Supplementary Information file of the paper

A New Perspective on Hydrogen Chloride Scavenging at High Temperatures for Reducing the Smoke Acidity of PVC Cables in Fires, IV: The Impact of Acid Scavengers at High Temperatures on Flame Retardance and Smoke Emission

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(1) Materials

Table S1 shows the commercial additives in Tables 1 and 2 of the paper.

Table S1

Inovyn 271 PC: PVC S K70 produced by Inovyn. https://www.ineos.com/businesses/inovyn/
Diplast N: Di Iso Nonyl Phthalate produced by Polynt S.p.A.. https://www.polynt.com/it/
Reaflex EP/6: Epoxidized Soy Bean Oil (ESBO) in the product portfolio of Reagens. https://www.reagens-group.com/
Arenox A10: Pentaerythritol tetrakis(3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate) in the product portfolio of Reagens. https://www.reagens-group.com/
RPK B-CV/3038: Thermal stabilizer for PVC. One pack in the product portfolio of Reagens. https://www.reagens-group.com/
RI004: Antimony trioxide from Quimialmel: https://quimialmel.it/
Atomfor S: Ground Calcium Carbonate produced by Omya: https://www.omya.com/it-it
Hydrocarb 95 T: Ground Calcium Carbonate produced by Omya: https://www.omya.com/it-it
Winnofil S: Precipitated Calcium Carbonate in the product portfolio of Imerys: https://www.imerys.com/
AS-6B: Acid scavenger at high temperatures acting in the condensed phase from Reagens S.p.A. https://www.reagens-group.com/
Aluprem T GR 4: Synthetic Aluminum tri hydroxide, produced by Tor Mineral. https://torminerals.com/
Ecopyren 3.5: Brucite in the product portfolio of Europiren. https://europiren.com/it/catalog/ecopiren/

(2) Sample preparation

PVC compounds in Tables 1 and 2 of the article are prepared by weighing the stabilizers' ingredients in the 0.001 g readability balance. PVC, plasticizers, fillers, flame retardants, and acid scavengers are weighed in the 0.1 g readability balance. All solid ingredients are introduced into a laboratory turbo mixer. Liquids such as ESBO and DINP are added when the temperature reaches 80°C, and stabilizer at 90°C. The mixing is stopped at 105°C, and the dry blend is dropped in PE bags and stored for 24 hours at 23°C to get the proper "maturation" of the dry blends.

130 g of the dry blend is processed in a laboratory calender for 3 minutes in some foils at a temperature of 160°C. The foils are pressed in the hydraulic press to wanted thicknesses plaques (1 mm and 3 mm) with the following cycles:

160°C per 1.5 minutes, pressure 0 bars

160°C per 1.5 minutes, pressure 60 bars

160°C per 1 min, pressure 100 bars

Cooling cycles, pressure 150 bars

(3) Measurements of the main properties

(3.1) pH and conductivity

The internal method 3 is performed according to EN 60754-2, [1] but with the heating regime of the EN 60754-1. [2] The measurements are obtained by burning test specimens in a tube furnace (SA Associates, standard model) and collecting the smoke in two bubblers containing double deionized water (DDW). 1 mm plaques are conditioned for 24 h at 23°C. The plaques are cut in slices with similar dimensions for all runs (approximately 1 mm x 1 mm). 1.0000 g +/- 0.0001 g of slices are weighed into the combustion boat of the dimensions according to EN 60754-1. The test apparatus has a touchscreen temperature controller through which the heating regime of EN 60754-1 is selected. The air flux is set at 300 ml/min according to the quartz tube dimensions, as EN 60754-1 requires. The determination of the heating regime is done according to EN 60754-1 (40 min +/- 5 min to 800 °C +/- 10 °C, 20 min +/-1 min in isothermal condition at 800 °C +/- 10 °C) and final temperature adjusted with a calibrated external thermocouple.

Before the first run, a preliminary test is done with an empty combustion boat to re-check the ramp and cleaning status of the quartz tube. That is to verify that residues inside the quartz tube do not affect the pH and conductivity of DDW in the bubblers.

The sample is introduced in the tube furnace at room temperature, and the heater is switched on. The smoke is collected in two bubblers containing DDW. The water from the bubbling devices and the washing procedures is collected in a 1 L volumetric flask filled to the mark. pH and conductivity are measured, and two replicates give mean value, standard deviation (SD), and coefficient of variation (CV).

pH and conductivity are measured simultaneously by inserting the electrodes in two different vessels. pH and conductivity measures are taken at 25 °C +/− 1 with the following procedure: the multimeter is calibrated with standard solutions before each measurement: pH at two points (4.01 and 7.00) and conductivity at 1 point at 141.3 μS/mm. The solutions closer to the measured value are chosen as correction standards, and the measurements are corrected accordingly through a correction factor. pH and conductivity electrodes have a reference thermocouple that adjusts the temperature fluctuation.

The data are reported in Tables 5 and 6 of the paper.

(3.2) Oxygen Index

LOI is measured with the FTT (Fire Technology Testing) test apparatus, according to ASTM D 2863, [3] as indicated in paragraph 12, procedure B. From 3 mm plaques, test specimens type IV are formed and burned in the test apparatus. Three replicates give mean value and SD.

The data are reported in Tables 7 and 8 of the paper.

(3.3) Cone calorimetry

The test apparatus is from FTT (Dual Cone Calorimeter). pHRR (peak of Heat Release Rate), FIGRA (Fire Growth Rate), THR (Total Heat Release), and TSP (Total Smoke Production) are measured according to ISO 5660-1. [4] Test specimens have the dimension of 100 mm x 100 mm with thickness of 1 or 3 mm. The exposed area is 88.4 cm^2. The measurements are performed with the sample holder in horizontal orientation, without containing wrapping in aluminum foil, using the sample holder frame (edge frame) and the retainer grid. The ISO 5660-1 standard is fully followed. The recording of the weight of the sample is performed before and after the measurement. The incident heat flux is 50 kW/m^2 and corresponds to 755 ± 5 °C. The flame ignition system (electric arc) is activated during the entire duration of the measurement (10 min).

The data are reported in Tables 9 and 10 of the paper.

(3.4) Micro Combustion Calorimetry calorimetry

The test apparatus is from FTT (standard model). The fire growth capacity (FGC), the specific Heat Release Rate (HRR(T)), the maximum of specific HRR(T) (Q max), the heat release temperature (T max), the specific (total) heat release (hc), the yield of pyrolysis residue (Yp), the specific heat of combustion of fuels gases (hc gas) are measured according to ASTM 7309.[5] The standard is performed on three replicates to calculate the mean and SD, according to method A. Fluxes of 80 mL/min of N_2 in the pyrolizer and 20 mL/min of O_2 in the combustor are set. The temperature of the combustor is adjusted at 750°C +/- 1 °C, and in the pyrolizer, a heating regime of 1 °C/min has been chosen up to 750°C. The sample's weight has been determined, evaluating the oxygen consumption of a trial test. The drop must stay between 20 % - 13 % and 20 % – 7 %.

The data are reported in Tables 11 and 12 of the paper.

References

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3. ASTM D 2863:2019; Standard Test Method for Measuring the Minimum Oxygen Concentration to Support Candle-Like Combustion of Plastics (Oxygen Index). ASTM International, West Conshohocken, PA. 2019. Available online: https://doi.org/ 10.1520/D2863-19, www.astm.org (accessed on 01/06/2023)
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