TPV Based Insulation for Medium Voltage Applications

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Abstract

In this work we present the development of three fully thermoplastic lead free medium voltage (MV) insulation compounds based on the thermoplastic vulcanizate (TPV) technology. The TPV MV insulation compounds were prepared starting from a peroxide curable lead free MV insulation, which is the actual market benchmark. For this reason, they were extensively investigated in comparison to the standard lead free MV insulation. To evaluate the results of the dynamic vulcanization process, the compounds were studied by means of differential scanning calorimetry (DSC). To simulate the extrusion behavior, their rheology was investigated. Mechanical properties were measured before and after ageing at 135°C and 150°C up to 21 days. Finally, a comprehensive study on their electrical features, in dry (from 25°C to 90°C) and wet (up to 28 days at 90°C in water) conditions, is presented.

Keywords: Thermoplastic Vulcanizate (TPV); Medium Voltage Insulation; Lead Free.

Figure 1. MV insulation containing lead (orange) and lead free (white), from pellets to cables

1. Introduction

Twenty years ago both XLPE and EPDM based insulation systems were used in many parts of the world for MV cable applications. North America remains a very active market for EPDM based MV insulations, while in other parts of the world the market for EPDM insulations has decreased with XLPE being preferred. Recently, we are seeing a renewed interest in EPDM based MV insulations in the global market due to the unmatched performance in cables lifetime for long term applications (>20 years). Since 1996, Mixer Spa has produced MV insulation compounds based on EPDM and EPDM/LDPE blends: our strategy is to offer innovative and competitive materials to the cable market, believing that the continuous improvement of materials will give a new life to rubber cables for special applications. The first step of this approach was the development of lead free EPDM solutions, which were presented in 2012 and are now commercially available (see Figure 1) [1]. Due to the fact that lead salts are insoluble in water and therefore not contributing to any leakage current through the insulation layer, lead oxide is one of the most effective additive in MV insulation compounds. However, lead oxide is listed in Reach SVHC (Substances of Very High Concern) for its well-known bioaccumulation risk and long lasting effects, leading severe damages to environment and life [2]. In Mixer, we have successfully replaced lead oxide with an inorganic ion scavenger system capable of immobilizing ions, succeeding in the production of EPDM based lead free MV insulation compounds with superior thermal and electrical stability. From this starting point, we have developed a new fully thermoplastic TPV for MV insulation dynamically crosslinking our lead free MV insulation compound in a PP matrix. We present three upgrades of MV TPV compounds towards a material able to pass thermomechanical testing for 90°C and 105°C continuous operation temperature and 250°C short circuit emergency, according to the Italian norm CEI 20-86, which is, so far, the only norm on thermoplastic compounds for MV insulation.

Firstly, we discuss the preparation and the macroscopic properties of the novel MV TPV compounds. Secondly, we have investigated the novel MV TPV compounds by means of DSC to study the dynamic vulcanization process. In the third part, the rheology of the MV TPV was analyzed at low shear to simulate their extrusion behavior. Subsequently, the MV TPV compounds were tested for mechanical properties before and after heat ageing up to 150°C and 21 days. Electrical properties of the compounds were studied at Imerys laboratories, Par, England. In detail, loss factor (Tanδ), dielectric constant (ε_r) and volume resistivity were measured up to 90°C in dry conditions. Additionally, Tanδ and ε_r were investigated after immersing the compounds in water at 90°C up to 28 days. Test results were compared to the standard lead free MV IS79 demonstrating that we can offer an innovative, highly electrically insulating compound that combine simultaneously the properties of our lead free XL-EPDM compound with the possibility to process it as a thermoplastic material.

2. Lead free MV TPV compounds

2.1 Preparation of the MV TPV compounds

Lead free MV insulation compound, MV IS79, and MV thermoplastic vulcanizate compounds, MV TPVs, were prepared in an internal mixer equipped with two counter-rotating rotors and a chamber with 8 cm^3 volume. The composition of the MV TPV compounds is summarized in Table 1. Obviously, MV TPV79 A and B have the same ratio between elastomeric and thermoplastic phase, nonetheless, different co-agents were utilized in their formulation. This was done following the studies on co-agents influencing the properties of TPVs compounds by preventing the decomposition of PP via β-scission caused by free radicals [3].

Table 1. Formulation of the MV TPVs

TPV Composition	MV TP79 A	MV TP79 B	MV TP79 C
MV IS79	75%	75%	70%
PP-1[1]	25%	25%	20%
PP-2[2]	-	-	10%

[1]d = 0.891 gr/cm^3, MFI (230 °C; 2.16 kg) = 8.0 gr/10min; [2]d = 0.900 gr/cm^3, MFI (230 °C; 2.16 kg) = 10.0 gr/10min.

MV IS79 was prepared by mixing all the components in the internal mixer leading to a complete blending of the ingredients. After unloading, peroxide was added at low temperature in a two roll mill. Samples for testing were obtained by pressing the milled sheets in a compression molding machine at 180°C for 10 minutes. Specimens for mechanical properties were die cut in the milling direction.

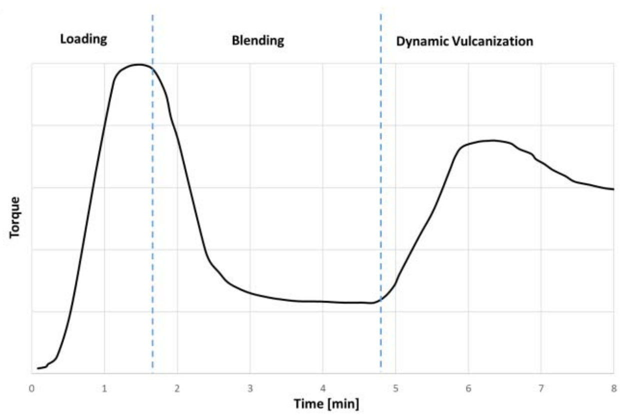

Figure 2. Representation of the torque pattern in function of time during the production of the MV TPV compounds. The three main steps of the process are indicated.

MV TP79 compounds were prepared by mixing the lead free compound (MV IS79) with thermoplastic polypropylene (PP) according to the ratio showed in Table 1. During the mixing process, as the radical reaction takes place, while the temperature rises continuously, the torque follows a characteristic pattern, which is graphically represented in Figure 2 [4,5]. After loading the ingredients, the torque grows due to the high viscosity of the components at low temperature. Increasing the temperature, the materials start to soften, the torque drops while the blending takes place. As the radical reaction begins, the simultaneous crosslinking of rubber phase and β-scission of PP phase occurs, with consequent phase inversion leading to the torque rapidly increasing. The final temperature, at which the TPVs were unloaded after about 8 minutes of processing, was between 200°C and 220°C. The still hot compounds were calendered in a two roll mill in sheet shape; plaques were obtained by pressing the sheets in a compression molding machine at 180°C for one minute. Specimens for mechanical properties were die cut in the milling direction.

As reported in Table 2, all the compounds show comparable mechanical properties, namely tensile strength (TS), elongation at break (EB) and TS at 200% elongation.

Table 2. Typical physical properties of the MV insulation compounds

	MV IS79	MV TP79 A	MV TP79 B	MV TP79 C
TS[1] [N/mm^2]	16.61	17.31	17.19	15.73
EB[1] [%]	321	360	310	341
TS @ 200% [N/mm^2]	14.23	13.57	14.48	13.62
HS[2] [Shore A-D]	80-/	96-45	95-46	96-48
MFI[3] [gr/10min]	27.6[4]	4.4	4.2	21.3

[1]ASTM D412; [2]ASTM D2240; [3]ASTM D1238 (190°C, 21.6 kg), [4] Measured on the compound without peroxide

The choice of PP and its ratio seem not to influence greatly the mechanical properties, which are close to the standard MV IS79. On the contrary, the crystallinity of PP leads to a conspicuous increment of hardness (HS), which is 48 Shore D for MV TP79 C, i.e. the compound with the highest content of PP. Due to the high viscosity of MV TP79 A and B, the melt flow index (MFI) was measured at 190°C with 21.6 kg weight. Their low flow rate can be ascribed principally to two main factors: the ratio between thermoplastic and elastomeric phases and the choice of a PP with low MFI at the test temperature. However, it can be noted that, by a careful balancing of the ratio between the two phases and an accurate choice of PP, we were able to obtain a MFI for MV TP79 C comparable to the standard MV IS79. Those results are confirmed by the rheological studies presented in the section 2.3.

For the sake of comparison and to highlight the successful achievement of the MV TPV compounds, reference materials without peroxide were produced. Thereby, in those compounds, the dynamic vulcanization could not take place after the blending of the components. The reference compound MV Ref AB, has the same composition of MV TP79 A and B (without peroxide and co-agents), the reference compound MV Ref C was formulated as MV TP79 C (without peroxide). Rheology and mechanical properties of both the reference compounds were analyzed in comparison to the MV TPV compounds presented in this paper to demonstrate our capability to obtain TPV compounds in a reproducible and controlled fashion.

2.2 DSC analysis

In order to determine the unreacted peroxide remaining in the compounds after the curing process, DSC was implemented. The spectra were measured in a Perkin-Elmer DSC 6000 in inert nitrogen atmosphere from 0°C to 230°C with a heating rate of 20°C/min, after heating the samples were cooled down to 0°C with 10°C/min rate. This cycle was repeated three times. However, as the aim of this study was to quantify the ratio between initial and residual (after curing or dynamic vulcanization) peroxide, only the first heating cycle is presented and discussed in the following.

Firstly, the uncured MV IS79 containing 100% of unreacted peroxide was analyzed and used as reference. From the DSC showed in Figure 3, the calculated enthalpy of reaction (ΔH) given by the peroxide decomposition was -8.97 J/g. In the same figure is represented the DSC plot of the cured MV IS79 (10 minutes at 180°C). A ΔH of -1.16 J/g was detected, corresponding to a residue of about 13% of unreacted peroxide. This indicates that MV IS79 was almost completely vulcanized. In the same way, the amount of unreacted peroxide of the MV TPV compounds was computed, considering that MV TP79 A, B and MV TP79 C were formulated with 75% and 70% of uncured MV IS79, respectively.

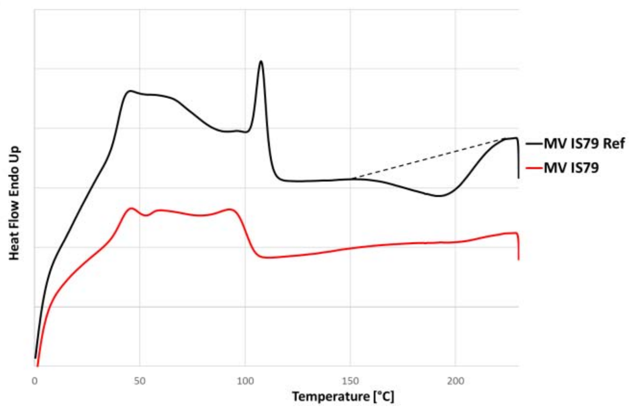

Figure 3. DSC analysis of uncured (top) and cured (bottom) MV IS79. Dotted line: graphical representation of the baseline used to compute the reaction enthalpy.

From the data collected and showed in Figure 4, the residual peroxide detected in MV TP79 A was about 4% (ΔH = -0.27 J/g) and in MV TP79 B was about 5% (ΔH = -0.33 J/g). For MV TP79 C the computed residual peroxide was around 11% (ΔH = -0.68 J/g). Those results, confirm beyond any doubt the almost complete decomposition of the initial peroxide during the dynamic vulcanization.

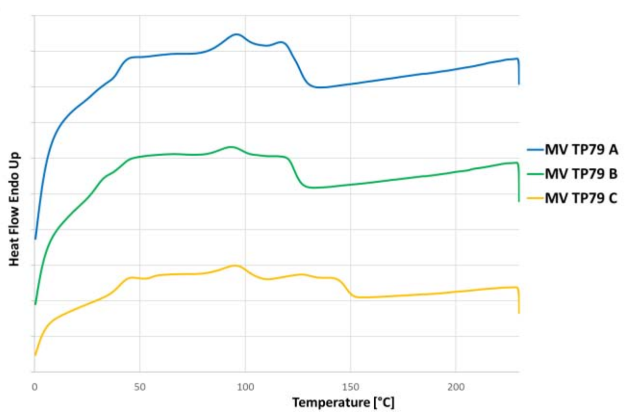

Figure 4. DSC analysis of MV TP79 A (top), MV TP79 B (middle) and MV TP79 C (bottom).

2.3 Rheology

Rheological studies are fundamental to predict the extrusion behavior of compounds. As such, we have investigated the rheology at apparent shear rates from 200 s^{-1} to 1 s^{-1} in a Göttfert Rheograph 2002 capillary rheometer. The L/D of the capillary was 30 and measurements were carried out at 180°C. The temperature was chosen to allow the complete fusion of the PP. Normally, standard compounds as MV IS79 are characterized at 125°C before the curing step, however, at this temperature the PP is not molten resulting in misleading results. Due to the high test temperature, to prevent the decomposition of the peroxide during the analysis, MV IS79 was investigated without peroxide. As aforementioned, the reference compounds MV Ref AB and C, were included in this study to underline the change of rheological behavior as a consequence of the dynamic vulcanization. The plots of the apparent shear stress in function of the apparent shear rate are showed in Figure 5.

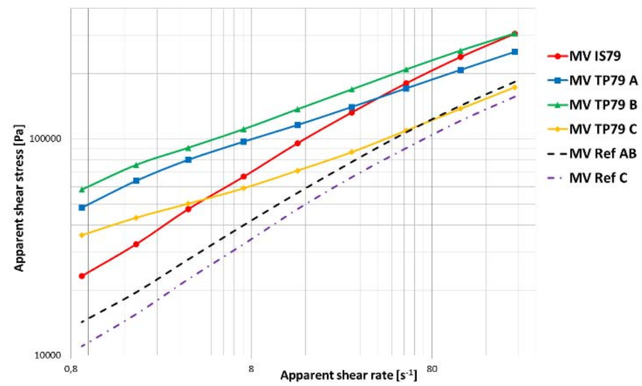

Figure 5. Apparent shear stress in function of apparent shear rate measured at 180°C of the MV insulation compounds. Dotted lines: reference compounds.

The response of MV IS79 is typical of EPDM/PE based compounds: the shear stress diminishes rapidly in an almost linear fashion decreasing the shear rate. Small deviations from a perfect linearity can be noted and are usually ascribed to EPDM rubbers. MV Ref AB and C exhibit the same pattern with the shear stress translated toward lower values. This effect is caused by the thermoplastic phase, which shows lower viscosity at this temperature. Accordingly, by increasing the content of PP the shear stress decreases. Owing to the different nature of the MV TPV compounds, their rheological behavior is rather different [6,7]. Essentially, such a dissimilar character stem from the elastic response of the elastomeric crosslinked particles, which is dominant at low shear stresses. On the contrary, at high shear stresses, the behavior of the TPV compounds is governed by the thermoplastic phase. As a result, the three MV TPV compounds have a similar behavior to the reference compounds at high shear rates. Diversely, at low shear rates, the curves are clearly divergent.

Focusing only on the MV TPV compounds, as noted previously for the MFI in Section 2.1, by careful balancing the components and a correct choice of PP, it is possible to "tune" the rheological behavior of the TPV MV compounds keeping or even improving the thermomechanical properties. In this regard, MV TP79 C exhibits lower stresses, i.e. viscosity, until very low shear rates together with the best thermomechanical properties among the studied TPV MV compounds.

2.4 Mechanical testing

The stress strain properties of the MV insulation compounds were measured according to the method ASTM D412 averaging the results of five dumb-bell test specimens obtained in a Gibitre Tensor Check Profile. The specimens were die cut along the milling direction from plaques obtained in a compression molding machine at 180°C. MV IS79 was pressed 10 minutes to complete the curing process. MV TP79 A, B and C were pressed for 1 minute and cooled down under pressure. MV Ref AB and C were treated identically to the MV TPV compounds to obtain the test specimens. Figure 6 illustrates one example of the stress strain curve for each compound.

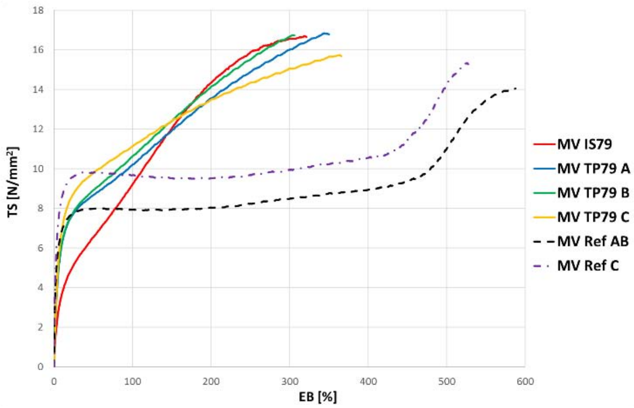

Figure 6. Stress strain plots of the MV insulation compounds. Dotted lines: reference compounds

At first sight, the analysis of the stress strain curves of the materials reveals that the MV TPV compounds have similar performance to the benchmark MV IS79 in terms of TS and EB, as already pointed out in section 2.1. Besides the absolute values, the outlined curves follow a similar pattern with a strong elastic response to the stress applied. The main difference which can be observed is the higher Young's modulus of the MV TPV compounds. This is caused by the crystallinity of the thermoplastic phase and therefore larger for MV TP79 C. The same behavior is recognizable in the reference compound MV Ref AB, which has a Young's modulus virtually identical to MV TP79 A and B. Likewise, MV Ref C has a similar Young Modulus compared to MV TP79 C. However, those reference compounds, not being vulcanized and lacking the elastic character, yield until the final rupture. In contrast, the MV TPV compounds behave as crosslinked materials with high elongation [8-10]. These results are in agreement with the rheological studies, confirming the successful achievement of thermoplastic vulcanizate compounds.

Table 3. Hot pressure test and longitudinal shrinkage at 130°C of the MV TPV compounds

	MV TP79 A	MV TP79 B	MV TP79 C
Hot Pressure Test[1] [%]	n.a.[2]	27	3
Longitudinal shrinkage[1] [%]	14	11	2

[1]CEI 20-86; [2]Not applicable.

According to CEI 20-86, to evaluate the performance of the MV TPV compounds at high temperature, we have carried out the hot pressure test and the longitudinal shrinkage at 130°C summarized in Table 3, which are mandatory for thermoplastic insulating materials rated for 90°C and 105°C. The results show an improvement of the results going from MV TP79 A to MV TP79 C. However, this is not a consequence of the ratio between thermoplastic and elastomeric phase but it results from the addition of a PP (see Table 1), which can withstand such high temperatures.

2.4.1 Heat ageing resistance. MV insulation compounds were tested at 135°C and 150°C for 168, 240 and 504h, to assess their resistance to accelerated ageing. Retained TS and EB are graphically showed in Figure 7 and Figure 8. MV TP79 A and B could not be tested at 150°C, as the thermoplastic phase completely melts at this temperature. In this regard, MV TP79 C, which contains PP with higher melting temperature, represents the only alternative to MV IS79 at the test temperature of 150°C.

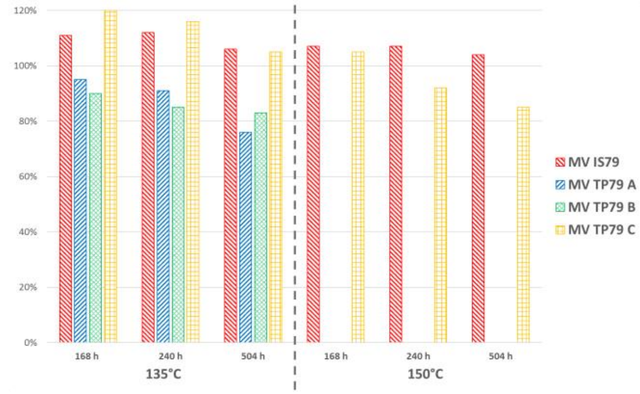

Figure 7. Tensile strength retained after air ageing at 135°C and 150°C for 168 h, 240 h and 504 h.

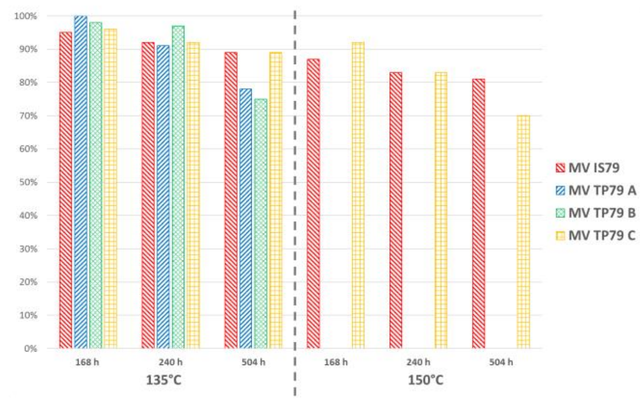

Figure 8. Elongation at break retained after air ageing at 135°C and 150°C for 168 h, 240 h and 504 h.

First, it must be pointed out that all the compounds have good to excellent resistance at 135°C in term of retained TS and EB, which are higher than 70% after 504h. Both MV IS79 and MV TP79 C excellently withstand the heat ageing at 135°C, achieving retained TS and EB > 90%. Although the heat resistance performance slightly decays in comparison to MV IS79, MV TP79 C exhibits a TS retained > 80% and a EB retained ca 70% after 504h at 150°C. Our tests indicate that MV TP79 C can withstand the same ageing conditions of MV IS79. It has to be considered that MV IS79 is rated for a service temperature of 105°C and therefore routinely tested for 508 h at 150°C with typical values of TS and EB retained of 95% and 75%. According to CEI 20-86, MV insulation compounds must withstand ageing for 240 h at 135°C and 150°C for service temperature rating of 90°C and of 105°C, respectively. Thus, MV TP79 C represents a valid thermoplastic alternative to standard lead free elastomeric MV insulation compounds.

2.5 Electrical performance

Insulating properties of the compounds were estimated by measuring loss factor (Tanδ), dielectric constant (ε_r) and volume resistivity in function of temperature from 25°C to 90°C in dry conditions. In addition, loss factor and dielectric constant were measured after immersing the compounds in water at 90°C for up to 28 days. The electrical properties were measured on 2 mm thick press molded samples. An Omicron MI600 System was utilized to evaluate Tanδ and ε_r; a QuadTech model 1868A was implemented in investigating volume resistivity. All the electrical properties of the compounds were studied at the Imerys laboratories.

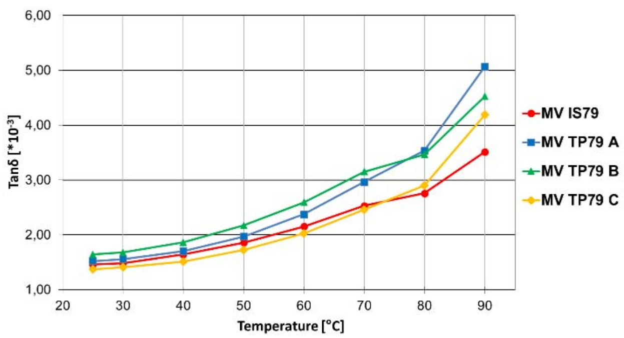

Figure 9. Loss factor (Tanδ) in function of temperature at 500 V and 50 Hz.

Figure 9 shows the plot of Tanδ from 25°C to 90°C in dry conditions. The four compounds are characterized by small variations of the loss factor, which remains in the same order of magnitude (10^{-3}) up to 90°C. Furthermore, all the compounds present a similar trend of Tanδ increasing the temperature. In more detail, the loss factor of the four compounds is virtually identical at room temperature, about $1.5 \cdot 10^{-3}$, and grows steadily with the temperature to values between $3.5 \cdot 10^{-3}$ and $5.0 \cdot 10^{-3}$ at 90°C for MV IS79 and MV TP79 A, respectively.

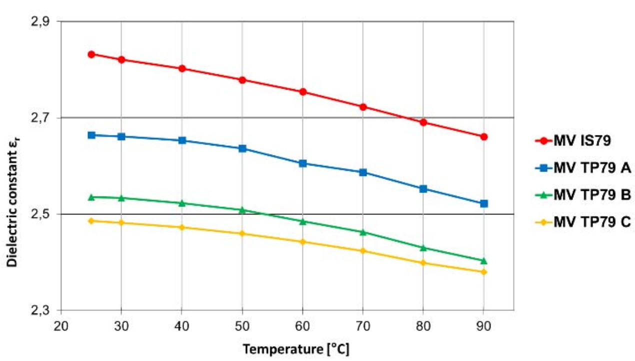

Figure 10. Dielectric constant (ε_r) in function of temperature at 500 V and 50 Hz.

As described for Tanδ, ε_r varies in a narrow range for all the compounds raising the temperature. In Figure 10, only a small lowering of the dielectric constant is observed increasing the temperature. As ε_r is calculated through the following formula:

$$\varepsilon_r = \left(\frac{C}{\varepsilon_0}\right)\left(\frac{t}{A}\right) \quad (1)$$

in which C is the capacitance measured by the instrument and ε_0 is the permittivity of vacuum, while t and A are geometrical factors indicating the separation between the plates (electrodes) and their area, respectively. The lower dielectric constant of the MV TPV compounds in comparison to MV IS79 is given by their content of PP, which is increasing the insulation performance of the overall compound. As a consequence, MV IS79 is characterized by the larger dielectric constant in contrast to MV TP79 C characterized by the lower. However, it has to be pointed out that the difference between the compounds is rather limited at either low or high temperature.

Table 4. Volume resistivity measured at 25°C and 90°C with 500 V potential

Volume Resistivity [$*10^{14}$]	MV IS79	MV TP79 A	MV TP79 B	MV TP79 C
At 25°C [Ω-cm]	47.0	41.6	41.3	50.3
At 90°C [Ω-cm]	2.54	0.378	0.284	0.321

Lastly, the volume resistivity was measured at 25°C and 90°C applying a potential of 500 V (see Table 4). At 25°C, all the compounds have a volume resistivity in the order of magnitude of 10^{15} Ω-cm, which is standard value for MV insulants. At 90°C the volume resistivity of the MV TPV compounds is about one order of magnitude lower than that of MV IS79. Most probably, this difference results from a partial melting of the thermoplastic phase of the TPV compounds, which leads to a higher mobility of the charge carriers in the material. However, besides this, the volume resistivity of the four MV TPV compounds is above 10^{13} Ω-cm.

2.6.1 Electrical performance in water. Electrical properties were also tested upon immersion in water at 90°C up to 28 days. At first, we estimated the absorption of water of the MV TPV compounds in comparison to MV IS79, according to the Italian norm CEI 20-86. The results summarized in Table 5 indicate that the compounds have virtually identical water absorption after 14 days in water at 85°C, well below the upper limit (5 mgr/cm^2).

Table 5. Water absorption according to CEI 20-86

	MV IS79	MV TP79 A	MV TP79 B	MV TP79 C
Water absorption[1] [mgr/cm^2]	0.34	0.32	0.35	0.34

[1]Gravimetric method, CEI EN 60811-402

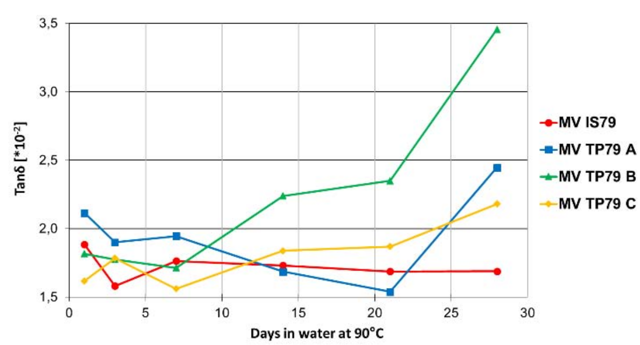

Figure 11. Loss factor (Tanδ) in function of days immersed in water at 90°C measured at 500 V and 50 Hz.

The low water absorption reflects on the variation of Tanδ after immersing the samples in water at 90°C (see Figure 11). The compounds have a good retention of the loss factor, which is, after 28 days in water, in the worst case about 0.035 and in the best 0.017. Again, MV TP79 C, thanks to its superior stability, has the best performance, close to the benchmark performance of MV IS79.

Having low water absorption, also ε_r remains almost unvaried after the immersion in water at 90°C. As illustrated in Figure 13, the increasing of the dielectric constant is rather small after immersion in water.

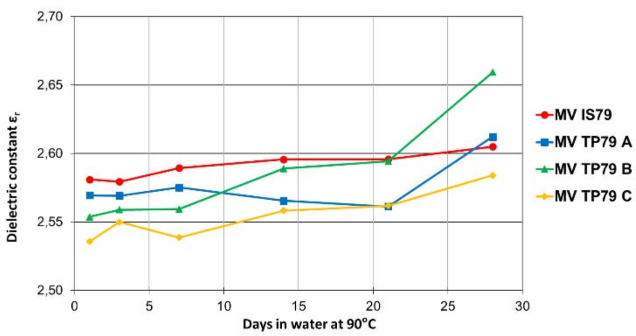

Figure 13. Dielectric constant (ε_r) in function of days immersed in water at 90°C measured at 500 V and 50 Hz

Among the MV TPV compounds, MV TP79 C displays the best stability over time having a lower ε_r compared to the benchmark MV IS79 even after 28 days in water.

3. Conclusions

Newly developed MV TPV compounds have been presented in this paper. The promise is to produce MV insulation compounds with properties equal to the actual lead free MV insulation market standard and the easy processing of thermoplastics. The preparation of such compounds was described along with their full characterization in comparison to the standard lead free MV insulant. By means of DSC we investigated the dynamic vulcanization process. Indeed, we demonstrated our capability to produce in an industrial pilot plant TPV compounds for application as MV insulation. Despite their complex formulation containing polymers, fillers, co-agents and antioxidants, the MV TPV were obtained in a fully reproducible and reliable process. Result of our technology, are the overall properties of the MV TPV compounds, which resemble the performance of the standard lead-free MV IS79. Rheological studies, besides confirming the TPV nature of the compounds, simulate their extrusion behavior, demonstrating that, thanks to an accurate choice of the thermoplastic PP it is possible to lower the shear stress maintaining unaltered the typical elastic response of TPV compounds. A detailed analysis of the stress-strain plots of the MV TPV compounds confirms their elastic behavior affected only partially by the crystallinity of the thermoplastic phase, resulting in mechanical properties similar to the benchmark MV IS79. Upon ageing at 135°C, MV TPV compounds proved their resistance up to 504 h with TS and EB retained > 70%. After ageing for 504 h at 150°C, MV TP79 C preserved the 80% of its TS and the 70% of its EB, almost matching the reference MV IS79. Lastly, dry and wet electrical properties were measured for all the compounds at 500 V and 50 Hz. Dry Tanδ raises with the temperature until an upper limit of about $5 \cdot 10^{-3}$ at 90°C for MV TP79 A, which is still comparable to Tanδ of MV IS79 at the same temperature, $3.5 \cdot 10^{-3}$. Similarly, ε_r is varying in a very narrow range (between 2.8 and 2.4) at 25°C and up to 90°C for all the compounds. Volume resistivity measurements confirm excellent insulating properties at 25°C (10^{15} Ω-cm) slightly decreasing at 90°C (10^{13} Ω-cm). Wet electrical properties were measured immersing the samples in water at 90°C up to 28 days. Wet Tanδ increases to a maximum of $3.5 \cdot 10^{-2}$ for MV TP79 B. MV TP79A and C exhibited better resistance to water; the latter close to the performance of MV IS79 after 28 days in water at 90°C, $2.2 \cdot 10^{-2}$ and $1.3 \cdot 10^{-2}$ respectively. The same trend was observed for ε_r, which slowly increases after immersing the samples in water. However, the fluctuations are virtually irrelevant, being between 2.53 and 2.66 and considering the error associate to the measure.

In conclusion, we presented a full study on TPV compounds as insulation materials for MV applications. We show our stepwise approach by which we could incrementally improve the properties of the compounds, obtaining a fully thermoplastic lead free material, namely MV TP79 C, with mechanical, rheological and electrical performance comparable to those of the lead free market standard MV IS79. According to the norm CEI 20-86, MV TP79 C has the potential to be implemented as MV insulation with 105°C rating for continuous operating temperature and emergency shortcut of 250°C. Pushing forward our strategy, we expect to develop MV TPV compounds with higher resistance and better electrical properties at high temperature and in water in the near future.

4. Acknowledgments

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5. References

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