Synergies of Metal Hydroxides and Metal Molybdates in Low-Smoke Flexible PVC

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Abstract

Various flame retardant additives have been used to formulate low-smoke flexible PVC for cable applications. Metal hydroxides, which primarily consist of aluminum trihydrate (ATH) and magnesium dihydroxide (MDH), are most commonly used. Typically used at high loading levels, these provide fire performance via endothermic cooling while also contributing to char formation. Molybdates are known to promote char formation during combustion by the Friedel-Crafts alkylation of alkene linkages that are formed during PVC thermolysis.

The most rigorous standards for flame and smoke propagation have been established for plenum rated communication cable. To meet the NFPA 262 standard, PVC jacket compound is typically compounded with combinations of additives. While the mechanism for individual additives in simple polymer systems may be well understood, interactions between additives have been much less studied. These interactions can result in enhanced performance through synergies, or in some cases, diminished performance through antagonistic effects.

In this work we examine the potential synergies between metal hydroxides (both ATH and MDH) and metal molybdates in several different flexible PVC formulas. Performance testing was conducted using the cone calorimeter and NBS smoke chamber. Thermal techniques were also used to gain insight into chemical interactions. The data generated provide strategies for the formulation of cost effective, low smoke PVC jacket compound.

Keywords: Magnesium hydroxide, aluminum trihydroxide, zinc molybdate, calcium molybdate, flexible PVC, low smoke, jacket compound, char formation. Introduction

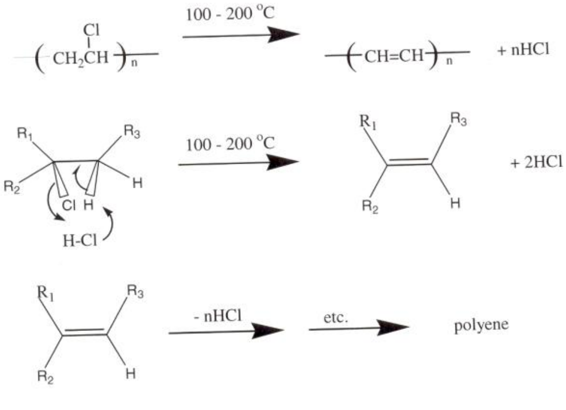
1. Introduction

Wire and cable applications are increasingly faced with meeting both flame spread and smoke standards. The most stringent standards have been established for cable applications in ceiling cavity plenums. The standard for plenum cable was established by the National Fire Protection Association (NFPA) and is based on NFPA 262, a modified Steiner Tunnel Test. To qualify for use, flame spread must not exceed 5 feet. In addition, peak smoke must not exceed 0.5 units and the average smoke throughout the test must be below 0.15 units.

Because of the NFPA requirements, the preferred materials for plenum rated communication jacketing are fluorinated ethylene propylene (FEP) and plasticized polyvinyl chloride (PVC). PVC enjoys a significant cost advantage as well as processing advantages. However, a number of additives are required in order to meet the various technical requirements. Plenum grade PVC jacket compound generally contains several flame retardant additives. Antimony trioxide, Sb_2O_3, is often added to PVC compounds and has been shown to enhance flame retardancy via a gas phase mechanism involving the scavenging of free radicals. In addition, flame retardancy can be improved by the use of halogenated and/or phosphate plasticizers. A deleterious consequence of the use of antimony and halogen flame retardants is the increase in smoke.

The first step in the thermal decomposition of PVC is dehydrochlorination of PVC accompanied by the release of HCl and the formation of a polyene. This process is autocatalytic since the reaction product HCl accelerates the rate of thermal decomposition. (Figure 1)[1]

Figure 1. Thermal Decomposition of PVC



The formation of smoke during combustion of PVC involves the cis,trans form of the polyene. On the other hand, both cis,trans and trans,trans polyenes form a cross-linked polymer, which can then form char. From previous studies, it has been determined that plasticized PVC produces much more smoke than char.[2]

In order to control smoke during combustion, molybdate based smoke suppressants are commonly added to low smoke PVC compound. Molybdenum based smoke suppressants chemically influence the formation of char to lower smoke. MoO_3 has been shown to react with the HCl produced by the pyrolysis of PVC to form MoO_2Cl_2, a potent Lewis acid. This can then promote crosslinking in a number of ways. One likely mechanism is the Friedel-Crafts alkylation of alkene linkages that are formed during PVC thermolysis.[3,4,5]

ATH and MDH are among several alternative "green" fire retardants being used to reformulate fire-rated PVC compounds in order to be regulatory compliant while maintaining cost effectiveness. Continued interest in delivering low-cost compound products with maximized performance has driven development of cost-performance effective ATH and MDH products. Both ATH and MDH function as a flame retardant by decomposing endothermically into water and metal oxide upon heating. The released water vapor, in an amount of about a third of total metal hydroxide used, dilutes the combustible ambience while removing the combustion heat. The oxide by-product forms chars on the surface of polymeric materials to prevent heat and oxygen from approaching the polymer fuel.

Fig. 2. Decomposition temperature of ATH and MDH measured by TGA

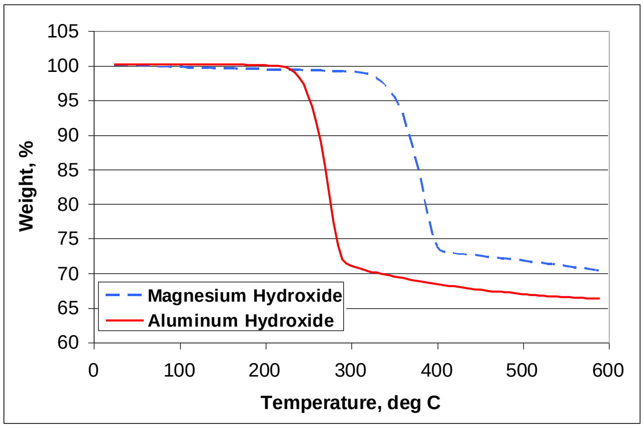


Figure 2 shows the thermal decomposition of ATH and MDH as measured by thermogravimetric analysis. The main difference between ATH and MDH is their temperature of decomposition – MDH decomposes at a temperature which is about 100°C higher than ATH. Both are appropriate for use in formulating low-smoke PVC compounds given the relatively low processing temperatures.

Earlier studies[6] showed that for plasticized PVC compounds, there may be a favorable synergism in smoke suppression between antimony oxide and ATH with an optimal ratio of ATH to antimony effectively reducing and delaying the smoke generation. It was also shown that for the flexible PVC compounds studied, there appeared to be no smoke performance synergism between ATH and MDH.

In another Huber's study[7] it was shown that MDH outperforms ATH as heat and smoke suppressant in flexible PVC formulations. At the same time, ATH-based flexible PVC formulations have higher LOI values.

In commercial practice, combinations of ATH, MDH and molybdate smoke suppressants are required to achieve targeted smoke values. The development of low smoke formulations can require extensive experimentation and anecdotal evidence of synergies between various additives are often cited. However, exact formulation compositions are rarely disclosed and are considered to be trade secrets. In this work we examine the interactions between metal hydrates (both ATH and MDH) and metal molybdates in a model flexible PVC formula. The data generated provide scientific strategies for the formulation of cost effective, low smoke PVC jacket compound.

2. Materials

2.1 Metal Hydroxides

A fine precipitated ATH grade, Micral 9400 made by J. M. Huber Corporation was used in this study. This product has a nominal median particle size of 1 micron with a uniform particle size distribution, surface area 5 m^2/g. A fine synthetically produced MDH grade, called Vertex 90 made by J. M. Huber Corporation with a nominal median particle size of 1.5 microns and surface area 13m^2/g was also used. Both Micral 9400 ATH and Vertex 90 MDH are designed for use in wire and cable compounds providing a desired balance of performance and economics.

Vertex 90 and its sister MDH product, Vertex 100 of Huber, are characterized by their uniform particle size distribution and relatively higher surface area of 13-14 m^2/g, made by Huber's proprietary process technologies. Previous performance compound studies by Huber [2] demonstrated both MDH grades as equally effective in reducing smoke density for cable compounds made from PVC or polyolefins.

2.2 Metal Molybdates

Two different molybdate based smoke suppressants manufactured by Sherwin-Williams Chemicals were included in this study. Kemgard 911C is a zinc molybdate based material with an inert talc core material. Kemgard 501 is a calcium molybdate based material with a calcium carbonate core. Both metal molybdate based smoke suppressants are produced by a patented process in which the metal molybdate is precipitated on to the surface of the mineral core. This approach makes more efficient use of the molybdate species by maximizing the active surface area.

Previous thermal analysis has shown that zinc molybdate lowers the temperature at which char is formed in PVC. While this may partially account for the mechanism of smoke suppression, it can also result in diminished thermal stability. On the other hand, calcium molybdate has very little effect on the onset temperature of char formation but does promote a more thermally robust char.

Both zinc and calcium molybdates have found wide use in various low smoke PVC applications.

2.3 PVC Compound

All flexible PVC compounds were based on the formulation, as shown in Table 2. The PVC resin, Oxvinyl 240F, had an inherent viscosity of 1.02, a relative viscosity of 2.37, and a *K* value of 70. Two different plasticizers were used, Santicizer 2148, a alkyl/aryl phosphate ester, from Ferro and Uniplex FRP-45, a brominated phthalate from Unitex.

Table 1. Model flexible PVC formulation.

Ingredient	Parts by weight	Supplier
PVC Resin	100	**Oxyvinyl**
RC204P Stabilizer	3	**Ferro**
Antimony Oxide	3	
Santicizer 2148	20	**Ferro**
Uniplex FRP-45	20	**Unitex**

The flexible PVC specimens were fused using a 75-mL Brabender Plasti-Corder Digi-System equipped with Type 6 roller blades (3:2 speed ratio). The fusion process parameters included a mixing temperature of 160^0C and a mixing time of 5 minutes. The fused samples were compression molded with a Carver press at 300^0F

3. Test Methods

3.1 NBS Smoke Chamber

The smoke generated by the combustion of the plaques was determined by using the National Bureau of Standards (NBS) Smoke Chamber method, standardized in the United States as ASTM-E662. Test plaques (75 x 75 x 3 mm) were subjected to a radiant heat flux of 25 kW/m^2, and all tests were run in the flaming mode. The maximum smoke densities, as well as the smoke density at various times were recorded; average deviations for duplicate burns generally were ≤ 10%.

3.2 Pyrolysis Combustion Flow Calorimeter

Lyon, Walters and coworkers at the Federal Aviation Administration Laboratory recently developed Pyrolysis Combustion Flow Calorimeter (PCFC) to study, on a small scale, the FR properties of polymeric materials. The method has recently standardized according to ASTM D7309. In this technique, heat release rates are determined as a function of temperature. The total heat release (THR) is the integral of the heat release rate/time curve.

3.3 Statistical Analysis of Smoke Data

Design-Expert Version 7 a computer–aided statistical software by Stat-Ease Inc. was utilized to generate the design of experiments (DOE) for examining the synergies between the three flame retardant additives. The DOEs were optimized using a D-optimal mixture designs. Per design, sixteen points (formulations) were suggested for evaluating the design space. These points consisted of replicates and lack of fit points. Experimental data was analyzed by multiple regression and used to develop predictive models for optimization within the design space.

4. Results and Discussion

4.1 ATH, MDH, Zinc Molybdate

To examine the potential synergies of ATH, MDH and zinc molybdate, an experimental design was established. The total filler content was fixed at 60 phr. The ATH level was not constrained, i.e. could vary from 0 to 60 phr. The MDH level was restricted to 50 phr and below while the Kemgard 911C level was restricted to 15 phr and below. The constraint on Kemgard level was based on cost considerations. The complete experimental design is shown in Table 2.

Table 2. First experimental design

Run #	Micral 9400 ATH	Vertex 90 MDH	Kemgard 911C
1	10.0	35.0	15.0
2	27.5	17.5	15.0
3	60.0	0.0	0.0
4	45.0	0.0	15.0
5	10.0	50.0	0.0
6	20.6	28.1	11.3
7	41.9	10.6	7.5
8	60.0	0.0	0.0
9	52.5	0.0	7.5
10	35.0	25.0	0.0
11	10.0	50.0	0.0
12	35.0	25.0	0.0
13	20.6	35.6	3.8
14	10.0	42.5	7.5
15	10.0	35.0	15.0
16	45.0	0.0	15.0

The 90 second, 4 minute and maximum smoke density developed in the NBS Smoke chamber (ASTH E662) are shown in the Table 3. The samples are ranked from lowest to highest based on the 4 minute smoke

Table 3. NBS Smoke results – Zinc Molybdate System

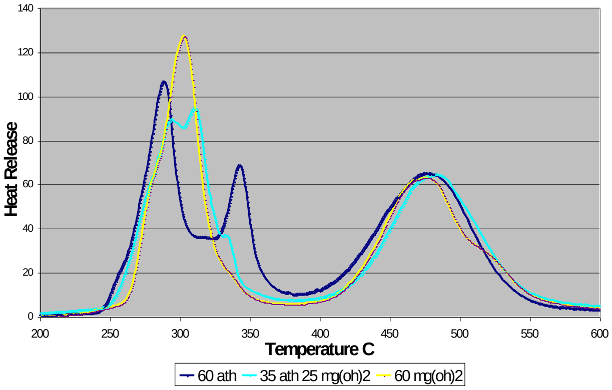
Run #	Micral 9400 (ATH)	Vertex 90 ($Mg(OH)_2$)	KG 911C	D 90	D 4.0	D max
4	45.0	0.0	**15.0**	**16**	**95**	**276**
6	20.6	28.1	**11.3**	**10**	**96**	**371**
16	45.0	0.0	**15.0**	**19**	**115**	**277**
2	27.5	17.5	**15.0**	**9**	**120**	**329**
7	41.9	10.6	**7.5**	**18**	**121**	**365**
15	10.0	35.0	**15.0**	**10**	**140**	**327**
9	52.5	0.0	**7.5**	**29**	**149**	**311**
14	10.0	42.5	**7.5**	**13**	**155**	**372**
1	10.0	35.0	**15.0**	**11**	**156**	**320**
13	20.6	35.6	**3.8**	**16**	**157**	**375**
10	35.0	25.0	**0.0**	**15**	**170**	**429**
11	10.0	50.0	**0.0**	**15**	**175**	**383**
12	35.0	25.0	**0.0**	**16**	**181**	**394**
5	10.0	50.0	**0.0**	**15**	**188**	**396**
8	60.0	0.0	**0.0**	**20**	**234**	**473**
3	60.0	0.0	**0.0**	**14**	**245**	**540**

Examination of the data clearly shows that the lowest 4 minute smoke values were obtained in the systems containing zinc molybdate. The best system overall was the binary ATH/KG 911C system (45phr/15phr). In the presence of 15 phr Kemgard 911C, replacement of ATH with MDH results in an increase in smoke (compare systems 1 and 4). Rather than a synergy, this may in fact represent an antagonistic behavior between MDH and zinc molybdate.

In the absence of zinc molybdate, there does appear to be a benefit to replacing some ATH with MDH. This synergy between the two metal hydroxides is consistent with the results of Chen and Isarov.[5]

To better understand the apparent metal hydroxide/zinc molybdate interactions, several systems from the DOE were examined using PCFC which determines heat release rate as a function of pyrolysis temperature. Figure 3 shows a representative curve obtained with Sample 8 (60 phr ATH). The system exhibits several peaks. The first two peaks with maxima at 290^0 C and 345^o are likely the result of several processes including pyrolysis of plasticizer as well as the initial pyrolysis of the PVC resin. In this regime char formation by dehydrochlorination occurs. The third peak, at approximately 480^o C corresponds to the thermal decomposition of the char layer.

Figure 3. PCFC of Flexible PVC with ATH and MDH



The second curve in Figure 3 shows the results obtained with a sample containing 60 phr MDH (no ATH). With MDH as the only filler, the first pyrolysis peak is shifted to higher temperature by roughly 10 C. The second peak has also disappeared, clearly indicating a change in the mechanism of char formation.

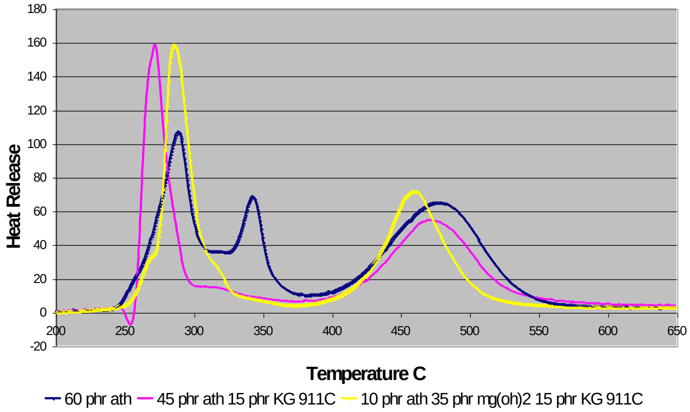
The shift in onset temperature for char formation can be explained using data published by Chen and Isarov.[6] Using high resolution, it was shown that ATH is completely converted to Al_2O_3 at temperatures well below that of PVC decomposition.[6] In contrast, MDH decomposition occurs at temperatures higher than that of PVC dehydrochlorination. Because MDH is highly basic, it is likely that it reacts with HCl, thereby interfering with the autocatalytic dehydrochlorination process and influencing char formation.

$$Mg(OH)_2 + 2\,HCl \rightarrow MgCl_2 + 2\,H_2O \qquad (1)$$

The third curve in Figure 3 represents the system containing 35 phr ATH and 25 phr MDH. Not surprisingly, the first main peak is separated into two overlapping peaks. However, the second peak that was so prominent in the ATH system is only a shoulder in the curve in the ATH/MDH system. Also the total heat (the integrated area under the curve is lower in the blended system than in either of the systems containing the pure metal hydroxides.

PCFC data can also be used to gain insight into the metal hydroxide/zinc molybdate interactions. The first curve in Figure 4 again shows the results form Sample 8 (60 phr ATH). The second curve shows the results obtained with Sample 4 which contains 45 phr ATH and 15 phr Kemgard 911C. Here the initial dehydrochlorination peak is shifted by approximately 15 C to lower temperature. Since many zinc containing compounds are known to promote char formation and reduce thermal stability, this shift in onset temperature is expected. Like MDH, the addition of zinc molybdate also results in the disappearance of the second dehydrochlorination peak.

Figure 4. PCFC of Flexible PVC with ATH, KG 911C, MDH



The peak occurring at around 480 C is associated with the thermal decomposition of of the PVC char layer. In the presence of Kemgard 911C, the area of the curve is lower suggesting that the char layer is more robust and thermally stable than in the pure ATH system.

The third curve in Figure 3 represents the system containing 15 phr Kemgard 911C with most of the metal hydroxide being MDH. Replacement of ATH with MDH again shifts the first peak to higher temperature, essentially negating the promotion of char formation by the zinc molybdate. The peak temperature associated with the thermal decomposition of the char is also shifted to lower temperature, indicating a less robust char formation. Both factors would be consistent with the apparent antagonistic effect seen with MDH and the zinc molybdate containing Kemgard 911C.

An interesting consequence of the antagonistic effect between zinc molybdate and MDH and the synergy between ATH and MDH is that the optimum ATH/MDH ratio appears to depend on the use level of zinc molybdate. Using regression analysis of the data in Table 3, a model was generated that allowed optimization within determined constraints. Using this model, the optimum ATH/MDH levels were predicted at levels of Kemgard 911C ranging from 1 phr to 15 phr. The optimum composition was one that minimized the four minute smoke (highest priority), maximum smoke density (second priority) and the 90 second smoke (third priority).

Table 4 shows the predicted optimized formulas as a function of Kemgard 911C use level. For each system identified, the predicted smoke densities are also presented.

Table 4. Optimized Compound Composition for KG 911C, ATH, and MDH and Calculated Response

IN ORDER BY: 4:00, Max, 90			Optimized Composition				Calculated Response		
KG Set Amt	Desirability		ATH	Mg(OH)2	Kemgard 911C		90 AVG	4:00 AVG	MAX AVG
15	0.80		45.00	0.00	15.00		17.34	106.79	299.16
14	0.79		46.00	0.00	14.00		17.78	108.00	302.34
13	0.77		47.00	0.00	13.00		18.26	109.86	306.51
12	0.76		46.91	1.09	12.00		18.32	112.64	311.26
11	0.74		43.73	5.27	11.00		17.24	116.04	314.81
10	0.72		41.03	8.97	10.00		16.48	119.08	318.24
9	0.70		38.66	12.34	9.00		15.93	121.88	321.57
8	0.69		36.61	15.39	8.00		15.53	124.50	324.85
7	0.68		34.73	18.27	7.00		15.21	127.02	328.05
6	0.66		33.03	20.98	6.00		14.97	129.47	331.22
5	0.65		31.49	23.51	5.00		14.79	131.88	334.36
4	0.64		30.01	25.99	4.00		14.64	134.28	337.49
3	0.62		28.52	28.48	3.00		14.51	136.68	340.59
2	0.61		27.46	30.54	2.00		14.45	139.07	343.72
1	0.60		26.26	32.74	1.00		14.38	141.50	346.82

At the highest Kemgard use level (15 phr), the best metal hydroxide system is predicted to be all ATH. However, as the Kemgard level is reduced, the optimum systems contain increasing levels of MDH. At the lowest Kemgard concentration (1 phr), the optimum metal hydroxide system is actually one with more MDH than ATH.

In practice this work suggests that in order to minimize smoke, one would use the maximum amount of zinc molybdate as economically feasible. To lower compound cost while minimizing the loss of smoke performance, one should consider increasing levels of MDH as the zinc molybdate is reduced.

4.2 ATH, MDH, Calcium Molybdate

To examine the interactions of ATH, MDH and calcium molybdate, the same experimental design was established using Kemgard 501. Again the total filler content was fixed at 60 phr. The constraints on ATH, MDH and Kemgard 501 levels were the same as those described previously.

The 90 second, 4 minute and maximum smoke density developed in the NBS Smoke chamber (ASTH E662) are shown in the Table 5 below. The samples are ranked from lowest to highest based on the 4 minute smoke.

Table 5. NBS Smoke results – Calcium Molybdate System

Run #	Micral 9400 (ATH)	Vertex 90 $(Mg(OH)_2)$	KG 501	D 90	D 4.0	D max
6	20.6	28.1	**11.3**	**11**	**132**	**309**
13	20.6	35.6	**3.8**	**14**	**150**	**477**
2	27.5	17.5	**15.0**	**19**	**151**	**289**
11	10.0	50.0	**0.0**	**19**	**187**	**374**
1	10.0	35.0	**15.0**	**19**	**200**	**410**
15	10.0	35.0	**15.0**	**21**	**211**	**372**
3	60.0	0.0	**0.0**	**32**	**213**	**428**
5	10.0	50.0	**0.0**	**18**	**215**	**396**
10	35.0	25.0	**0.0**	**21**	**218**	**377**
7	41.9	10.6	**7.5**	**23**	**223**	**389**
12	35.0	25.0	**0.0**	**34**	**232**	**335**
14	10.0	42.5	**7.5**	**23**	**239**	**434**
4	45.0	0.0	**15.0**	**33**	**261**	**395**
8	60.0	0.0	**0.0**	**32**	**295**	**466**
16	45.0	0.0	**15.0**	**34**	**298**	**470**
9	52.5	0.0	**7.5**	**33**	**321**	**499**

Unlike the zinc molybdate system, trends in the data are not so obvious. In fact, comparing the system with 60 phr ATH (Sample 8) and one with 45 phr ATH and 15 phr Kemgard 501 (Sample 16), there is very little difference in smoke numbers. The synergy between ATH and MDH, discussed in the previous section, is still apparent (compare samples 8 and 12 in the Table). However, the best performing systems were ones that contained all three additives, ATH, MDH and Kemgard 501. (Samples 2 and 6).

PCFC data was obtained on selected samples to gain insight into the effects of the various additives. In many cases the shapes of the curves were very similar. The only distinguishing data was the total heat release which tended to be lower in the better performing systems (see Table 6).

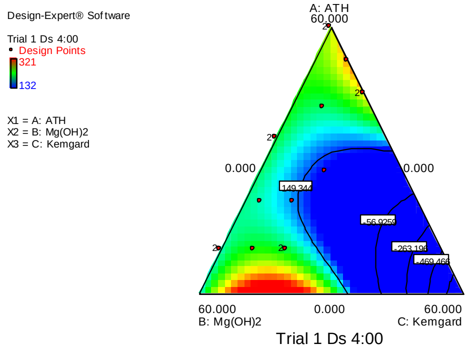
Table 6. Selected Results from DOE

Run #	**8**	**16**		**12**	**2**	**6**
Micral 9400 (ATH)	60	45		35	27.5	20.63
Vertex 90 $(Mg(OH)_2)$	0	0		25	17.5	28.13
KG 501	0	15		0	15	11.25
NBS Smoke						
D 90	**32**	**34**		**34**	**19**	**11**
D 4.0	**295**	**298**		**232**	**151**	**132**
D max	**466**	**470**		**335**	**289**	**309**

Microcalorimeter						
HRC	**236**	**203**		**167**	**177**	**162**
Total heat	**10.5**	**9.9**		**9.9**	**9.2**	**9.1**

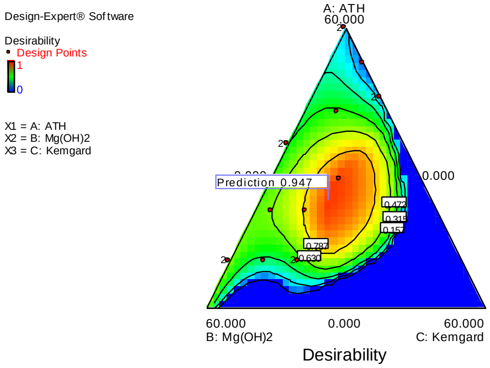
Statistical analysis of the ATH, MDH, Kemgard 501 system is best depicted graphically. Figure 5 shows the response space for the four minute smoke data. Within the established constraints the best performance is expected in the rang of 10 – 15 phr Kemgard 501, with the ATH and MDH at approximately equel levels. Although the analysis also suggests the best performance at very high Kemgard 501 levels, this space was unexplored in our design and is considered to be not commercially viable from a cost perspective.

Figure 5. Predicted Response for Four inute Smoke



Analysis of the date to include the 90 second, four minute and maximum smoke density (all weighted equally) gives a slightly different picture (Figure 6). Here the optimum composition is centered around a composition of 15 phr Kemgard 501 with the balance of the filler split between ATH and MDH.

Figure 6. Predicted Response for All NBS Criteria



5. Conclusions

To meet the stringent smoke requirements of NFPA 262, PVC plenum jacket compound requires the use of various additives. Typically these will include metal hydroxides such as ATH and MDH as well as a molybdate based smoke suppressant. Metal hydroxides decompose endothermically at temperatures ranging from 200 C to 400 C, thereby absorbing the heat of combustion. The water liberated may also have a diluting effect in the gas phase, displacing oxygen above the condensed phase. In contrast molybdate based additives function in the condensed phase by enhancing char formation.

In commercial practice there is anecdotal evidence of synergies between metal hydroxides and molybdates. However, exact formulation compositions are rarely disclosed and are considered to be trade secrets. In this work we attempted to examine the interactions between metal hydrates (both ATH and MDH) and metal molybdates using a statistally designed set of experiments. PCFC data was also collected to gain scientific insight into raw material interactions.

In this particular plasticized PVC system dramatically different results were observed with zinc and calcium molybdate, In the case of zinc molybdate, the best performance was achieved using ATH as the sole metal hydroxide. MDH appears retard the zinc molybdate promote char formation. This results in an apparent

antagonistic behavior between MDH and zinc molybdate. At lower levels of zinc molybdate, replacement of some ATH with MDH does lower smoke

In the case of calcium molybdate system, the optimum smoke performance is achieved at a composition is centered around 15 phr Kemgard 501 with the balance of the filler split between ATH and MDH.

The results obtained may be specific for the plasticizer combination used in this study. Phosphate ester plasticizers are believed to lower smoke by enhancing char formation so it is very likely that the optimum metal hydrate/molybdate composition will vary with other plasticizers. The use of Design of Experiments may be the most expedient way of optimizing performance. However, thermal techniques may provide valuable mechanistic information as well.

6. Acknowledgments

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8. Authors

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