



A new generation of cable grade poly(vinyl chloride) containing heavy metal free modifier

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Abstract

Many additives are used to improve the performance of cables in terms of increasing their flame retardancy, thermal stability, thermal conductivity, and other characteristics. Unfortunately, most of these additives contain heavy metals. Therefore, the main objective of this study is to introduce a material representing a new generation of environmentally friendly heavy metal-free stabilizers for cable grade poly(vinyl chloride) that can compete with traditional materials in terms of performance and distinctive properties. This unique additive is Oxydtron, a synthetic silicate or simply nanocement. The tests performed are rheological properties represented by a capillary rheometry analysis, limiting oxygen index, and volume resistivity. The most significant improvement in Bagley correction measurements was 14.61%; 18.13%; and 27.20% more than poly(vinyl chloride) basic formulation when using 5wt.% Oxydtron at 160 °C, 170 °C, and 180 °C, respectively. Also, the mean increases in relaxation time were 3.200 times, 8.825 times, and 12.458 times more than poly(vinyl chloride) basic formulation with 1wt.%, 3wt.%, and 5wt.% of Oxydtron, respectively. Furthermore, the Oxydtron lowered the value of the accompanying thermal gradient of the L.O.I test, reducing the heat-affected zone. The best result was with the extrusion processing method due to the uniformity of the processing conditions. However, the thermal gradient analysis showed residual heat stress in the test samples after cutting the burning layer and re-testing the samples again; this causes them to burn faster. This situation requires caution for designs that are exposed to high temperatures without burning. The optimum improvement in volume resistivity value was 14.71% and 38.24% more than poly(vinyl chloride) basic formulation after adding 5wt.% and 7wt.% of Oxydtron, respectively.

Keywords Cable grade · Oxydtron · Plasticized poly(vinyl chloride)

Introduction

It is well recognized that heavy metals are found in many additives used to improve the poly(vinyl chloride) performance, such as stabilizers and flame retardants. Despite their environmental and health threats, they are now widely used [1–5]. The accumulation of these heavy metals from stabilizers and flame retardants in soil, water (whether in rivers, lakes, groundwater), or even in the air leads to environmental pollution, leading to flame retardant transfer to organisms in that polluted environment, including humans. For example, in European countries, concentrations of flame retardants were found in human milk and the bodies

of birth cohorts (but their concentrations were lower than in the United States) and household dust. Concentrations of flame retardants were also found in the bodies of birds and their eggs [6–14]. Therefore, accountable to these environmental challenges and responsibilities, most of these flame retardants must be reconsidered in terms of use and finding an environmentally friendly and sustainable alternative. Especially since countries have already started issuing laws prohibiting the use of certain types of flame retardants due to their severe damage to the environment after research has proven this [15–17]. Many flame retardants currently in use are included in European Union regulation (EC) No. 1272/2008 and its amendments for materials classification, classified as dangerous materials. So it has become imperative to search for safe alternatives to these materials [18]. Therefore, there must be a precise harmony between environmentally friendly flame retardants and preserving the materials' properties. This matter requires more time and

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research for a complete shift from traditional flame retardants to those environmentally friendly [1, 18–21]. Certain recent flame retardants are now available to comply with the successful flammability tests regulations. Also, the interference of retardants with flame reaction chains will restrict the oxidation of the hydrocarbon. This interference will prevent the process of converting carbon monoxide to carbon dioxide, which causes highly volatile, very smoky fire effluents and rich in incomplete combustion products [22]. On the other hand, a significant effort has been made in recent years to develop new stabilization systems for poly(vinyl chloride) processing to get as far away as possible from heavy metal-based stabilizers. Due to the toxicity of the lead and cadmium stabilizers, they are now in limited use, but despite there are various alternatives now to occupy a place of these stabilizers, such as calcium/zinc, organotin, and barium/zinc systems, but still has its disadvantages for the plasticized vinyl formulator.

Therefore, a complete switch to environmentally friendly stabilizers needs more research in this field. With the tendency to replace traditional stabilizers and flame retardants with environmentally friendly ones, the environmental conditions surrounding the product containing this type of stabilizers and retardants must be considered and the applications for which it is used. One study revealed that environmentally friendly retardants could be harmful when breaking down by heat and ultraviolet rays [22–24]. Therefore, it is necessary to use these retardants carefully. Obtaining the ideal flame retardant requires great research efforts that may not take as long as previous research due to scientific research development. Simultaneously, the chemical stability of environmentally friendly stabilizers and flame retardants in different conditions is critical to increasing safety levels when using these retardants.

Experimental procedure

Materials

There are two types of materials used in this article:

1. The essential components of PVC: These are the mixture materials necessary to form the PVC basic formulation after processing, which consists of:
 - i. The suspension powder: which is the main component of the mixture. Ongrovil® S-5070 suspension type PVC produced and supplied by BorsodChemZrt., Hungary, was used in my work with a quantity of 100 phr.

- ii. Plasticizer: This material aims to give sufficient flexibility to the product, and here a DOP (or DEHP), Bis(2-Ethylhexyl) ortho-phthalate Plasticizer was used with a quantity of 70 phr. DEZA, a. s., CO., Valašské Meziříčí, Czech Republic supplies this Plasticizer.
- iii. Stabilizer: and its purpose is to improve PVC resistance for many functional conditions. Newstab-50® Calcium-Zinc-based stabilizer supplied by Betaquímica CO., Spain, was used for this purpose with a quantity of 1.5 phr.
- iv. Lubricant: this material helps facilitate PVC processing through reducing friction and shear-dependent viscosity control. Licowax® E Wax supplied by Clariant International Ltd, Muttenz Switzerland, was used in this work with a quantity of 0.3 phr.

2. Additive: To improve specific properties in any polymer, many additives are added. In this article, Oxydtron® Nanocement-type A, an environmentally friendly heavy metal-free, has been added with three different quantities 1, 3, and 5 wt.%. Bioekotech Hungary Kft supplied Oxydtron® Nanocement.

Mixing process

All the materials (except Oxydtron) had been mixed by a high-speed fluid mixer type Mischtechnik MTI 10. After the mixing process begins, the temperature increases steadily from room temperature to 150 °C due to the shearing of particles between the components, rising from 600 to 2700 rpm. The mixture stays at 2700 rpm for about 15 min while the temperature increases to 150 °C. It allows the plasticizer to completely penetrate poly(vinyl chloride) particles to achieve a homogenized structure and optimum manufacturing characteristics. The mixing speed is then decreased to 600 rpm, cooling water is circulated in the jacket of the mixer, and the temperature drops below 45 °C. The mixture stays at this temperature and speed until the mixing phase is completed. It takes about 40 min to complete the mixing process. After the main mixing phase, Oxydtron is applied to the basic formulation mixture of poly(vinyl chloride) by weight fraction (1, 3, and 5 wt.%) and mixed in a small electrical mixer to uniformly dispersed and homogenized into sample powder mixtures.

Preparation of samples

In all tests, Four blends of plasticized poly(vinyl chloride) had been made, (1) poly(vinyl chloride) basic formulation; (2) poly(vinyl chloride) containing 1wt.% Oxydtron; (3)

poly(vinyl chloride) containing 3wt.% Oxydtron; and (4) poly(vinyl chloride) containing 5wt.% Oxydtron. All samples have been prepared according to ISO standards, and all preparation steps have been completed at BorsodChem Zrt., Hungary as follows:

1. Capillary Rheometry Analysis Samples: the samples were produced by using extrusion machine type SCHLOEMANN BT-50 as a disk shape with 3 mm diameter and 2 mm thickness.
2. Limiting Oxygen Index Samples (L.O.I): L.O.I samples were fabricated as a rod by using an extrusion machine type GÖTTFERT with 170 °C temperature and 60 rpm speed.
3. Volume Resistivity Samples: these samples were prepared by using laboratory roll mill type Schwabenthan polymix 150 U, at 170 °C temperature, time 5 min, rolling speeds 21 rpm (front roller) and 24 rpm (back roller). The samples have a circular shape with 70 mm diameter and 1 mm thickness.

The tests

The capillary rheometry analysis

This test was done at BorsodChem Zrt. And according to ISO 11443:2014 standard [25], at different temperatures rang (160 °C, 170 °C, and 180 °C) and different dies lengths (15, 30, and 45 mm) with (3 mm) die hole diameter for all lengths. Laboratory extruder type Göttfert Extrusimeter G20 was used to complete this test with a processing speed of 60 rpm. Capillaries rheometer tests are often achieved by applying pressure above the capillary inlet. Therefore, an additional pressure drop at the die entrance hides the true pressure drop along the capillary, where the flowing material goes from a wide reservoir (the main cylinder or barrel) to a narrow capillary, possibly also generating turbulence. Assuming that the same extra pressure drop occurs with different capillary lengths (but maintaining a constant barrel and capillary diameter and inlet shape), the pressure reading can be corrected, and the true pressure drop can be estimated much more accurately. The state called Bagley correction.

Limiting oxygen index test

L.O.I test was done according to ISO 4589–2 standard [26] by using a Stanton Redcroft FTA flammability unit at BorsodChem Zrt., Hungary; Laboratory of Vinyl Technology.

Relaxation time measurement

DMA test results were used to calculate the relaxation time. The DMA test was done according to ISO 6721–11:2012

standard [27] by using dynamic mechanical, thermal analyzer MK III manufactured by Rheometric Scientific, Inc. and found at BorsodChem Zrt., Hungary, used for testing the samples.

Volume resistivity measurement

IM6 Megohmmeter Radiometer Copenhagen manufactured by RE Instruments was used to record the volume resistivity of samples according to ASTM D257-14 standard [28]. This device is found at BorsodChem Zrt., Hungary.

FLIR thermal gradient image analysis

Thermal analysis for L.O.I samples by using a thermal imaging camera (FLIR Systems) has been done to determine the thermal gradient of HAZ. The image and object parameters of this analysis are shown in Table 1.

SEM–EDX analysis

SEM–EDX analysis was used to analyze the chemical composition of Oxydtron and the dispersion of this additive in the poly(vinyl chloride). This test was carried out using a Carl Zeiss EVO MA10 SEM at the University of Miskolc. The results of SEM–EDX analysis are shown in Fig. 1, which represents the chemical composition analysis for Oxydtron, Fig. 2 represents the SEM images for dispersion Oxydtron in the poly(vinyl chloride). Finally, Fig. 3 represents the EDX mapping analysis for poly(vinyl chloride) containing Oxydtron.

Results and discussion

Capillary rheometry analysis

It is known that the inlet pressure drop (Δp) can be measured practically by using capillaries that are of different lengths while maintaining a constant diameter. And in the case of round hole capillaries, the Bagley correct is required to measure this inlet pressure drop [29–31]. Figure 4 represents the length correction of plasticized poly(vinyl chloride) basic formulation at speed 60 rpm, accomplished with

Table 1 Image and object parameters of FLIR analysis

Parameter	Description/ Value
Camera Model	FLIR SC660
Emissivity	0.5
Reflected apparent temperature	20 °C
Object distance	1.0 m

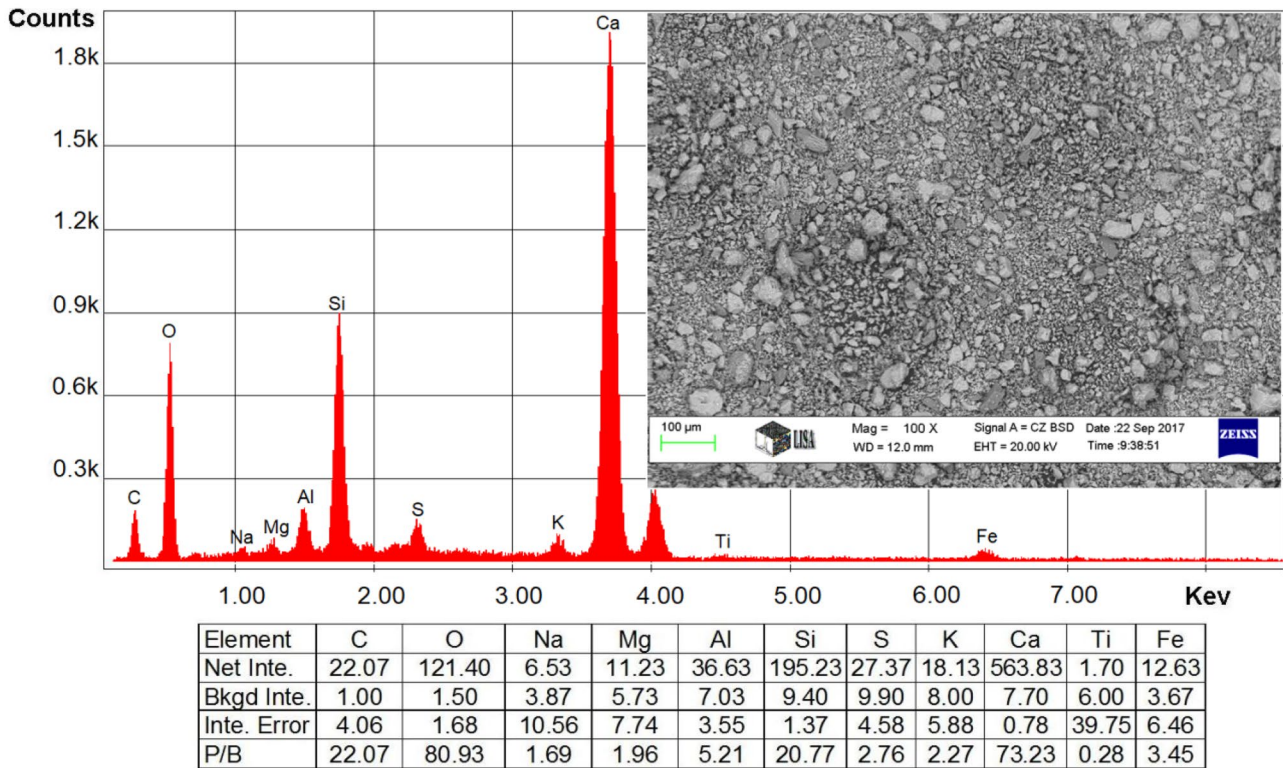


Fig. 1 SEM- EDX analysis for Oxydtron

three different capillaries length (15, 30, and 45 mm) have the same diameter (3 mm) and (L/D) were (5, 10, 15). And Table 2 represents Bagley correction and slope values of plasticized poly(vinyl chloride) basic formulation at the same three temperatures. As the plasticized poly(vinyl chloride) melt travels and is extruded from the greater diameter of the barrel to the smaller diameter of the capillary, the excess pressure begins to drop due to extensional viscosity [32, 33]. The pressure drop is proportional to the capillary length, which is more significant with the longer capillary. However, it is inversely proportional to the temperature as the higher the temperature, the lower the pressure drop. This condition is also similar to the Bagley correction, as shown in Table 2. The length correction will decrease with increasing the temperature because the poly(vinyl chloride) melt becomes less elastic at higher temperatures. Also, We can observe from this figure that the extrapolation of the linear Bagley correction plots of poly(vinyl chloride) basic formulation indicated an excellent fitting.

Figure 5 represents the length correction of plasticized poly(vinyl chloride) containing 1wt.% Oxydtron at 160 °C, 170 °C, and 180 °C, respectively, at speed 60 rpm. Table 3 represents the Bagley correction and slope values for poly(vinyl chloride) containing 1wt.% Oxydtron at the same temperature and speed. This figure and the table show that the Bagley correction value has increased after adding

Oxydtron. It is a fact that the shear stress is directly proportional to the pressure drop [34–36]; therefore, the shear stress values in the case of plasticized poly(vinyl chloride) containing 1wt.% Oxydtron will be higher than poly(vinyl chloride) basic formulation. Still, the higher correction values show higher melt elasticity, and it can be evident that the length correction could be the measure of melt elasticity. For that, I didn't find any references. Also, through Fig. 3 we observe that the fitting will be much better, and the slope values are higher than in poly(vinyl chloride) basic formulation, where the particles will increase the shear stress because it is an effective filler in terms of mechanical properties. We also found that the plasticized poly(vinyl chloride) containing Oxydtron is less sticky to the exit mold hole, which will happen to the extrusion barrel, reducing the viscosity of the melt.

With an increase in the weight fraction of Oxydtron to 3wt.%, the improvement of length correction increases, which is what we can see in Fig. 6, which represent the Bagley correction for of plasticized poly(vinyl chloride) containing 3wt.% Oxydtron at 160 °C, 160 °C, 170 °C, and 180 °C respectively. The Bagley correction and slope values for poly(vinyl chloride) containing 3wt.% Oxydtron at the same temperature and speed are presented in Table 4.

There has also been an improvement in the drop in pressure, which means that the melt's elasticity has

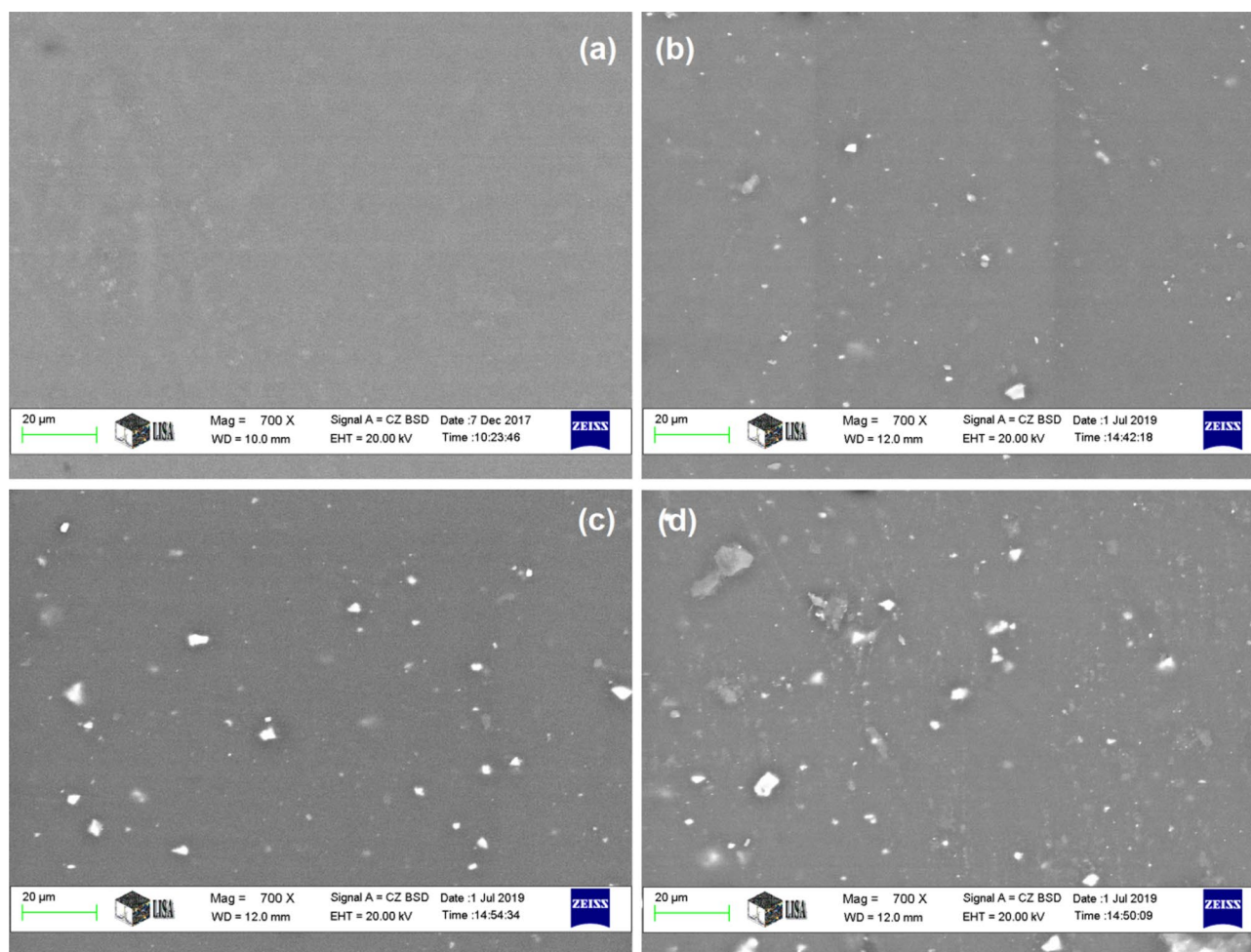


Fig. 2 SEM analysis images for dispersion of Oxydron in the poly(vinyl chloride), where (a) Poly(vinyl chloride) basic formulation, (b), (c), and (d) Poly(vinyl chloride) containing 1wt.%, 3wt.%, and 5wt.%, Oxydron respectively

improved further, as explained earlier. Although the length correction continues to decrease with increasing temperatures (which is normal), it maintains higher accuracy levels than the poly(vinyl chloride) basic formulation or containing 1wt.% Oxydron. This is possibly the key reason why Oxydron is so successful because of this additive's role in counteracting the segmental movement, and the polymer structure will consist of liquid-like molecules that increase the elasticity.

The perfect results were achieved in the length correction enhancement by applying 5% Oxydron, which can be seen in Fig. 7, which display the Bagley correction for plasticized poly(vinyl chloride) containing 5wt.% Oxydron at 160 °C, 160 °C, 170 °C, and 180 °C respectively, and in Table 5. The Oxydron continues to sustain a high degree of length correction as the additional percentage increases to 5 wt.%.

Also, we can see from Fig. 8, which represents Bagley correction as a function of Oxydron weight fraction, that the Bagley correction increases with increasing Oxydron

addition. Even 1wt.% of Oxydron significantly improves the Bagley correction and adding further Oxydron continues to rise. That means the additive increased the melt elasticity. The poly(vinyl chloride) melt elasticity increases by increasing temperature, and the viscous flow becomes dominant. Because the Bagley correction represents the energy necessary to alter the flow geometry, the Bagley correction of the plasticized poly(vinyl chloride) decreases by increasing temperature. But this drop in Bagley correction will be less in the presence of Oxydron, as shown in Fig. 9, which represents Bagley correction as a function of the processing temperature.

Relaxation time

As we know, the relaxation time in polymers represents the time required for polymer chains to return to equilibrium after deformation, where substantial mobility in the polymer's structure has happened [37–39].

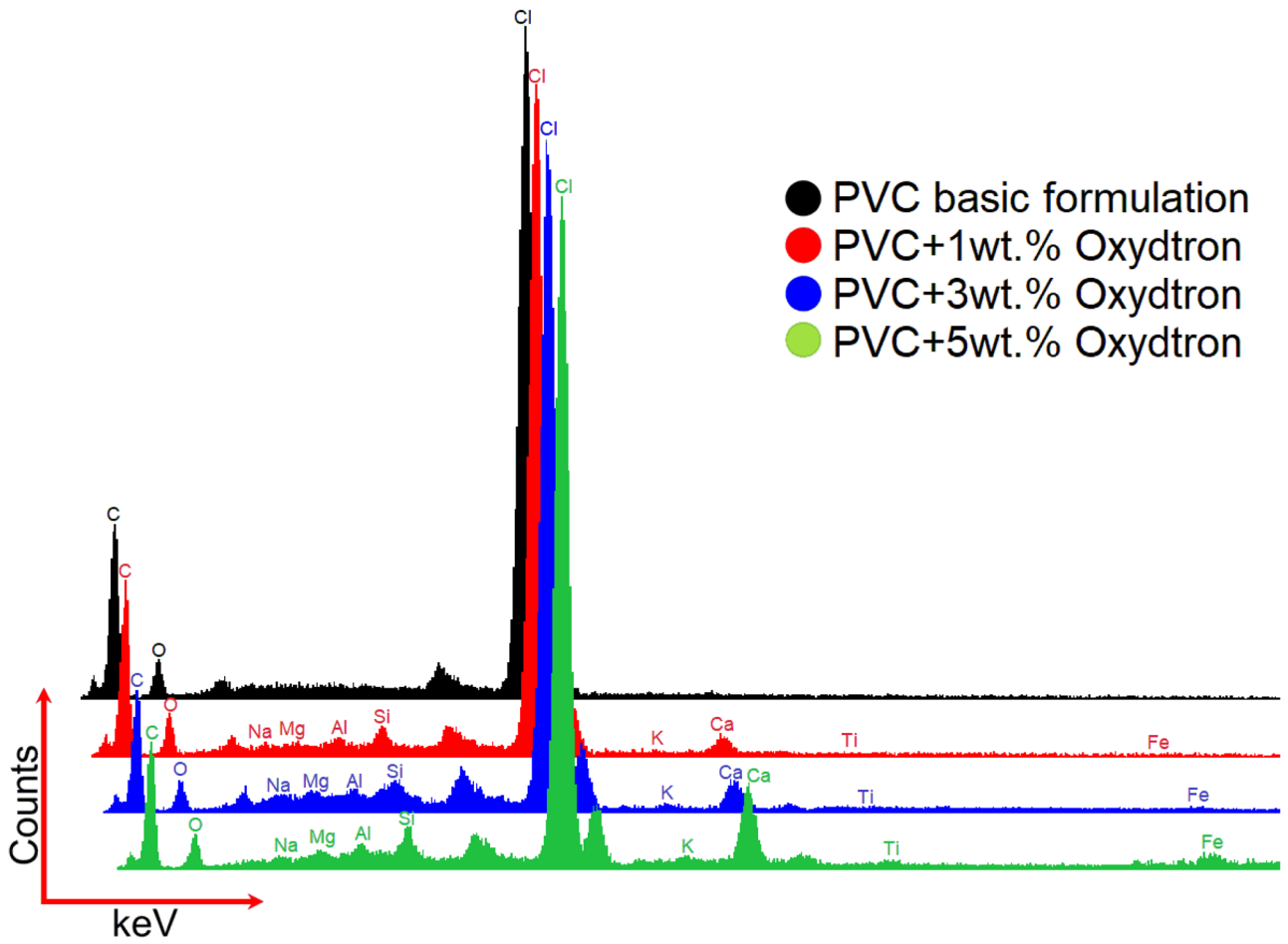


Fig. 3 EDX mapping analysis for poly(vinyl chloride) containing Oxydtron

Fig. 4 Length or Bagley correction of for plasticized poly(vinyl chloride) basic formulation at 160 °C, 170 °C, and 180 °C respectively

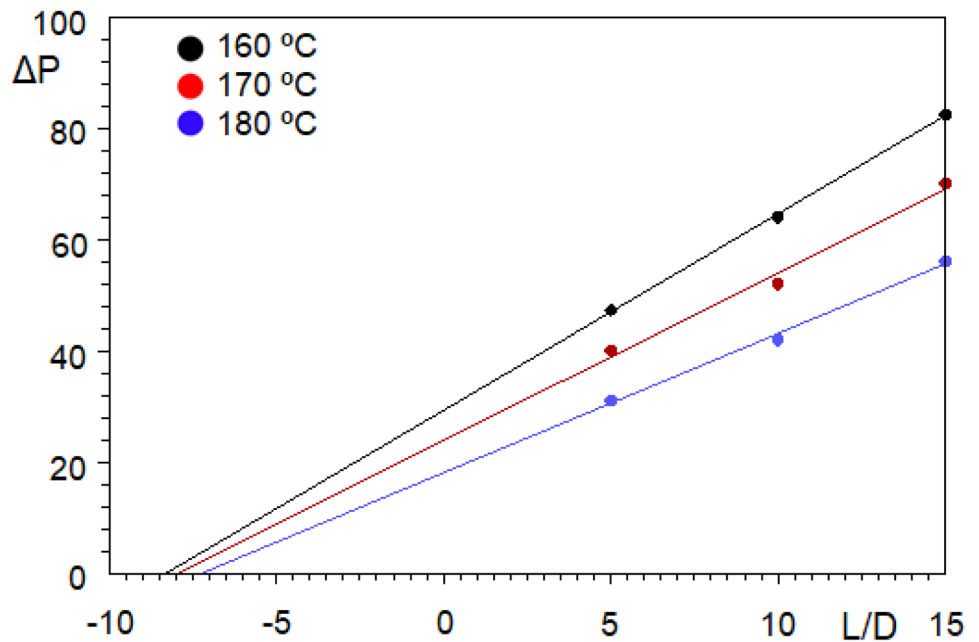
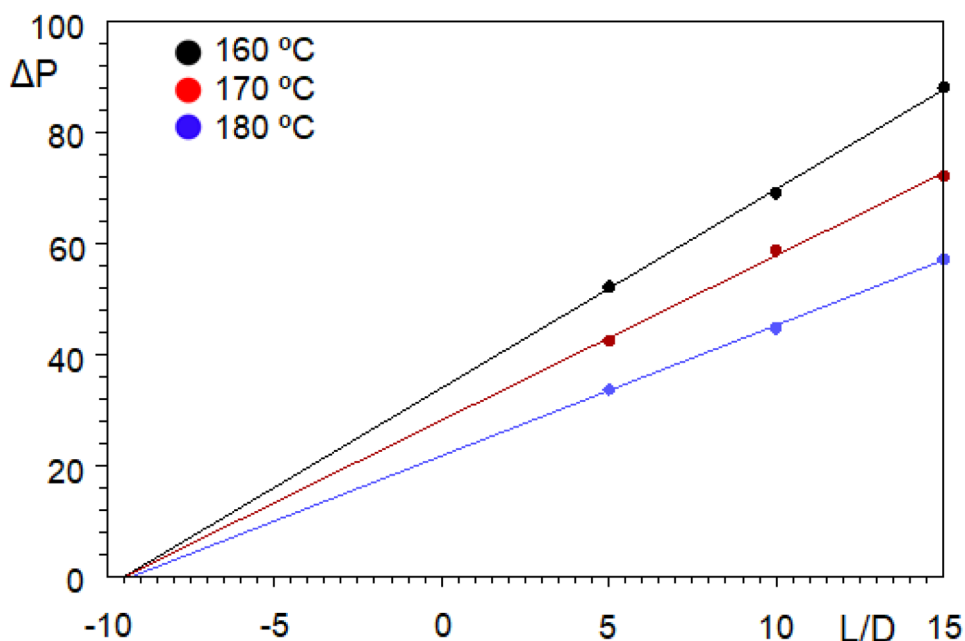


Table 2 Bagley correction and slope values of plasticized poly(vinyl chloride) basic formulation at 160 °C, 170 °C, and 180 °C

Temperature, °C	160	170	180
Bagley Correction	8.35	8.00	7.28
Slope	3.52	3.00	2.49

Figure 10 represents the relaxation time (τ) for plasticized poly(vinyl chloride) containing Oxydtron, calculated based on the temperature test data with temperatures range -20 °C, -10 °C, and 0 °C. Evidently, τ (T) (relaxation time at T temperature) is independent of frequency; the temperature of maxima is shifted to higher temperatures by increasing frequency because shorter relaxation time appears at the higher temperature. Also, we can see that the relative increase of relaxation time is almost linear by Oxydtron content, as shown in Fig. 11, and at 5wt.% Oxydtron reached 12 times (12.458) more than poly(vinyl chloride) basic formulation. The relative increase is the same at different temperatures.

From Table 6 which represents the glass transition temperatures (T_g) values for plasticized poly(vinyl chloride) containing Oxydtron, we can see that there is an interaction between poly(vinyl chloride) and Oxydtron particles, which causes the reduction of the kinetic energy of plasticized poly(vinyl chloride), and reducing the segmental movement as mentioned, which will prevent the molecules from assembling in crystalline systems, so the relaxation time of this movement increases and the polymer will consist of long physically crosslinked liquid-like molecules [37].

Fig. 5 Length or Bagley correction for of plasticized poly(vinyl chloride) containing 1wt.% Oxydtron at 160 °C, 170 °C, and 180 °C respectively**Table 3** Bagley correction and slope values of plasticized poly(vinyl chloride) containing 1wt.% Oxydtron at 160 °C, 170 °C, and 180 °C

Temperature, °C	160	170	180
Bagley Correction	9.10	9.07	8.90
Slope	3.64	3.04	2.38

Volume resistivity (ρ)

The cable cover material must have high electrical insulation properties conjugated with high flammability resistance, providing maximum protection and safety levels [40–43]. Figure 12 represents the electrical behavior (volume resistivity- ρ) of plasticized poly(vinyl chloride) containing Oxydtron. From this figure, we can notes that the Oxydtron increased the volume resistivity, i.e., decreased the conductivity, where the increase in resistivity is directly proportional to the percentage of Oxydtron. Because silicate compounds might contain imperfect structures, both anions and cations are fixed to the crystal lattice. Therefore, these immobile ions cannot neutralize each other better, trapping mobile ions from the matrix polymer. The effect is utilized using calcined clay filler, improving the electrical properties of PVC compounds. Since the Oxydtron is a clay-based product, it can be assumed that it works similarly.

SEM and thermal gradient analysis for heat affected zone (HAZ)

Despite the fact that poly(vinyl chloride) has a superior flame resistance compared to other polymers, it will suffer

Fig. 6 Length or Bagley correction for of plasticized poly(vinyl chloride) containing 3wt.% Oxydtron at 160 °C, 170 °C, and 180 °C respectively

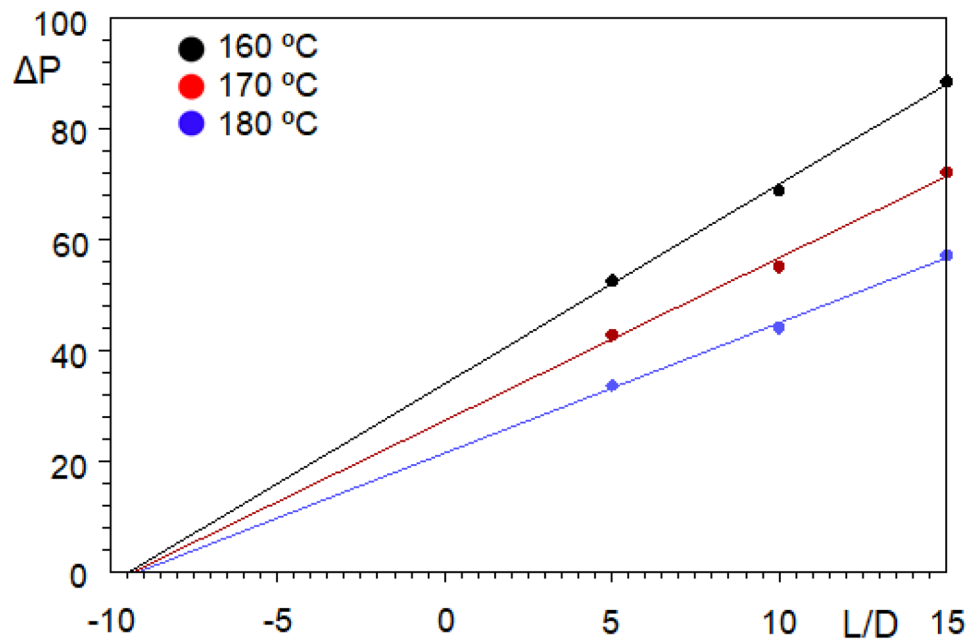


Table 4 Bagley correction and slope values of plasticized poly(vinyl chloride) containing 3wt.% Oxydtron at 160 °C, 170 °C, and 180 °C

Temperature, °C	160	170	180
Bagley Correction	9.44	9.31	9.17
Slope	3.60	2.93	2.34

from structural changes due to exposure to high temperatures, which causes deterioration of its distinctive properties [44–49]. The poly(vinyl chloride) structure will

begin to release HCl from its internal structure at elevated temperatures, i.e., it begins to degrade in these temperatures. Thus, the poly(vinyl chloride) structure will lose HCl, not just from burnt layers directly exposed to fire. Still, this loss of chlorine-containing compounds extends to the under layers far from the combustion zone, which I called heat-affected zone (HAZ), as shown in Fig. 13. This behavior is due to the dehydrochlorination of poly(vinyl chloride). Through Fig. 13a we notice that the heat-affected zone is wide in poly(vinyl chloride) basic formulation, which is evidence of its low thermal and flame

Fig. 7 Length or Bagley correction for of plasticized poly(vinyl chloride) containing 5wt.% Oxydtron at 160 °C, 170 °C, and 180 °C respectively

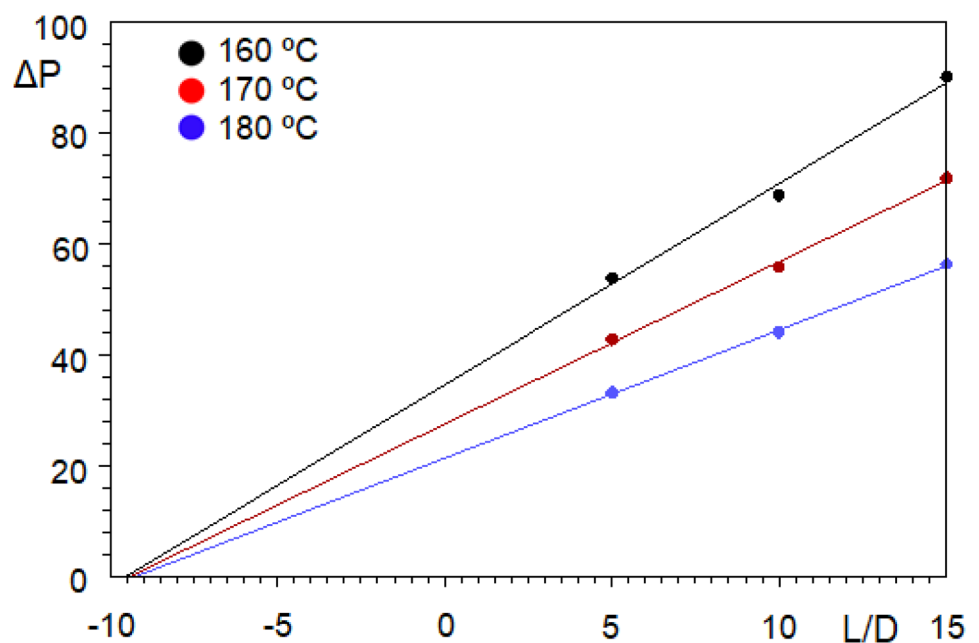


Table 5 Bagley correction and slope values of plasticized poly(vinyl chloride) containing 5wt.% Oxydtron at 160 °C, 170 °C, and 180 °C

Temperature, °C	160	170	180
Bagley Correction	9.57	9.45	9.26
Slope	3.62	2.92	2.31

resistance and its rapid degradation at high temperatures of heating or combustion. The actual change in this critical zone begins after the addition of 1wt.% Oxydtron, as shown in Fig. 13b. The FLIR imaging test showed the beginning of the receding of the thermal effect in this zone

than before adding the Oxydtron, leading to an increase in the thermal stability of the poly(vinyl chloride). The HAZ recedes further with the rise in the weight fraction of Oxydtron to 3wt.% and 5wt.% as shown in Fig. 13c, d, resulting in increased resistance and stability of the poly(vinyl chloride) at such high temperatures, which reduces the degradation risk.

Also, I found that when re-testing the same sample again after cutting the burned area, the under layers have been ignited faster, and the HAZ area is wider, as shown in Fig. 14. This behavior was dissimilar to the original sample tested for the first time, which means the heat-affected layer has low flame resistance due to less chlorine [50].

Fig. 8 Bagley correction as a function of Oxydtron wt.% at 160 °C, 170 °C, and 180 °C respectively

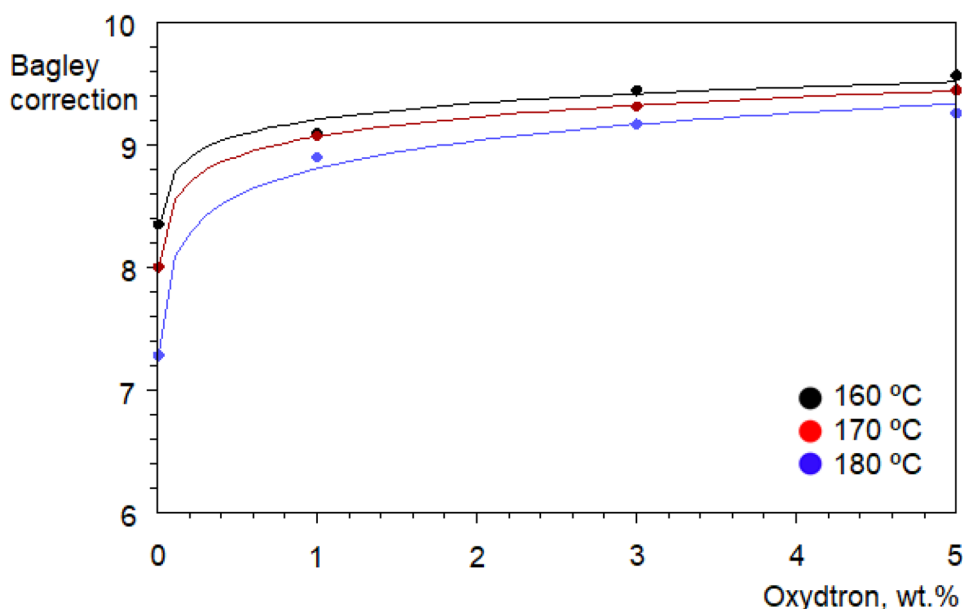


Fig. 9 Bagley correction as a function of processing temperature

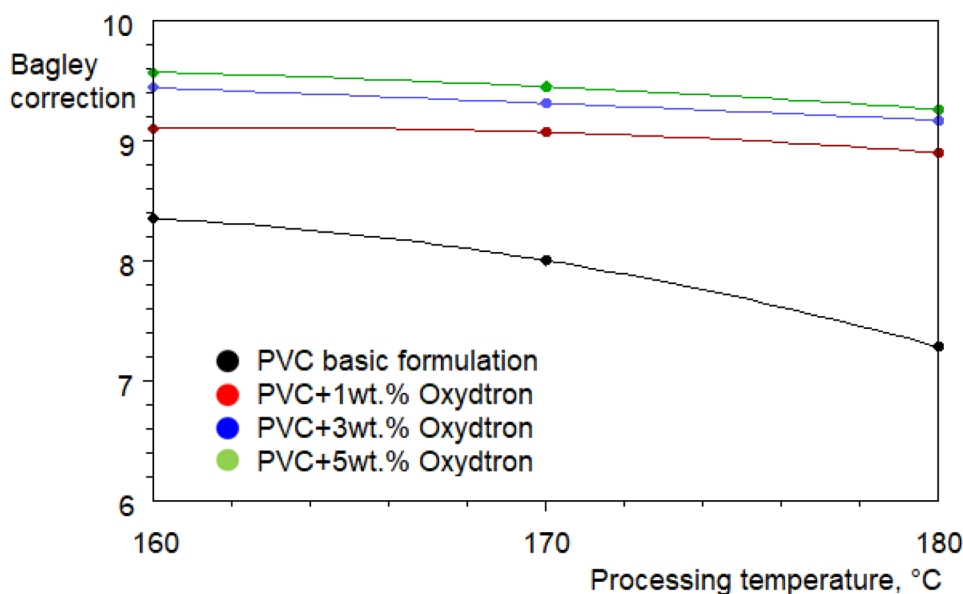


Fig. 10 The relaxation time for plasticized poly(vinyl chloride) containing Oxydtron as a function of temperature

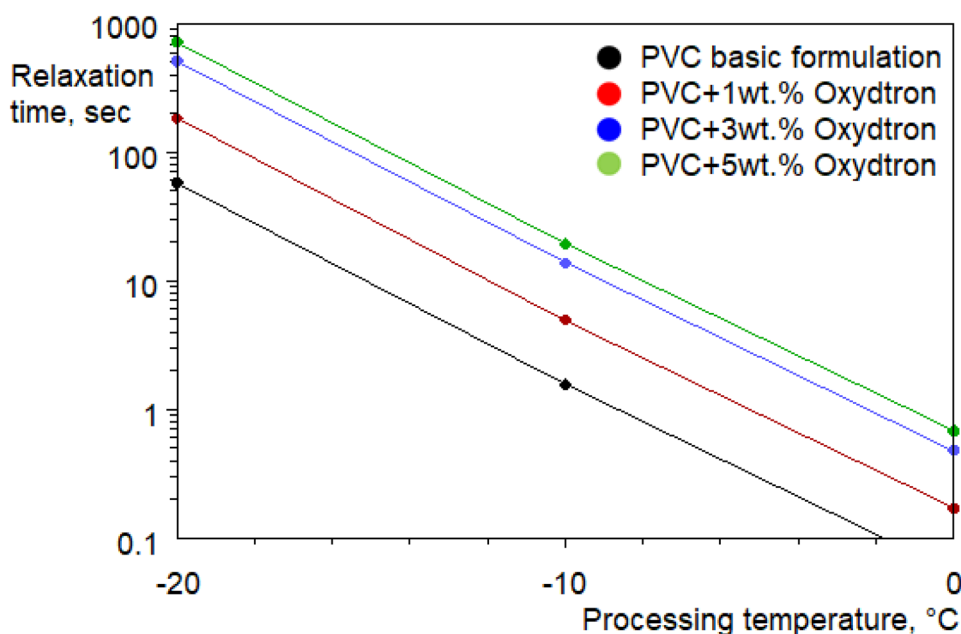
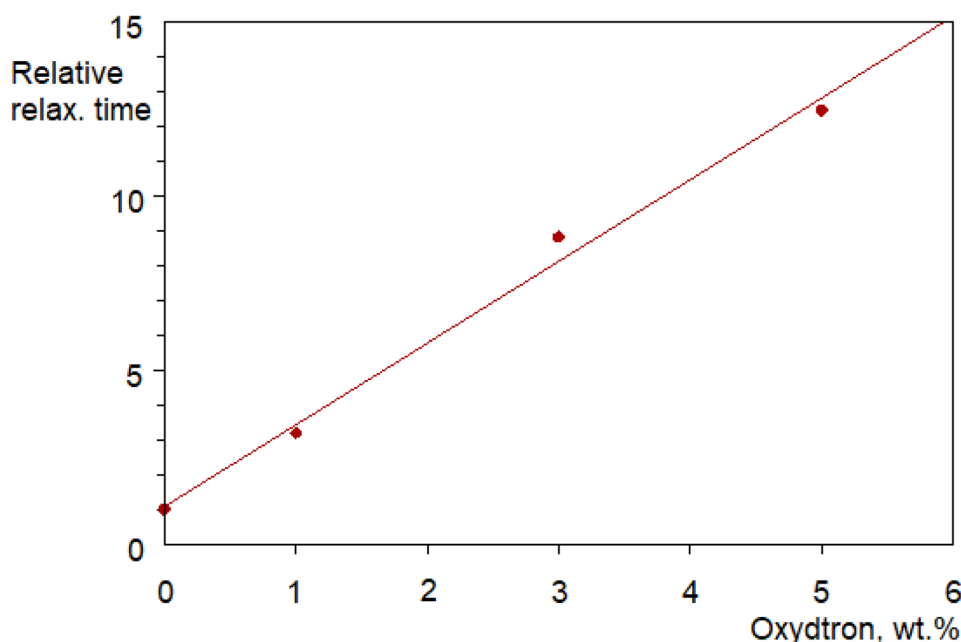


Fig. 11 The relative relaxation time as a function of Oxydtron content



The size of the heat-affected zone depends on the type of processing method, as it is wider in rolling samples than in extrusion samples, as shown in Fig. 15. Since the rolling sample is exposed to processing temperatures more

Table 6 Glass transition temperatures (T_g) measured by DMA for plasticized poly(vinyl chloride) containing Oxydtron

Material	PVC	PVC+1% Oxydtron	PVC+3% Oxydtron	PVC+5% Oxydtron
T_g , °C	-3.60	0.30	3.50	4.66

prolonged than the one-stage extrusion, the rolling sample is more heat stressed. Also, the rolling temperature is irregular along the length of the roll, which causes the concentration of the elements to varying from one point to another depending on the amount of heat that was exposed during the rolling. This thing will accelerate its yielding and softening when exposed to fires, unlike the extrusion sample, which resists fires better. Therefore, it is necessary to pay attention to the correct selection of the processing method in applications requiring high flame resistance.

Fig. 12 Volume resistivity of plasticized poly(vinyl chloride) containing Oxydtron

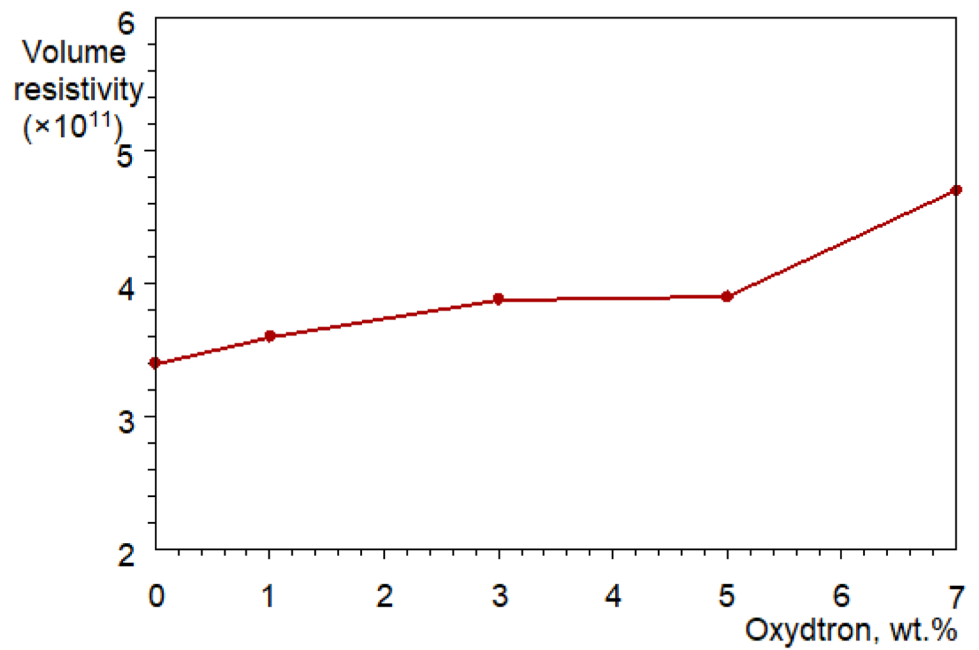


Fig. 13 FLIR analysis for HAZ after L.O.I test in (a) PVC basic formulation, (b) PVC + 1wt.% Oxydtron, (c) PVC + 3wt.% Oxydtron, and (d) PVC + 5wt.% Oxydtron

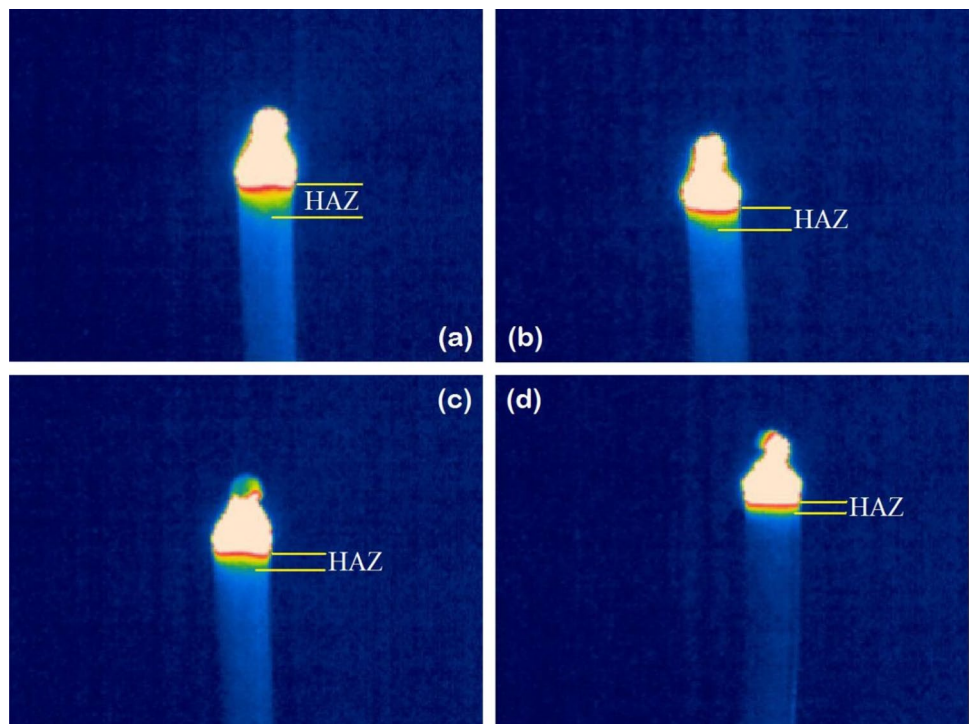


Fig. 14 FLIR for extruded plasticized poly(vinyl chloride) basic formulation HAZ when (a) testing for the first time, (b) re-testing after cutting the burnt layer

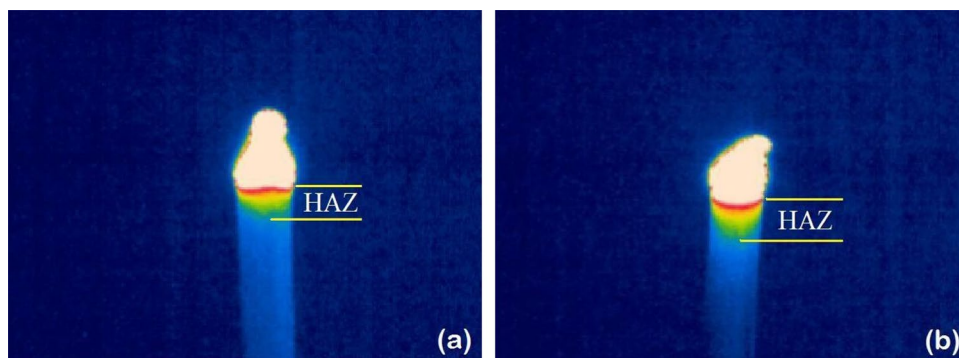
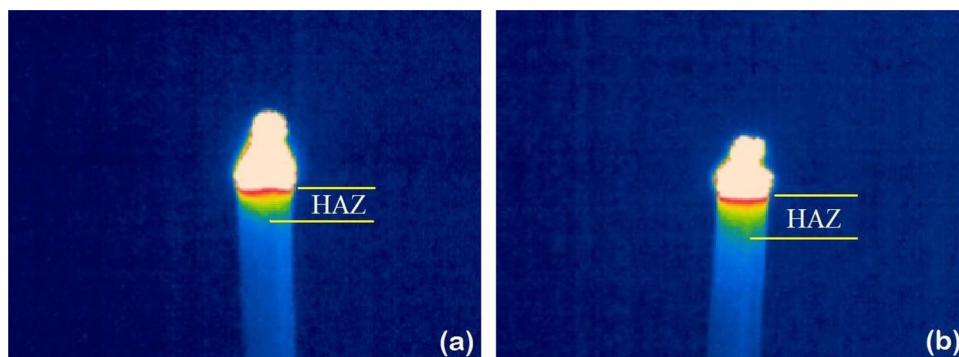


Fig. 15 FLIR for HAZ of (a) extruded plasticized poly(vinyl chloride) basic formulation, (b) rolled plasticized poly(vinyl chloride) basic formulation



Conclusions

The critical points obtained from the tests performed on plasticized poly(vinyl chloride) (basic formulation and containing Oxydtron) will be summarized. Using Oxydtron for the first time as a poly(vinyl chloride) stabilizer and flame retardant agent with highly efficient thermal stabilizing. Oxydtron was used first time as a flame retardant agent in plasticized poly(vinyl chloride) compounds. It was discovered that Oxydtron is also a highly efficient thermal stabilizer. This stabilizing-flame retarding synergistic effect created by Oxydtron additives was unexpected behavior of a material used as a cement additive just for construction purposes. The performance of plasticized poly(vinyl chloride) at high temperatures also improved significantly using Oxydtron additive. Oxydtron additives significantly improved the poly(vinyl chloride) melt elasticity even at low concentrations, e.g., 1%. The Oxydtron minimized the Bagley correction drop, which means the poly(vinyl chloride) flowability was improved. And Bagley correction values will decrease with increasing temperature. Since the Oxydtron works to shift the glass transition temperatures of poly(vinyl chloride) to higher levels, which affects kinetic energy and segmental movement, so the relaxation time will increase. The combustion of PVC is not limited to the exposed flame layer but continues beyond the combustion

layer. Experiments have shown a decrease in chlorine content in the heat-affected non-burning zone. In the absence of Oxydtron, heat affected zone will ignite easily due to structural changes. The analysis of this zone proved to be important in assessing thermal stability. The high volume resistivity will improve the insulation resistance and flame retardancy of the cable cover.

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Declarations

Conflicts of interest The author certify that he NO affiliations with or involvement in any organization or entity with any financial interest (such as honoraria; educational grants; participation in speakers' bureaus; membership, employment, consultancies, stock ownership, or other equity interest; and expert testimony or patent-licensing arrangements), or non-financial interest (such as personal or professional relationships, affiliations, knowledge or beliefs) in the subject matter or materials discussed in this manuscript.

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