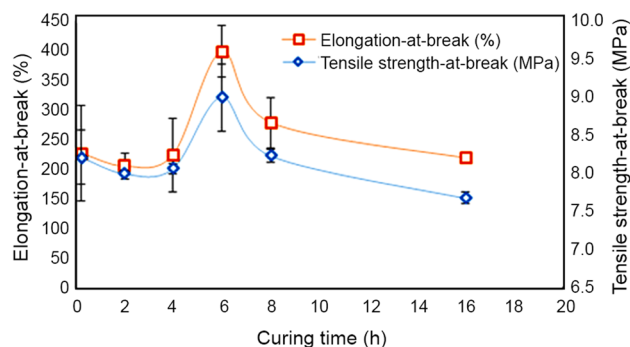


Fig. 5 Effect of curing time on the elongation-at-break and tensile strength of the cross-linked compound

60, and 85 °C, respectively. Study of tensile strength and elongation-at-break of the cross-linked compound showed the maximum respected values of 9 MPa and 397% after 6 h curing time. The curing did not change the hardness, density, and tear strength of the cross-linked compound significantly, but a 12% decrease in the compression set and a 16% decrease in the tension set were observed for the cross-linked compound. After curing, the tensile strength measured by ISO 37: 2011 showed an increase from 5.9 to 7.4 MPa values, but the measurement according to ISO 527: 2012 showed an increase from 6.6 to 7.5 MPa.

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