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ORIGINAL PAPER

Cross-linked poly(ethylene vinyl acetate) (EVA)/low density polyethylene (LDPE)/metal hydroxides composites for wire and cable applications

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Abstract Formulations of chemically cross-linked poly(ethylene vinyl acetate) (EVA) and low density polyethylene (LDPE) blends containing metal hydroxides flame retardants such as aluminum hydroxide (ATH) and magnesium hydroxide (MH) were prepared. Comparison of both type of metal hydroxides in respect of their influence on flammability as well as mechanical, thermal, and electrical properties of EVA/LDPE composites is presented. Most of the investigated properties are better for composites containing MH in comparison with composites containing ATH. Influence of various EVA/LDPE ratios on investigated properties is presented as well. Importance of improving compatibility using compatibilizers to improve some of the investigated properties is described. Polyethylene grafted with maleic anhydride (PEgMA) was found to be better compatibilizer for ATH than vinyl silanes.

Keywords Mechanical properties · Flame retardants · Metal hydroxide · Vinyl silane · PEgMA · Cross-linking

Introduction

Poly(ethylene vinyl acetate)/low density polyethylene (EVA/LDPE) blends are widely used in the wire and cable industry due to their good mechanical and

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physical properties as well as easy processing. However, they have some disadvantages such as low thermal stability, high flammability as well as poor compatibility with additives. Thermal properties of polyolefins can be improved by small addition of stabilizers. A small quantity of processing stabilizers is usually added to prevent oxidative degradation of polyolefins caused by the combined action of shear, heat, and oxygen during their melt processing. Phenolic antioxidants are widely used as processing stabilizers for polyolefins [1, 2]. They act as scavengers of oxygen-centered alkoxy and peroxy radicals, but they are not able to provide long-term thermal stability [3].

Flame retardancy of polyolefins is usually enhanced by physical incorporation of additives. The halogen-containing compounds, despite their high effectiveness in flame retardancy, give rise to problems of toxicity, corrosion, and smoke. Therefore, there is a trend to avoid use of halogenated flame retardants due to environmental and safety concerns. Thus, as main non-halogenated flame retardants, mainly metal hydroxides [4–8] or intumescent flame retardant (IFR) systems [9–16] are used. Metal hydroxides are gaining an increasing importance in the wire and cable industry because of the desirable combination of low cost, low smoke, and relatively high flame retardant efficiency. However, a high loading of metal hydroxides is needed to obtain an adequate level of flame retardant. This leads to deterioration of mechanical properties. Addition of compatibilizers can improve the compatibility of fillers to matrix polymers, and this has a great effect on the mechanical properties of the composites [4, 17].

In order to improve the thermal stability of polyethylene, it is cross-linked in the presence of peroxides or silanes. Generally, cross-linking of cable insulation materials is used to prevent loss of the insulation during overheating. Cross-linking (i.e., vulcanization) of rubber blends is a common practice in the industry, although cross-linking of highly filled polyethylene compounds is hardly used. Addition of silane cross-linkable polyethylene copolymer was used to cross-link and improves the fire retardancy and mechanical properties of the metallic hydroxide filled polyethylenes [18, 19]. Recently, it was also demonstrated that cross-linking can increase tensile strength of polyethylene/ammonium polyphosphate composites and thus, at reasonable yield of cross-linking, improve their mechanical properties and thermal stability [20].

In this article, aluminum and magnesium hydroxide filled EVA/LDPE composites cross-linked by dicumyl peroxides were prepared (Table 1) and their mechanical and thermal properties as well as flame retardancy and volume resistivity are evaluated.

Experimental

Materials

Low density polyethylene (LDPE) HP2022J (MFI = 2.0 g/10 min) from SABIC (Jubail, Saudi Arabia), poly(ethylene vinyl acetate) (EVA) Alcudia PA-538 with 18 wt% of vinyl acetate (MFI = 2.0 g/10 min) from REPSOL YPF (Madrid, Spain)

Table 1 Prepared formulations

Formulation code[a]	LDPE	EVA	ATH (phr)	MH (phr)	PEgMA (phr)	VSi (phr)	oVSi (phr)
M-1	60	40	–	150	–	–	–
M-2	40	60	–	150	–	–	–
M-3	20	80	–	150	–	–	–
M-4	–	100	–	150	–	–	–
A-1	60	40	150	–	–	–	–
A-2	40	60	150	–	–	–	–
A-3	20	80	150	–	–	–	–
A-4	–	100	150	–	–	–	–
A-1-1	60	40	150	–	3	–	–
A-1-2	60	40	150	–	–	3	–
A-1-3	60	40	150	–	–	–	3

[a] All formulations contain also 0.3 phr of Irganox 1010, 0.3 phr of paraffin wax, and 3 phr of DCP

were used as received. Antioxidant, Irganox 1010 from Ciba Specialty Chemicals (Basel, Switzerland); processing aid, paraffin wax from Fluka Chemical Co.(Steinheim, Germany); flame retardants, aluminum hydroxide (ATH) Apyral 30CD (particle size $D_{50} = 1.3$ μm; specific surface area BET = 3.5 m^2/g) from Nabaltec (Schwandorf, Germany) and magnesium hydroxide (MH), modified with a vinyl silane surface treatment, Magnifin H-10A (particle size $D_{50} = 0.8$ μm; specific surface area BET = 10 m^2/g) from Martinswerk (Germany); Polyethylene grafted with maleic anhydride (PEgMA) 12031 from Solvay Co. (Brussels, Belgium); oligomeric Vinyl silane Dynasylan 6498 (oVSi) and monomeric Vinyl silane Dynasylan VTMOEO (VSi) both from Degussa (Germany); cross-linking agent, dicumyl peroxide Perkadox BC-40K from Akzo Nobel (Arnhem, The Netherlands), were procured and used as received.

Compounding and cross-linking

All additives except dicumyl peroxide were mixed with resins for 10 min at 150 °C using an internal mixer (Model 350 S) from Brabender Co., Duisburg, Germany with speed of 50 rpm. Then the mixture was moved to two-roll-mill from Brabender Co., Duisburg, Germany and dicumyl peroxide was added at 110 °C and mixed for 3–5 min. Cross-linking was performed by molding sheets for 30 min at 170 °C. Sheets with thickness 2 mm were prepared and used for all testing methods.

Testing methods

Tensile strength and elongation at break before and after thermal aging were measured using a universal testing machine from Instron Co., Canton Massachusetts, USA in accordance with ASTM D 638M (test speed of 50 mm/min).

Thermal aging of samples was performed at 135 °C for 168 h using a heat aging oven in accordance with IEC 60811-1-2.

Flammability of prepared formulations was characterized by limiting oxygen index (LOI) flammability test as well as by Cone calorimeter. LOI test was performed using an apparatus from Fire Testing Technology Limited (Incorporating Stanton Redcroft, London, UK) in accordance with ISO 4589 (ASTM D2863). Cone calorimeter from Fire Testing Technology Limited (Incorporating Stanton Redcroft, London, UK) was used to measure heat release as well as smoke production in accordance with ASTM 1354-04a under a heat flux of 50 kW/m^2, which corresponds to the heat evolved during a fire.

Volume resistivity was measured using a high resistance meter, Model HP4339B in accordance with ASTM D257-99.

Results and discussion

Mechanical properties

Mechanical properties of prepared formulations are shown in Table 2 and Figs. 1, 2. Tensile strength of prepared composites depends strongly on the type of metal hydroxides (see Fig. 1). In the case of MH, tensile strength is comparable with tensile strength of pure EVA, although generally addition of high amount of fillers leads to a decrease of tensile strength. Moreover no change is observed with the change of EVA content. High tensile strength regardless of EVA content (over the investigated range) can be attributed to good compatibility between filler and resin as well as improvement due to cross-linking. On the other hand, in the case of ATH, the tensile strength is lower than that of pure resin and it decreases unexpectedly

Table 2 Mechanical properties and retention of mechanical properties after thermal aging of prepared formulations

Formulation code	TS (MPa)	Eb (%)	TS after aging (MPa)	Retention of TS (%)	Eb after aging (%)	Retention of Eb (%)
LDPE	11.9 ± 0.7	496 ± 30	–[a]	–[a]	–[a]	–[a]
EVA	21.2 ± 1.9	736 ± 32	–[a]	–[a]	–[a]	–[a]
M-1	19.5 ± 0.5	75 ± 7	19 ± 1	95	54 ± 7	79
M-2	20 ± 1.8	88 ± 8	19.5 ± 0.6	98	82 ± 5	93
M-3	20.3 ± 1.3	118 ± 10	19.6 ± 0.1	94	93 ± 3	79
M-4	19 ± 0.5	138 ± 12	20 ± 0.8	105	130 ± 14	94
A-1	12 ± 0.5	35 ± 3	12 ± 1.2	100	9.2 ± 3.3	26
A-2	10.7 ± 0.2	35 ± 3	10 ± 0.4	93	7 ± 2	20
A-3	10 ± 0.4	59 ± 5	11 ± 0.1	110	7.2 ± 6	12
A-4	9 ± 0.1	77 ± 15	7.5 ± 0.6	83	14.5 ± 7	18
A-1-1	15.8 ± 1.2	155 ± 14	15.2 ± 0.3	96	122 ± 13	79
A-1-2	14.6 ± 0.9	108 ± 9	9.2 ± 1.3	63	7 ± 3	6
A-1-3	14.5 ± 0.3	113 ± 13	14.4 ± 1.4	99	93 ± 10	82

[a] Samples were destroyed during thermal aging

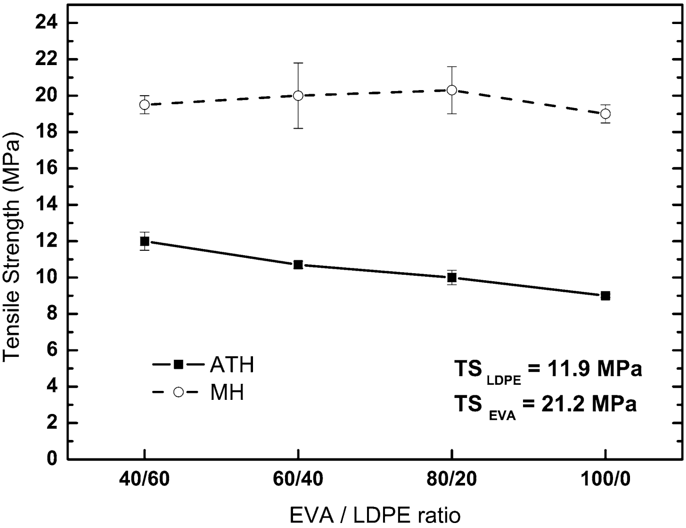

Fig. 1 Tensile strength of chemically cross-linked EVA/LDPE/metal hydroxides formulations as a function of EVA/LDPE ratio

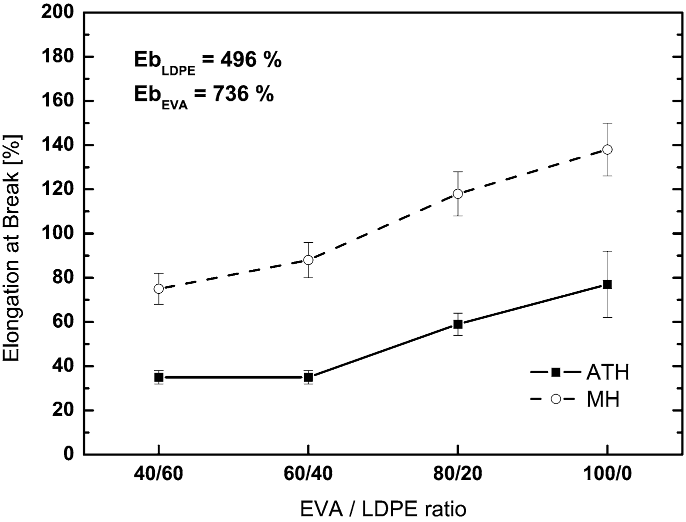

Fig. 2 Elongation at break of chemically cross-linked EVA/LDPE/metal hydroxides formulations as a function of EVA/LDPE ratio

with increase of EVA content. It should be mentioned that ATH—Apyral 30CD is not surface modified unlike MH—Magnifin H-10A, which is modified by vinyl silane treatment. It was described recently that modification of the metal hydroxide surface can improve its dispersion in polymer matrix and so increase also tensile strength [17]. Thus, it can be supposed that worse tensile strength in ATH composites can be caused by bad dispersion of ATH in a resin. Therefore, three various compatibilizers such as poly(ethylene grafted by maleic anhydride) (PEgMA), vinyl silane, and oligomeric vinyl silane were used in order to improve compatibility of ATH with the resin. From Table 2, it is shown that the use of the three types of compatibilizers improved tensile strength of ATH composites. The best improvement was found when PEgMA was used as a compatibilizer. However, the achieved tensile strength was still much lower (15.8 MPa) compared with the same EVA/LDPE ratio composites containing MH, where the tensile strength was 19.5 MPa. One could imagine that this difference could be attributed to the particle size of 0.8 μm for MH compared to 1.3 μm for ATH. However, it was recently described that although the particle size of ATH, in the range 0.8–1.7 μm, has influence on the elongation at break in EVA/ATH composites, there was no influence on the tensile strength [21].

Generally, the addition of high amount of fillers to a resin and subsequent cross-linking of the compound lead to decrease in elongation at break [17]. From Fig. 2 and Table 2, it is observed that elongation at break is different for ATH and MH composites. In both cases a trend of increase in the elongation at break with increase of EVA content is evident, probably because of better compatibility with EVA resin [22] as well as the inherited high elongation at break of pure EVA resin in comparison with pure LDPE. The elongation at break for MH composites is about twice as high as that of ATH composites. This can be attributed again to surface modification of MH. Therefore, elongation at break of ATH composites containing various compatibilizers was compared as well. As seen from Table 2, elongation at break of ATH composites was considerably increased after addition of compatibilizers. The elongation at break of ATH composites containing vinyl silane as a compatibilizer was higher than modified MH. Further increase in elongation at break was obtained with oligomeric vinyl silane and the best value, 155%, was achieved with PEgMA. Thus, PEgMA seems to be a better compatibilizer for EVA/LDPE/metal hydroxides composites than monomeric or oligomeric vinyl silanes.

Thermal stability

Thermal stability of prepared formulations was characterized by retention of tensile strength and elongation at break after thermal aging at 135 °C for 168 h. Unlike the uncross-linked pure LDPE and EVA resins, which were destroyed during thermal aging, all cross-linked formulations demonstrated reasonable retention of mechanical properties after thermal aging. As seen from Table 2, all prepared formulations exhibit very good retention of tensile strength of 100 ± 20%. However, retention of elongation at break, which is more sensitive and therefore more important for evaluation of thermal properties, was different for ATH and MH composites. While all MH formulations exhibit very good retention of elongation at break of at least about 80%, a very poor retention, <30%, was observed for formulations containing ATH without compatibilizer or ATH with vinyl silane as a compatibilizer. Retention of elongation at break, however, was adequately improved by mixing ATH with oligomeric vinyl silane or PEgMA. Thus, it can be seen, that although cross-linking can improve thermal stability of composites, the use of a proper compatibilizer is very important as well. Thus, good compatibility of highly filled resins can lead to more homogenous cross-linking, which is important for thermal stability of composites.

Flammability

Flammability of prepared formulations was tested by two methods including LOI and cone calorimeter measurements. LOI test is widely used to evaluate flame retardancy of materials, especially for screening flame retarded formulations of polymers. It can be seen from Table 3 that addition of metal hydroxides to the EVA/LDPE blend followed by cross-linking leads to significant increase in LOI and values from 28 up to 37% were obtained, depending on the composition of formulations, compared to 18 or 19% for pure LDPE and pure EVA, respectively. In the case of formulations containing MH, LOI of at least 31% is achieved and it increases with increase of EVA content (Fig. 3). In the case when EVA was used alone, LOI as high as 37% was achieved. Similar increase in LOI with increase of EVA content is observed for formulations containing ATH, however, the values are about 1–3% of oxygen in volume lower than those of MH contained formulations. Mixing of ATH with compatibilizers leads to a decrease in LOI of about 2% of oxygen in volume. Thus, although compatibilizer can significantly improve mechanical properties of EVA/LDPE/ATH composites, it has negative influence on LOI. From these results, it is clear that in cross-linked EVA/LDPE/metal hydroxides composites, MH inhibits combustion more effectively than ATH. This phenomenon can be explained by the fact that MH has a high endothermic decomposition temperature (>340 °C) in comparison with ATH, which begins to decompose at 180–200 °C [21]. Thus, MH may perform better than ATH in flame

Table 3 Flammability of prepared formulations based on LOI and cone calorimeter measurements

Formulation code	LOI (%)	THR[a] (MJ/m^2)	TTI (s)	PRHR (kW/m^2)	TSR[a] (m^2/m^2)
LDPE	18.0	2.76	43	666	19.1
EVA	19.0	2.52	33	803	25.5
M-1	31.2	1.58	71	210	11.7
M-2	33.8	1.58	72	193	13.6
M-3	35.0	1.85	74	234	14.1
M-4	37.3	1.66	76	230	14.1
A-1	30.5	2.31	68	415	16.5
A-2	30.1	2.40	73	460	18.0
A-3	31.1	2.22	72	420	17.7
A-4	34.1	2.32	78	323	16.4
A-1-1	28.4	1.68	55	417	11.9
A-1-2	27.7	NT	NT	NT	NT
A-1-3	27.7	NT	NT	NT	NT

NT not tested, *LOI* limiting oxygen index, *THR* total heat release, *TTI* time to ignition; *PHRR* peak of heat release rate, *TSR* total smoke release

[a] Values are normalized to 1 g of a tested material

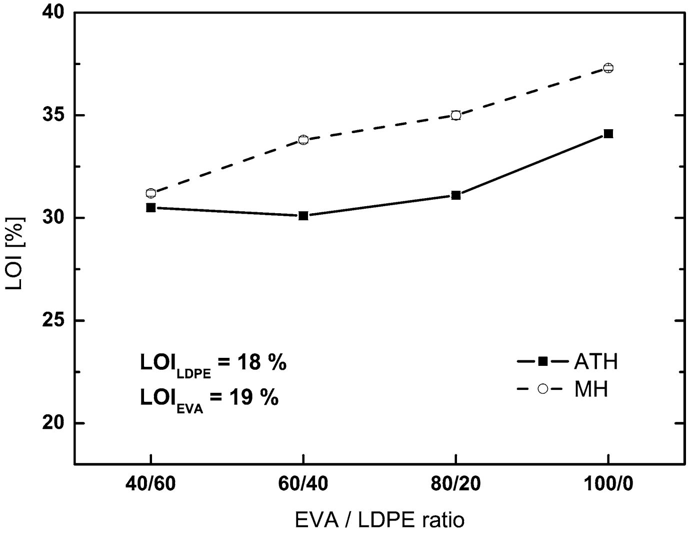

Fig. 3 LOI (%) of chemically cross-linked EVA/LDPE/metal hydroxides formulations as a function of EVA/LDPE ratio

retardancy of polyolefins because it decomposes almost at the same temperatures as polyolefins.

Some more information about dynamic flammability of prepared formulations can be gained from cone calorimeter measurements. Some of the results of cone calorimeter have been found to correlate well with those obtained from large-scale fire tests and can be used to predict the behavior of materials in real fires [23, 24]. All basic characteristics from cone calorimeter are presented in Table 3 and sample in the course of burning showing a comparison between pure resins and various metal hydroxides composites is shown in Fig. 4.

As presented in Table 3, a decrease in total heat release (THR) in comparison with pure resins is observed for all prepared metal hydroxides composites and the value of THR depends on the type of metal hydroxide, and it is independent of EVA content. In the case of MH contained formulations a significant decrease in THR of 60% is achieved. On the other hand, only a slight decrease in THR is observed in the case of ATH contained formulations. However, mixing of ATH with compatibilizer such as PEgMA leads to a significant decrease in THR similar to those of MH composites. Since this trend was not observed when PEgMA was added to IFR composites in our previous work [25], this observation indicates that the decrease in THR in ATH composites after addition of PEgMA is due to improved compatibility between ATH and resin. Better compatibility with metal hydroxide leads to homogenous mixing and distribution of additives and consequently more effective inhibition of combustion.

The course of burning (see Fig. 4 and Table 3) shows that the time to ignition (TTI) increases from 43 and 33 s for pure LDPE and pure EVA, respectively, to 68–78 s for EVA/LDPE/metal hydroxides formulations and it increases with

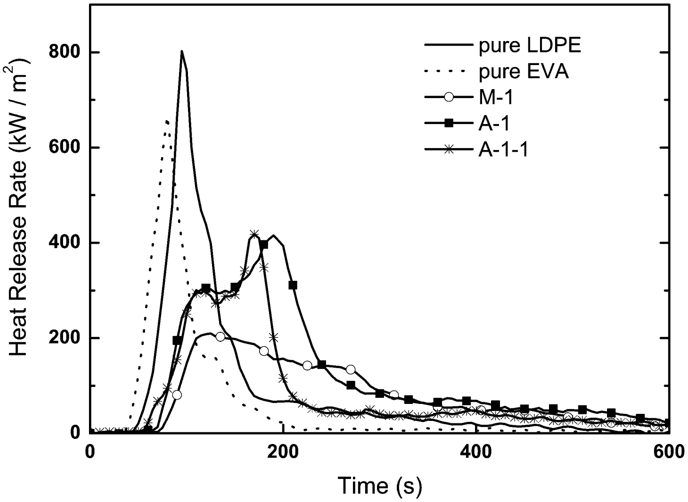

Fig. 4 Heat release rate of pure LDPE, pure EVA, and EVA/LDPE/metal hydroxides formulations containing EVA/LDPE resin ratio 40/60 in combination with magnesium hydroxide (M-1), aluminum hydroxide and aluminum hydroxide (A-1) modified with PEgMA compatibilizer (A-1-1) as a function of burning time

increase of EVA content. Almost the same TTI is observed for MH and ATH contained formulations. After ignition, there are high sharp peaks of heat release rate (HRR) for pure LDPE and EVA (see Fig. 4), however, after addition of flame retardant this peak decreases followed by different course of burning for MH and ATH formulations. In the case of MH formulations a peak maximum as high as 200 kW/m^2 is observed after ignition followed by slow decrease of HRR. In the case of ATH formulations a first peak maximum as high as 300 kW/m^2 is observed shortly after ignition, followed by a second peak maximum as high as 400 kW/m^2 after about 80 s from the first peak maximum, followed by rapid decrease in HRR. Mixing of ATH with compatibilizer such as PEgMA gives almost no change in the course of burning of HRR where a decrease in TTI and time to the peak maximum was only observed. Thus, the difference in course of burning between MH and ATH formulations is not attributed to better compatibility of MH, but it can be attributed to higher thermal stability of MH in comparison with ATH similar to studies on flammability tests with LOI.

A decrease in smoke production in comparison with pure LDPE and EVA resins was observed for EVA/LDPE/metal hydroxides in the case of both MH and ATH contained formulations. It is well known that metal hydroxides serve not only as flame retardants, but also as smoke suppressants. It was also proved that not only metal hydroxides, but also metal oxides formed from metal hydroxides during the combustion lead to smoke suppression. Metal oxides can catalyze the oxidation of carbon, resulting in smoke reduction [22, 26]. From Table 3, more significant decrease in total smoke release (TSR) for MH formulations compared with ATH formulations can be seen. However, when ATH was mixed with compatibilizer such as PEgMA, higher decrease in TSR compared to that of MH contained formulations

Table 4 Volume resistivity of prepared formulations

Flame retardant	Volume resistivity (Ω cm)			
	Resin ratio			
	EVA/LDPE 40/60	EVA/LDPE 60/40	EVA/LDPE 80/20	EVA/LDPE 100/0
MH	2.04×10^{15}	4.22×10^{15}	1.95×10^{15}	3.35×10^{15}
ATH	1.98×10^{14}	3.16×10^{14}	2.68×10^{14}	2.60×10^{14}

Note: Volume resistivity of pure LDPE and pure EVA is 2.29×10^{16} and 0.93×10^{16}, respectively

is observed. It is rational that better compatibility and thus more homogenous distribution of additives in the composites can lead to more effective smoke reduction.

Electrical properties

Volume resistivity for most of the formulations was investigated as well (see Table 4). Pure LDPE has generally very good electrical properties whereas slightly lower volume resistivity is observed for pure EVA. Generally for filled cross-linked LDPE compounds used for cable insulations (usually used for 0.6/1 kV cables), volume resistivity should be at least of order 10^{14} Ω cm. As shown in Table 4, all cross-linked formulations containing MH have volume resistivity more than one order higher than value required for cable insulations application. The ATH contained formulations have volume resistivity one order lower than MH contained formulations, however, the values for both are still acceptable.

Conclusions

EVA/LDPE cross-linked composites containing metal hydroxides such as MH or ATH with high flame retardancy, good mechanical, thermal, and electrical properties were prepared. Cross-linking of prepared composites by chemical method using DCP prevented destruction of samples upon heating at 135 °C and provided good retention of mechanical properties after thermal aging. Using a compatibilizer for better dispersion of metal hydroxides in the resin improved not only mechanical properties of the composites, but also their thermal properties and led to reduction of smoke production. Among the modified metal hydroxides, the EVA/LDPE/MH composites had better flame retardancy, higher tensile strength, and higher volume resistivity than EVA/LDPE/ATH composites. In cross-linked EVA/MH composite, a LOI as high as 37% was achieved. MH as a filler for cross-linked EVA/LDPE composites was found to give superior properties for wire and cable application in comparison with ATH.

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