

Article A New Perspective on Hydrogen Chloride Scavenging at High Temperatures for Reducing the Smoke Acidity of PVC Cables in Fires. II: Some Examples of Acid Scavengers at High Temperatures in the Condensed Phase

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Citation: Sarti, G. A New Perspective on Hydrogen Chloride Scavenging at High Temperatures for Reducing the Smoke Acidity of PVC Cables in Fires. II: Some Examples of Acid Scavengers at High Temperatures in the Condensed Phase. *Fire* **2022**, *5*, 142. https://doi.org/10.3390/ fire5050142

Academic Editors: Ying Zhang, Boyun Liu, Xiaoyu Ju and Xianjia Huang

Received: 15 August 2022 Accepted: 15 September 2022 Published: 21 September 2022

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Copyright: © 2022 by the author. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). Reagens S.p.A., Via Codronchi, 4, 40016 San Giorgio di Piano, Italy; gianluca.sarti@fastwebnet.it

Abstract: In the European Union, according to Regulation (EU) n. 305/2011, cables permanently installed in residential and public buildings need additional classification for acidity. EN 60754-2 is the test method for assessing acidity, and three classes are provided: a1, a2, and a3 (less performant). The research on PVC compounds with low smoke acidity helps to produce cables in the best additional classes for acidity, giving the PVC cables the possibility to be used in medium and high fire risk locations. This paper shows the behavior of some acid scavengers at high temperatures performing EN 60754-2 in isothermal conditions at different temperatures between 400 °C and 950 °C. The test apparatus is a tube furnace where the test specimen is burnt, and the released gases are trapped in bubbling devices containing double deionized water. pH and conductivity are measured, the efficiency of the acid scavengers is assessed, and chemical analysis of the ashes is performed. That allows us to understand why some substances succeed and others fail in trapping hydrogen chloride (HCl). The most important conclusion in this paper is that the higher the temperature, the lower the performance of the acid scavenger, showing that HCl concentration in the gas phase depends strongly on the heating conditions of the test specimen.

Keywords: acid scavengers; PVC; cables; smoke acidity

1. Introduction

In the European Union (EU) in 2006, separate classes of reaction-to-fire performance were established for electric cables, including the additional classification for acidity, according to the Commission Decision of 27 October 2006, amending the Decision 2000/147/EC and implementing Council Directive 89/106/EEC, called Construction Product Directive or just CPD. At that time, the test method used to assess acidity was EN 50267-2-3 [1]. Since the Construction Product Regulation (Regulation (EU) n. 305/2011, or CPR) entered into force in 2017, EN 50267-2-3 became the test method for assessing smoke acidity, substituted later by EN 60754-2 [2]. EN 60754-2, EN 60754-1 [3] are similar to their siblings EN 50267-2-2 [4] and EN 50267-2-1 [5], used in EN 50525 series (see EN 50525-1 [6], annex B, table B2) to assess if a compound can be considered "halogen-free;" therefore, both are standards used by halogen-free producers. The paradox is that, while EN 60754-1 is a well-known and corroborated test method for PVC compound producers, EN 60754-2 is entirely unknown to them. The result is a historical lack of data on the pH and conductivities of PVC compounds for cables and difficulties in understanding how the classes a2 and a1 can be reached.

Some acid scavengers in the condensed phase at high temperatures are special calcium carbonates with fine particle sizes. In particular, precipitated calcium carbonate (PCC) was and still is the more suitable HCl scavenger for decreasing PVC compound smoke acidity. However, other extremely fine ground calcium carbonates (GCC) can be used [7,8]. In the past, the behavior of some HCl scavengers was evaluated, dynamically and statically, using different test apparatuses and conditions [9–12]. In particular, [12] shows how



different heating regimes can affect the evolution of HCl in the gas phase revealing how high temperatures and the absence of "gradual heating runs" hinder the action of the acid scavenger in trapping HCl.

This paper illustrates the behavior of 5 acid scavengers, alone and in combinations, performing EN 60754-2 in isothermal conditions for 30 min at 400 °C, 500 °C, 600 °C, 800 °C, and 950 °C. The performance has been evaluated through pH, conductivity, and efficiency. The efficiency has been calculated by the equation indicated in Part 1 of this paper [13]. This new way of evaluating the efficiency of acid scavengers has been proposed because simple and fast, involving a measure, the pH, directly quantified by EN 60754-2. The scientific community should test and evaluate it to establish its functionality and robustness.

Despite this, two main aspects can be highlighted when the potent acid scavengers have been evaluated in this research performing EN 60754-2 with isothermal profiles at different temperatures:

- the increase in the temperature severely affects their scavenging performance.
- at 950 °C, they go into crisis due to the too fast evolution of HCl in the gas phase.

This research has been developed in the regulatory context of smoke acidity in the EU, as explained in Part 1 of this paper [13].

2. Materials and Methods

2.1. Materials

Table 1 shows the first series of formulations. The amount of ingredients is expressed per hundred resin (phr). The formulation F50.0 represents the typical PVC jacket compound used in low voltage cables. The remaining formulations have been derived by F50.0, substituting CaCO₃ with different quantities of acid scavengers at high temperatures.

Table 1. First series of formulations: DINP means Di Iso Nonyl Phthalate. ESBO stands for Epoxidized Soy Oil Bean. The used antioxidant is Arenox A10, which is Pentaerythritol tetrakis(3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate), CAS number 6683-19-8. COS stands for Calcium Organic Stabilizer. PCC means Precipitated Calcium Carbonate. AS-1B and AS-6B are potent acid scavengers at high temperatures.

Raw Materials	Trade Name	F50.0 [phr]	F50.1 [phr]	F50.2 [phr]	F50.3 [phr]	F50.4 [phr]	F50.5 [phr]
PVC	Inovyn 271 PC	100	100	100	100	100	100
DINP	Diplast N	50	50	50	50	50	50
ESBO	Reaflex EP/6	2	2	2	2	2	2
Antioxidant	Arenox A10	0.1	0.1	0.1	0.1	0.1	0.1
COS	RPK B-CV/3037	3	3	3	3	3	3
CaCO ₃	Riochim	90	0	0	0	0	0
Al(OH) ₃	Apyral 40 CD	0	90	0	0	0	0
Mg(OH) ₂	Ecopyren 3.5	0	0	90	0	0	0
PCC	Winnofil S	0	0	0	90	0	0
HTAS 1	AS-1B	0	0	0	0	90	0
HTAS 2	AS-6B	0	0	0	0	0	90

The formulations in Table 2 are designed to test the effect on the efficiency of PCC and $Mg(OH)_2$ at high loading levels. Here we focused on the synergism increasing the scavenging efficiency and the growth of smoke acidity due to dispersion phenomena.

Table 3 displays the third series of formulations focused on the $Mg(OH)_2/PCC$ synergism.

The following materials have been used to perform EN 60754-2: Double Deionized Water (DDW) is internally produced by an ion exchange deionizer. The pH of DDW must be between 5.50 and 7.50, and the conductivity less than 0.5 μ S/mm. Buffer and conductivity standard solutions come from VWR International (pH: 2.00, 4.01, 7.00, and 10.00, conductivity: 2.0, 8.4, 14.7, 141.3 μ S/mm).

Table 2. Second series of formulations. pH and conductivity are measured according to EN 60754-2 at 950 °C. DINP means Di Iso Nonyl Phthalate. ESBO stands for Epoxidized Soy Oil Bean. The used antioxidant is Arenox A10, which is Pentaerythritol tetrakis(3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate), CAS number 6683-19-8. COS stands for Calcium Organic Stabilizer. PCC means Precipitated Calcium Carbonate.

Raw Materials	Trade Name	F50.6 [phr]	F50.7 [phr]	F50.8 [phr]	F50.9 [phr]	F50.10 [phr]
PVC	Inovyn 271 PC	100	100	100	100	100
DINP	Diplast N	50	50	50	50	50
ESBO	Reaflex EP/6	2	2	2	2	2
Antioxidant	Arenox A10	0.1	0.1	0.1	0.1	0.1
COS	RPK B-CV/3037	3	3	3	3	3
CaCO ₃	Riochim	0	0	0	0	0
Al(OH) ₃	Apyral 40 CD	0	0	0	0	0
Mg(OH) ₂	Ecopyren 3.5	0	130	40	80	104
PCC	Winnofil S	130	0	90	180	234

Table 3. Synergism of PCC/Mg(OH)₂ couple. The measures are performed according to EN 60754-2 at 950 °C. DINP means Di Iso Nonyl Phthalate. ESBO stands for Epoxidized Soy Oil Bean. The used antioxidant is Arenox A10, which is Pentaerythritol tetrakis(3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate), CAS number 6683-19-8. COS stands for Calcium Organic Stabilizer. PCC means Precipitated Calcium Carbonate.

Raw Materials	Trade Name	50.6 [phr]	50.7 [phr]	50.18 [phr]	50.8 [phr]	50.19 [phr]	50.20 [phr]	50.21 [phr]	50.22 [phr]	50.23 [phr]
PVC	Inovyn 271 PC	100	100	100	100	100	100	100	100	100
DINP	Diplast N	50	50	50	50	50	50	50	50	50
ESBO	Reaflex EP6	2	2	2	2	2	2	2	2	2
Antioxidant	Arenox A10	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
COS	RPK B-CV/3037	3	3	3	3	3	3	3	3	3
Mg(OH) ₂	Ecopyren 3.5	0	130	30	40	90	100	0	0	0
PCC	Winnofil S	130	0	100	90	40	30	100	90	40

2.2. Test Apparatus

Table 4 gives the list of utilized test apparatuses.

Table 4. Main test apparatuses utilized.

Test Apparatus	Producer	Model	Additional information
Plasticorder	Brabender	Palatograph EC	50 cm ³ chamber
Thermostat	Liebisch Labortechnik	LT-PVC-210-36-5	Test at 200 °C +/ $-$ 0.5 °C
Halogen Acid Gas test apparatus	SA Associates	Standard model	Porcelain combustion boats
Multimeter	Mettler Toledo	S213 standard kit	
Conductivity electrode	Mettler Toledo	S213 standard kit	
pH electrode	Mettler Toledo	S213 standard kit	
FTIR-ATR	Thermo Fisher Scientific	IS20	ATR correction to transmission
WD-XRF	Thermo Fisher Scientific	PERFORM'X	Borate Beads
Tensile testing machine	Hounsfield	H10KS	Specimen type 1A, 500 mm/min

2.3. Sample Preparation

PVC compound samples are prepared by weighing the stabilizers' ingredients in the 0.001 g balance. PVC, plasticizers, fillers, flame retardants, and acid scavengers are weighed in the 0.1 g balance. PVC and all the ingredients are mixed in a 20 L turbo-mixer up to 105 °C, producing 3 Kg dry-blend. The dry blend is processed in the plasticorder for 10 min at 160 °C, 30 rpm, getting 60 g kneaders. The kneaders are pressed at 160 °C for 4 min in 0.5 mm, 1 mm, and 6 mm sheets from which test specimens are obtained for the tests indicated in Tables 5 and 6.

Standard	Measurement	Temperature	Note
ISO 527-1 [14]	Elongation at break	23 °C	Test specimens conditioned for 24 h at 23 °C
ISO 527-1 [14]	Tensile strength	23 °C	Test specimens conditioned for 24 h at 23 °C
ISO 1183 [15]	Specific Gravity	23 °C	After 24 h of conditioning at 23 °C
ISO 868 [16]	Hardness	23 °C	Shore A at 15, test specimens conditioned for 24 h at 23 $^{\circ}$ C
IEC 60811-405 [17]	Thermal Stability	200 °C	Test specimens conditioned for 24 h at 23 °C

 Table 5. Tests for the main properties of the compound.

Table 6. Tests for acidity assessment.

Technical Standard	Measurement	Temperature [°C]	Note
EN 60754-2	Smoke acidity	950	DDW, pH, and conductivity. The general method, according to the 2014 version.
Internal Method 2	Smoke acidity	400, 500, 600, 800	DDW, pH, and conductivity. The general method is according to the 2014 version.

2.4. Internal Tests and International Technical Standards Used

Tables 5 and 6 recall the used technical standards.

General method of EN 60754-2 is performed as follows: a calibrated reference thermocouple is used to control the temperature. The probe is introduced in the central part of the quartz glass tube, where an empty combustion boat is carried from the sample carrier. The temperature measured by the reference thermocouple is adjusted to 950 + 1.5 °C, maintaining it for at least one hour. The tube furnace is ready for the first run when the temperature is stable. Then, a sample of 1.000 + / - 0.001 g is weighed in a combustion boat. The porcelain combustion boat has dimensions according to the standard. It is quickly introduced into the quartz glass tube, moving the magnet along the sample carrier, while the countdown is activated when the combustion boat reaches the central part of the quartz glass tube. The smokes are purged in the bubbling devices containing DDW for 30 min by a normalized air flux (set according to the standard EN 60754-2 considering the geometry of the quartz glass tube). After 30 min, the connectors are disconnected, and the magnet extracts the combustion boat from the quartz glass tube. The water of the bubbling devices and from washing procedures is collected in a 1 L volumetric flask filled to the mark, and pH and conductivity are measured. The precautions indicated in Part I of this paper have been adopted to minimize the errors, which lead to poor repeatability and reproducibility [13].

Internal method 2 is performed as EN 60754-2 but applies different isothermal profiles. That procedure permits the evaluation of the performance of an acid scavenger at different temperatures, evaluating pH, conductivity, and efficiency. pH and conductivity measures are taken at 25 + / - 1 °C with the following procedure: the multimeter is calibrated with standard solutions before each measurement. The pH is calibrated at two points (4.01 and 7.00). Conductivity is calibrated at 1 point at 141.3 μ S/mm. The solutions closer to the measured values 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 fluctuation of temperature.

The smoke acidity measurements usually have low repeatability, especially if the temperature is high. This weakness is intrinsic to dynamics affecting the sample burning in a tube furnace. Samples do not burn the same way, and passivation can lead to some fluctuations in the results. Furthermore, as indicated in Part 1 of this paper [13], most procedures are done manually, which is the most significant source of errors. Therefore, a series of three measurements for each sample is performed, and this statistical method is used to calculate the mean value and outliers: from three test determinations, the mean value (μ), standard deviation (*SD*), and coefficient of variation (*CV*) are calculated for pH and conductivity using the following formulations:

$$\mu_x = \frac{1}{N} \sum_{i=1}^N x_i \tag{1}$$

$$SD_x = \sqrt[2]{\frac{1}{N}\sum_{i=1}^{N} (x_i - \mu_x)^2}$$
 (2)

$$V_x = \frac{SD_x}{|\mu_x|} \tag{3}$$

If the *CV* is higher than 5%, further three measurements are performed, and the mean value, standard deviation, and coefficient of variation are recalculated, including the previous values. That is the general method according to EN 60754-2:2014. EN 60754-2/A1:2020 [18] has introduced a new procedure for evaluating the data not considered in this paper.

С

Appendix B, Figures A12 and A13 give a schematic diagram of the sample preparation and testing process.

3. Results

3.1. First Series of Formulations

Table 7 shows the main properties of the first series of formulations. The main properties give a preliminary indication if the compound is suitable for manufacturing jackets according to EN 50525 series.

Internal method 2 has been performed at different temperatures, 400 °C, 500 °C, 600 °C, 800 °C, and EN 60754-2 at 950 °C. Table 8 shows the results of pH and conductivity for each formulation. Table 9 reports the elements found in ashes determined by XRF spectrometry. Figures A1–A6 show the FTIR spectra of the ashes of F50.0–F50.5 at different temperatures. Figures A7–A11 display the FTIR spectra of some standards compared to the FTIR spectra of F50.0–F50.5 ashes. Table 10 gives the principal FTIR bands of the substances found in the ashes.

Formulation	F50.0	F50.1	F50.2	F50.3	F50.4	F50.5
Specific Gravity [g/cm ³]	1.542 +/-0.012	1.505 + / - 0.021	1.503 + / - 0.004	1.542 +/-0.007	1.445 + / - 0.014	1.446 + / - 0.009
Shore Hardness [type A, 15"]	88 +/- 1	89 +/-1	89 +/- 1	88 +/-1	90 +/- 1	90 +/-1
Tensile strength [N/mm ²]	13.0 + / - 0.8	11.8 +/- 0.5	11.7 +/- 0.8	13.4 +/- 1.1	13.0 +/- 1.0	13.1 + / - 0.7
Elongation at break [%]	246.4 +/- 3.8	236.1 +/- 4.9	233.1 +/- 2.5	240.1 +/- 2.4	221.5 +/- 1.7	225.5 +/- 1.5
Thermal Stability [min]	104 +/- 3	79 +/-2	73 +/- 4	76 +/- 5	291 +/- 11	299 +/- 12

Table 7. Focus on the main properties of the compounds. The mean values and the standard deviations are reported.

Table 8. Mean values and SD of pH and conductivities at 400 °C, 500 °C, 600 °C, 800 °C, and 900 °C.

Method 2 at 400 °C	F50.0	F50.1	F50.2	F50.3	F50.4	F50.5
pH	2.48 +/- 0.06	2.37 +/- 0.06	2.81 +/- 0.07	3.71 +/- 0.03	4.03 +/- 0.19	3.88 +/- 0.12
Conductivity [µS/mm]	142.9 +/- 3.6	179.4 +/- 1.5	88.7 +/- 3.5	8.1 +/- 0.3	4.0 + / - 0.1	5.3 +/- 0.1
Method 2 at 500 $^\circ C$	F50.0	F50.1	F50.2	F50.3	F50.4	F50.5
pН	2.48 +/- 0.04	2.41 +/- 0.03	2.41 +/- 0.09	3.73 +/- 0.10	3.70 +/- 0.15	3.69 +/- 0.13
Conductivity [µS/mm]	139.1 +/- 1.2	177.2 +/- 2.5	177.3 +/- 6.2	7.7 +/- 0.3	8.2 +/- 0.4	8.6 +/- 0.3
Method 2 at 600 °C	F50.0	F50.1	F50.2	F50.3	F50.4	F50.5
pH	2.51 +/- 0.02	2.30 +/- 0.01	2.31 +/- 0.03	3.69 +/- 0.07	3.70 +/- 0.10	3.65 +/- 0.05
Conductivity [µS/mm]	132.6 +/- 3.7	201.7 +/- 4.1	195.7 +/- 5.0	9.2 +/- 0.4	7.8 +/- 0.3	9.5 +/- 0.2
Method 2 at 800 $^\circ C$	F50.0	F50.1	F50.2	F50.3	F50.4	F50.5
pH	2.63 +/- 0.13	2.30 +/- 0.09	2.29 +/- 0.09	3.26 +/- 0.11	3.52 +/- 0.02	3.20 +/- 0.03
Conductivity [µS/mm]	100.4 +/- 4.4	206.4 +/- 2.5	208.9 +/- 7.8	23.7 +/- 0.6	13.5 +/- 0.2	25.7 +/- 0.6
Method 2 at 950 °C	F50.0	F50.1	F50.2	F50.3	F50.4	F50.5
pH	2.62 +/- 0.03	2.27 +/- 0.10	2.27 +/- 0.02	2.74 +/- 0.06	2.89 +/- 0.08	2.79 +/- 0.02
Conductivity [µS/mm]	97.3 +/- 3.7	221.5 +/- 8.4	224.3 +/- 3.1	74.0 +/- 1.6	70.1 +/- 0.7	70.1 +/- 2.0

Table 9. Main elements in ashes detected by XRF.

Formulation	F50.0	F50.1	F50.2	F50.3	F50.4	F50.5
400 °C	Ca, Cl	Mg, Cl	Al, Cl	Ca, Cl	n.a.	n.a.
500 °C	Ca, Cl	Mg, Cl	Al, Cl	Ca, Cl	n.a.	n.a.
600 °C	Ca, Cl	Mg, Cl	Al, Cl	Ca, Cl	n.a.	n.a.
800 °C	Ca, Cl	Mg, Cl	Al, Cl	Ca, Cl	n.a.	n.a.
950 °C	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.

n.a. = not available.

Table 10. Main FTIR bands in the ashes.

	$1 [cm^{-1}]$	2 [cm ⁻¹]	3 [cm ⁻¹]	4 [cm ⁻¹]	5 [cm ⁻¹]	6 [cm ⁻¹]	
CaCO ₃	2509.64	1794.92	1417.26	873.10	846.70	710.66	
MgCl ₂	1616.43	1606.44					
CaCl ₂	1628.43	1614.21					
MgO	Broadband centered at 548 $\rm cm^{-1}$						
Al_2O_3		Broa	dband betwee	n 400 and 900	cm^{-1}		

The ashes are analyzed through the following procedure. After the combustion boat is extracted from the tube furnace, it is left to cool down and put in a PE zip lock bag.

Before the measurement, the combustion boat and standards are dried for 2 h in the oven at 105 °C. Metal oxides or chlorides are in the ash residue and are highly hygroscopic. During FTIR measurements, we cannot exclude the possibility that the samples can reabsorb some water. The measurement should be taken as fast as possible. XRF is conducted using borate-fused beads.

3.2. Second Series of Formulations

Table 11 shows the pH and conductivity of the second series of formulations used to evidence the $Mg(OH)_2/PCC$ synergism.

Table 11. Synergism of PCC/Mg(OH)₂ couple. The measures are performed according to EN 60754-2 at 950 °C. The mean values and the SD are reported.

Formulation	F50.6	F50.7	F50.18	F50.9	F50.19	F50.20	F50.21	F50.22	F50.23
pН	2.93	2.34	3.19	3.75	2.65	2.51	2.90	2.89	2.33
	+/-0.06	+/- 0.04	+/- 0.11	+/- 0.08	+/-0.02	+/-0.04	+/- 0.01	+/- 0.00	+/-0.02
Conductivity	49.4	183.8	28.8	8.4	106.2	133.5	56.9	51.6	193.0
[µS/mm]	+/-1.4	+/- 9.0	+/- 0.3	+/- 0.3	+/- 4.2	+/- 6.2	+/- 2.3	+/- 1.8	+/- 3.0

3.3. Third Series of Formulations

Table 12 shows the main properties of the formulations F50.6–F50.10 and pH and conductivities, focusing on the couple PCC/Mg(OH)₂ and the effect of its high loading level on acidity (the decrease of the smoke acidity performance due to the reduced dispersion of both additives). The smoke acidity has been measured using EN 60754-2 at 950 °C.

Table 12. Second series of formulations. Main properties of the compound. Mean values and SD are reported.

Formulation	F50.6	F50.7	F50.8	F50.9	F50.10
pH	2.93 + / - 0.06	2.34 + / - 0.02	3.32 +/- 0.06	3.75 + / - 0.08	3.75 + / - 0.09
Conductivity [µS/mm]	49.4 + / - 6.1	183.8 + / - 9.0	20.7 + / - 2.6	8.4 + / - 1.0	8.4 + / - 1.0
Specific Gravity [g/cm ³]	1.644 + / - 0.011	1.590 + / - 0.014	1.627 + / - 0.002	1.853 + / - 0.007	1.946 + / - 0.007
Hardness [SHA 15"]	91 + / - 1	92 + / - 1	91 + / - 1	97 + / - 1	98 + / - 1
Tensile strength [N/mm ²]	8.2 +/- 0.5	5.8 +/- 0.3	7.5 +/- 0.7	2.3 +/- 1.3	1.2 +/- 1.3
Elongation at break [%]	210.3 +/- 3.2	195.1 + / - 5.2	205.0 + / - 1.1	177.0 + / - 7.5	172.0 + / - 8.5
Thermal Stability [min]	76 + / - 5	71 + / - 3	61 + / - 2	57 + / - 4	38 + / - 3

3.4. The Efficiency of Scavenging

Tables 13–15 display the efficiency values, performing EN 60754-2 and the internal method 2 at the indicated temperatures.

Table 13. The mean efficiency and the SD of formulations F50.0–F50.5: EN60754-2 at different temperatures.

Temperature [°C]	F50.0	F50.1	F50.2	F50.3	F50.4	F50.5
400	8.0 +/- 1.5	5.1 + / - 1.6	16.9 + / - 1.9	40.0 + / - 0.8	50.8 + / - 5.1	45.1 +/- 3.1
500	8.1 + / - 1.0	6.3 + / - 0.7	6.3 + / - 2.4	40.6 + / - 2.6	41.8 + / - 4.1	40.2 + / - 3.4
600	8.6 + / - 0.4	3.1 + / - 0.3	3.4 + / - 0.7	39.7 + / - 1.8	40.9 + / - 2.7	38.9 + / - 1.2
800	12.0 + / - 3.5	3.3 + / - 2.5	3.1 + / - 2.3	28.2 + / - 2.8	36.9 + / - 0.7	27.1 + / - 0.9
950	12.2 + / - 0.7	2.4 + / - 2.6	2.3 + / - 0.4	14.7 + / - 1.5	18.7 + / - 2.2	16.0 + / - 0.4

Table 14. The mean efficiency and SD of formulations F50.6–F50.10: EN60754-2 at 950 °C.

	F50.6	F50.7	F50.8	F50.9	F50.10
950 °C	18.4 + / - 1.6	2.4 + / - 0.5	28.8 +/- 1.5	37.5 +/ - 2.3	34.9 +/- 2.7

 F50.18
 F50.19
 F50.20
 F50.21
 F50.22
 F50.23

 950 °C
 25.2 +/- 2.8
 10.8 +/- 0.4
 7.0 +/- 0.9
 18.5 +/- 0.3
 18.6 +/- 00
 6.3 +/- 0.5

4. Discussion

4.1. Description of Used Acid Scavengers and Why They Have Been Selected

The acid scavengers have been chosen for different needs:

- GCC and PCC highlight the impacts of particle size on scavenging efficiency.

Table 15. The mean efficiency and SD of formulations F50.18–F50.23: EN60754-2 at 950 °C.

- AS-1B and AS-6B are mixtures of basic substances (different kinds and concentrations), comparing their performances with PCC, the standard acid scavenger for PVC.
- Al(OH)₃ shows the behavior of an inert and scarcely reactive acid scavenger.
- Mg(OH)₂ shows what happens when the reaction product with HCl decomposes.

4.2. Effect of Chemical Properties of a Substance on Efficiency

The chemical properties of acid scavengers greatly influence efficiency. For example, AS-6B (a mix of basic substances) shows higher efficiency than GCC or Al(OH)₃ (Tables 8 and 13). That confirms what was outlined by O'Mara in [19] and Brown and Martin in [20]. Thus, substances with high reactivity with HCl are always a good starting point for their evaluation as acid scavengers at high temperatures in the condensed phase. Figure 1 shows the efficiency of AS-6B, GCC, and Al(OH)₃ at different temperatures, performing internal method 2.



Figure 1. Efficiency % (T) of F50.5 (Orange), F50.0 (Gray), and F50.2 (Blue). Standard deviation ranges are reported. ATH stands for Al(OH)₃.

4.3. Effect of Particle Size on Efficiency

GCC has less efficiency than PCC (Tables 8 and 13), which is valid for all temperatures. F50.0 contains a GCC with a mean particle size of around 2 microns, while F50.3 has Winnofil S, a PCC having a particle size in the scale of nanometers and a BET between $15 \text{ m}^2/\text{g}$ and $24 \text{ m}^2/\text{g}$ [21]. O'Mara made the same assumption without any measurement in [19], claiming that the Molar Absorption Efficiency (MAE) depends on several variables, such as acid scavengers' dispersion efficiency and particle size. Matthews and Plemper in [7,8] have also shown how the reactivity of CaCO₃ is linked to particle size, and it can cause a substantial effect on flame retardancy. We will discuss this aspect in detail in Part IV of this paper.

In conclusion, substances with finer particle sizes show more reactivity with HCl because of a higher BET, which means more probability of intercepting gaseous HCl molecules. For this reason, PCC is more performant in scavenging than GCC.

One of the points to be highlighted is that the advantage of PCC is reduced as temperature increases, and at 950 °C, none of the acid scavengers at high temperatures can compete with the fast evolution of HCl (Table 13 and Figures 1 and 2).



Figure 2. The efficiency % (T) of F50.0 containing 90 phr of GCC (gray), and F50.3, containing 90 phr of PCC (yellow). Standard deviation ranges are reported.

4.4. Effect of Dispersion on Efficiency

The formulation F50.8 contains PCC and $Mg(OH)_2$, 90 phr, and 40 phr, respectively, with a ratio of PCC/Mg(OH)₂ of 2.25. That is the best ratio found for the best efficiency. The efficiency of this acid scavenger couple is 28.8% (Table 14, Figure 3), with a pH of 3.32 (Table 12). By doubling the quantity of the couple with the same ratio, 37.5% of efficiency with a pH of 3.75 are reached (Tables 12 and 14). With a further improvement of the dosage, no further advantages are obtained (Table 14 and Figure 3). This behavior indicates poor dispersion as the dosage increases, impacting the scavenging performances. An inadequate

distribution weakens the intimate contact between PVC chains and acid scavengers. If this contact is missing in some zones, HCl is released, lowering the efficiency of acid scavengers. Sometimes, this can be compensated by increasing the shear during the process and using some tricks during the blending. Nevertheless, nanoscale fillers and substances prone to uptake water, such as Mg(OH)₂, are always inclined to give bad dispersion if we enhance their dosage. O' Mara in [19] claimed the impacts of the dispersion of acid scavengers on their efficiency.



Figure 3. Efficiency % (T) of formulations containing $PCC/Mg(OH)_2$ at different dosages. Standard deviation ranges are reported.

4.5. Effect of Temperature on Efficiency

AS-6B gives stable reaction products with HCl, so the efficiency is only due to the kinetic of the reactions involved in trapping HCl. Its acid scavenger efficiency falls dramatically as temperature increases (Figure 4). The efficiency of other good acid scavengers, such as PCC, shows a similar trend (Figure 2). The explanation lies in the competition between two reactions. HCl evolves from the burning matrix, and acid scavengers try to fix it in ashes. The higher the temperature, the quicker the evolution of HCl. Over certain temperatures, the solid acid scavenger is too slow to trap HCl efficiently, and the system goes into crisis. Chandler and alt. in [12] and Bassi in [22] highlighted this phenomenon. Finally, a non-performant acid scavenger such as Al(OH)₃ fails at all tested temperatures.



Figure 4. Efficiency % (T) of formulation F50.1 (blue) and F50.5 (orange) as a function of temperature. Standard deviation ranges are reported. ATH stands for Al(OH)₃.

4.6. Effect of Decomposition of Reaction Products on Efficiency

Kipouros and Sadoway claimed the MgCl₂ decomposition last step at 550 °C [23]. Galwey and Laverty between 350 °C and 550 °C [24]. The efficiencies reported in Figure 5 and FTIR spectra in Figure A3 confirm that over 400 °C, MgCl₂ decomposes. The ashes at 400 °C are black, and the FTIR spectrum at 400 °C shows the presence of an unknown substance, probably a mix of a variety of crosslinked organic compounds forming a black and solid char. Here, the second stage of pyrolysis and combustion zone starts, and the formation and rearrangement of the crosslinked matrix are expected. However, the FTIR spectrum shows the presence of weak MgCl₂ bands at 1616.4 cm⁻¹ and 1606.1 cm⁻¹, confirming that MgCl₂ is diluted in the black char (Figure A3 at 400 $^{\circ}$ C). The signal of the organic crosslinked char entirely disappears at 500 °C, and therefore the MgCl₂ bands become evident. Nevertheless, at 500 °C, the principal band at 546.2 cm⁻¹ confirms the presence of MgO (Figures A3, A9, and A11, Tables 9 and 10). MgCl₂ bands vanish completely at 600 °C, where MgO is the only source of Mg (Figures A3 and A11 and Tables 9 and 10). The decomposition of $MgCl_2$ is why the efficiency of $Mg(OH)_2$ decreases after 400 °C. Therefore, by performing internal method 2, $Mg(OH)_2$ shows the maximum efficiency at 400 °C (16.9%), and its efficiency drops to 2.3% at 950 °C.



Figure 5. Efficiency % (T) of the formulation F50.2 containing Mg(OH)_{2.} Standard deviation ranges are reported.

4.7. Focus on the Single Acid Scavenger

4.7.1. Single-Step Reaction: GCC

GCC reacts with HCl in a single-step reaction, yielding CaCl₂, CO₂, and water. Its reaction product, CaCl₂, is stable up to 950 °C. The efficiency of GCC in F50.0 remains low, slightly increasing from 400 °C to 950 °C (Table 13), probably due to the formation of small quantities of CaO, which is more likely to happen at 800 °C and 950 °C, acting as a potent acid scavenger. This phenomenon gives a slight advantage to all kinds of CaCO₃ in the scavenging at temperatures over 800 °C. In PCC, this advantage is not visible (F50.3) because "covered" by PCC's high scavenging performances. FTIR (Figures A1 and A10, and Table 10) and XFR (Table 9) point to the involved reactions. At 400 °C, 500 °C, and 600 °C, CaCO₃ and CaCl₂ are in the ashes. At 800 °C, the decarbonation of CaCO₃ takes place when its bands disappear, leaving only CaCl₂ bands. Chandler and others highlight the tendency of CaCl₂ to be hydrolyzed over its fusion point by water vapor when watersaturated air fluxes are used [12]. Nevertheless, with dry air fluxes at 950 °C, CaCl₂ is a stable, transparent liquid, not showing any tendency to be hydrolyzed.

4.7.2. No Reaction: Al(OH)₃

Al(OH)₃ starts the decompositions between 180 °C and 200 °C, releasing water [25]. Therefore, during the combustion, the actual substance in the matrix is alumina (Al₂O₃). Al₂O₃ is an inert substance not capable of reacting with HCl. Figure A2 shows the FTIR spectra of the ashes of formulation F50.1 at different temperatures. Figure A8 indicates that all spectra of Figure A2 have an excellent match with Al₂O₃ (see also Table 10), and therefore alumina is in the ashes obtained at 400 °C, 500 °C, 600 °C, 800 °C, and 950 °C. The presence of Al is confirmed by XFR (Table 9), and chlorine is probably due to HCl trapped in the alumina surface. Additional measurements should clarify the presence of Cl in the F50.1 ashes. All these considerations explain why formulation F50.1 gives low and constant efficiency values at different temperatures (Figure 4 and Table 13). Hence, Al(OH)₃ is an extremely weak acid scavenger at high temperatures due to the chemical inertia of its reaction product, Al₂O₃.

4.7.3. Single-Step Reaction: Mg(OH)₂

Mg(OH)₂ reacts fast with HCl generating MgCl₂. Mg(OH)₂ starts the decompositions between 300 °C and 320 °C, releasing water [25]. Thus, it is a perfect flame retardant; nevertheless, it is an ineffective acid scavenger at temperatures over 500 °C. The for-

mulation F50.2 gives maximum efficiency at 400 °C, suddenly dropping down due to the instability of its reaction product, MgCl₂ (Table 13). Water vapor hydrolyzes MgCl₂ through the reactions reported in [23,24]. The result is the production of 2 moles of HCl and 1 mole of MgO per mole of decomposing MgCl₂. The ashes analysis (FTIR measures in Figures A3, A9 and A11, and Table 10) shows that the MgCl₂ hydrolysis is almost complete over 600 °C. Table 9 indicates that chlorine remains trapped in ashes (maybe passivation, preserving small quantities of MgCl₂, but further measurements should clarify this point).

All this puts in evidence a single-step reaction failing the scavenging at high temperature because Mg(OH)₂ yields an unstable product rereleasing HCl.

Table 8 shows how the formulations F50.1 and F50.2 reach almost the same pH and conductivities, indicating high acidity for different causes. As Al(OH)₃, Mg(OH)₂ is a very ineffective acid scavenger at high temperatures in the condensed phase, and therefore it does not show any effect in efficiency as temperature increases (Table 13). Again, a weak acid scavenger usually performs poorly at all temperatures.

4.7.4. Single-Step Reaction: PCC

Tables 8 and 13 show that PCC alone is a good acid scavenger. PCC is not a strong base, and due to its small particle size, it can scavenge HCl with good efficiency, yielding CaCl₂, CO₂, and water. CaCl₂ is stable at 950 °C. FTIR spectra confirm the formation of CaCl₂ and the disappearance of CaCO₃ bands (Figures A4 and A7, and Table 10) starting from 800 °C. The XRF measurements (Table 9) show the presence of chlorine and calcium. PCC shows good scavenging up to 800 °C.

4.7.5. Multiple-Step Reaction: PCC and Mg(OH)₂

Table 14 indicate that, if alone, Mg(OH)₂ shows a low impact on smoke acidity reduction. At 950 °C, the efficiency of F50.2 is extremely low, 2.3%. On the contrary, PCC performs better, and the efficiency reaches 14.7% in F50.3. Table 14 also shows that they have a strong synergism when used together. Figure 6 represents the efficiency at different ratios of loadings. Mg(OH)₂ and PCC reach maximum efficiency (28.8%) when PCC is 90 phr and Mg(OH)₂ 40 phr (F50.8). That efficiency is much more than the sum of the efficiency of Mg(OH)₂ and PCC alone (14.7% and 2.3%, respectively). The maximum is reached when the ratio PCC/Mg(OH)₂ is 2.25, as Figure 6 shows. Probably the ratio that gives the maximum efficiency depends on the quantity of the pair and particle size of PCC and Mg(OH)₂ because both substantially affect the scavenging performance. The synergism can be explained by a double-step reaction through which Mg(OH)₂ and PCC help each other. As a strong base, Mg(OH)₂ is the main reaction product. The reactions during the matrix combustion are the following:

$$Mg(OH)_2 + 2HCl (g) \rightarrow MgCl_2 (s) + 2H_2O (v)$$
(4)

$$MgO(s) + 2HCl(g) \rightarrow MgCl_2(s) + H_2O(v)$$
(5)

The second acid scavenger, PCC, reacts with HCl in less extent through reaction 6.

$$CaCO_{3}(s) + 2HCl(g) \rightarrow CaCl_{2}(s, l) + CO_{2}(g) + H_{2}O(v)$$
(6)

MgCl₂ is formed but decomposes between 350 °C and 550 °C [23,24] with a slow kinetic passing back its HCl to PCC. The synergism is explainable with a slow HCl release from MgCl₂ to PCC, enhancing the efficiency of PCC. All these aspects should be clarified deeper by FTIR-TGA measurements, pointing to the IR signals of HCl and CO₂ in the range 350 °C–550 °C. That will be discussed in a separate article. Schemes 1–3 represent the hypothesis on the synergism between PCC and Mg(OH)₂.



F50.18

Figure 6. The efficiency of the formulations F50.7 (Mg(OH)₂ = 130; PCC = 0), F50.8 (Mg(OH)₂ = 40; PCC = 90), F50.18 (Mg(OH)₂ = 30; PCC = 100), F50.19 (Mg(OH)₂ = 90; PCC = 40), F50.20 (Mg(OH)₂ = 100; PCC = 30). Standard deviation ranges are reported.

F50.19

F50.20

Pre pyrolysis and combustion zone	Pyrolysis &Combustion Zone	Post Pyrolysis &Combustion Zone			
PCC		CaCl ² stable			
23 50 77 104 131 158 185 212 239 26	6 293 320 347 374 401 428 455 482	509 536 563 590 617 644 671 698 725	752 790 800 810	837 864 891	935 948 965 975

Temperature [°C]

2.4 I

F50.7

F50.8

0

Scheme 1. The single-step reaction of PCC in HCl scavenging at high temperatures in the condensed phase.

Pre pyrolysis and combustion zone	Pyrolysis &Combustion Zone	Post Pyrolysis &Combustion Zone				
Mg(OH)2	MgClz	MgO + HCI (HCI reaches the bubbling devices)				
23 50 77 104 131 158 185 212 239 266	293 320 347 374 401 428 455 482	509 536 563 590 617 644 671 698 725 752	790 800 810	837 864 891	935 948 965	975

Temperature [°C]

Scheme 2. The single-step reaction of $Mg(OH)_2$ in HCl scavenging at high temperatures in the condensed phase.

Pre pyrolysis and combustion zone	Pyrolysis &Combustion	Post Pyrolysis &Combustion				
	Zone	Zone				
PCC less performant		CaCl ₂ stable				
Mg(OH)2 more performant	MgClz	HCI + MgO				
23 50 77 104 131 158 185 212 239 266	293 320 347 374 401 428 455 482	509 536 563 590 617 644 671 698 725 75.	2 790 800 810	837 864 891	935 948 965	975

Temperature [°C]

Scheme 3. Multiple-step reactions of $PCC/Mg(OH)_2$ in HCl scavenging at high temperatures in the condensed phase.

5. Conclusions

Acid scavengers at high temperatures in the condensed phase differ in chemical nature and particle size, and some of them have been tested with different thermal profiles, performing EN 60754-2. Al(OH)₃ and Mg(OH)₂ are not efficient acid scavengers. When the matrix burns, the former generates an inert substance, alumina, while the latter reacts with HCl, yielding MgCl₂, which decomposes over 500 °C, rereleasing HCl.

GCC is not a good acid scavenger, but its efficiency increases as its particle size decreases. PCC is, therefore, more efficient in scavenging HCl than GCC. It reacts well with HCl in a single-step reaction, yielding CaCl₂ stable up to 950 °C. When Mg(OH)₂ and PCC work together, PCC scavenges the HCl from MgCl₂ decomposition. Probably the synergism happens because MgCl₂ releases HCl slower than the PVC matrix, and PCC can scavenge it more efficiently. Poor dispersion eliminates all advantages of the synergism when the quantity of Mg(OH)₂ and PCC is too high.

Another aspect emerging from the data is the effect of temperature on the kinetic of HCl release. The higher the temperature, the lower the acid scavenger efficiency. This aspect confirms other researchers' past observations [12,22]. The rapid HCl evolution causes the collapse of the efficiencies of acid scavengers at high temperatures. Acid scavengers are solid substances, and while some advantages are obtained when a lower particle size grade is used, they are annihilated when temperatures are too high.

In conclusion, it must be highlighted that different heating regimes give different acidity results. This aspect confirms how difficult it is to estimate the HCl concentration in real fire scenarios from bench-scale tests because HCl concentration in the gas phase will depend not only on its decay [26–28] but also on temperatures reached in the fire.

Supplementary Materials: The supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/fire5050142/s1.

Funding: This research received no external funding.

Institutional Review Board Statement: Not applicable.

Data Availability Statement: The data presented in this study are available in Supplementary Materials.

Acknowledgments: The author wants to acknowledge Ing. Carlo Ciotti, Ing. Marco Piana, all PVC Forum Italia and PVC4cables staff, and Emma Sarti for their help and support.

Conflicts of Interest: The authors declare that there is no conflict of interest regarding the publication of this paper.

Glossary/Nomenclature/Abbreviations

- PVC poly(vinyl chloride);
- HCl hydrogen chloride;
- EU European Union;
- CPD Construction Product Directive;
- CPR Construction Product Regulation;
- PCC Precipitated Calcium Carbonate;
- CCC Ground Calcium Carbonate;
- Phr Part per Hundred Resin;
- DINP Di Iso Nonyl Phthalate;
- ESBO Epoxidized Soy Bean Oil;
- COS Calcium Organic Stabilizer;
- DDW Double Deionized Water;
- μ Mean
- SD Standard Deviation;
- CV Coefficient of variation;
- MAE Molar Absorption Efficiency



Appendix A. FTIR Spectra of the Acid Scavengers at Different Temperatures and Some Substances Used to Confirm the Chemical Nature of the Substances in Ashes









Figure A3. FTIR spectra of ashes of F50.2 at 950 °C (red), at 800 °C (light blue), at 600 °C (green), at 500 °C (purple), at 400 °C (blue).



Figure A4. FTIR spectra of ashes of F50.3 at 950 $^{\circ}$ C (red), at 800 $^{\circ}$ C (light blue), at 600 $^{\circ}$ C (green), at 500 $^{\circ}$ C (purple), at 400 $^{\circ}$ C (blue).



Figure A5. FTIR spectra of ashes of F50.4 at 950 $^{\circ}$ C (red), at 800 $^{\circ}$ C (light blue), at 600 $^{\circ}$ C (green), at 500 $^{\circ}$ C (purple), at 400 $^{\circ}$ C (blue).



Figure A6. FTIR spectra of ashes of F50.5 at 950 °C (red), at 800 °C (light blue), at 600 °C (green), at 500 °C (purple), at 400 °C (blue).



Figure A7. FTIR spectra: anhydrous CaCl₂ vs. ashes of F.50.3 at 600 °C: (**a**) scale 4000–400 cm⁻¹ and (**b**) focus in the range 1660 cm⁻¹–1580 cm⁻¹, anhydrous CaCl₂ (blue) and ashes of F.50.3 (red). Anhydrous CaCl₂ shows two bands at 1628.43 cm⁻¹ and 1614.21 cm⁻¹, and the sample spectrum matches them perfectly.



Figure A10. FTIR spectra: CaCO₃ versus ashes of F50.0 at 400 °C. F50.0 shows all the main CaCO₃ bands: 2509.64 cm⁻¹, 1794.92 cm⁻¹, 1417.26 cm⁻¹, 873.10 cm⁻¹, 846.70 cm⁻¹ and 710.66 cm⁻¹.



Figure A11. FTIR spectra: anhydrous MgCl₂ versus ashes of F50.2 at 500 °C. (a) scale 4000–400 cm⁻¹ and (b) focus in the range 1660 cm⁻¹–1580 cm⁻¹, anhydrous MgCl₂ (red) and ashes of F50.2 (blue). The FTIR spectrum of the ashes matches the main bands of anhydrous MgCl₂ (1616.43 cm⁻¹ and 1606.44 cm⁻¹ and a minor band at 1653.26 cm⁻¹). Another band is present at 1629.91 cm⁻¹. The different structure of the spectrum of the ashes is probably due to different hydration states of MgCl₂.



Appendix B. A Schematic Diagram of the Sample Preparation and Testing Process

Figure A12. A schematic diagram of the sample preparation.



Figure A13. A schematic diagram of the testing process and main conditions.

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