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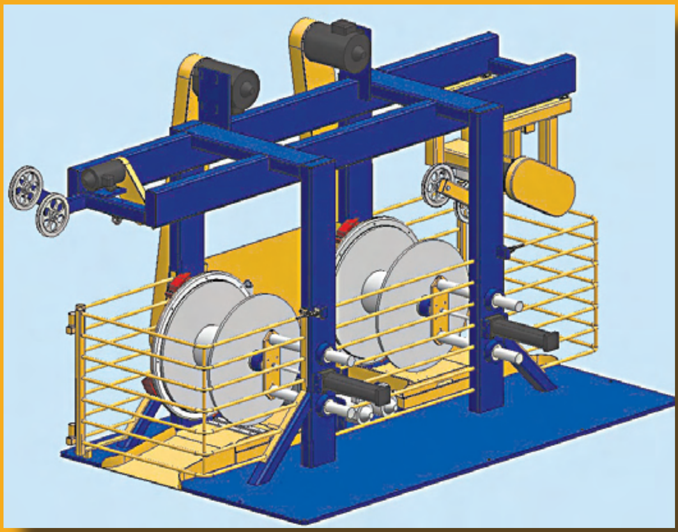
TOP PRODUCTS OF 2007...P. 38 & P. 90

Cables 2008 p. 84

Wire Processing, P. 85



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Inside this issue...

- News & Info
- Top Products of 2007
- Just Load, Click and Print
- Failed Bridge Cables Replaced with Smaller, Stronger Ones
- Superior Flame Resistance Cable
- On-Line Cable Design Tool
- New Products

Load, Click & Print, P. 92...

Page 90...WCC's Top Products of 2007

*Straight From The Shows...Pages 50 & 66*

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*P. 12 & P. 61*

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# New Magnesium Hydroxides Enabling Low-Smoke Cable Compounds

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With continuously rising industry-wide interest to favor the low-smoke and/or zero-halogen cable compositions, wire and cable producers are paying increased attention to the need for having easy access to certain types of fire retardant chemicals that are considered as environmentally friendly. The metal hydroxide family, which primarily consists of alumina trihydrate (ATH) and magnesium dihydroxide (MDH), has been among those groups of desirable fire retardants due to their chemical nature of being nontoxic and noncorrosive both during their use and throughout cable service life. Unlike other common fire retardants that are based on bromine or phosphorus, the metal hydroxides would provide fire performance via both flame (heat) reduction and smoke suppression, i.e., a dual-functionality FR additive.

One well-know performance trade-off of using the metal hydroxide for formulating flame retardant cable compounds is the requirement for high loading levels of the hydroxide, typically ranging from 40% to 70% by weight in order to satisfy the fire performance. The high hydroxide loading often results in reduced material mechanical properties as well as less favored compounding or extrusion efficiency due to increased processing viscosities. Such disadvantages associated with the metal hydroxides have from time to time outweighed the benefit of producing environmentally friendly cables especially when cable designs are geared toward lighter and more compact constructions while higher cable production throughput becoming more of an expectation than a desire.

From a technical standpoint, ATH can be used in cable compounds that are made of certain thermoplastics such PVC and polyolefin and elastomers. Until today, a wide variety of fire-retardant cable compositions have been based on the ATH technology, with or without other fire-retardant co-additives. This historically high use of ATH is attributed in part to its wide availability and more importantly, to the lower cost of its use. Rising demands for better-performing FR wire and cable and for increased cable production rates have begun to show an inherent material deficiency of ATH, namely, its lower thermal stability of beginning to decompose at around 220°C, or even at lower temperature when under mechanical shear. The thermal stability of ATH can limit compound processing and thus cable extrusion throughput even though ATH remains stable and intact under typical processing conditions for those thermoplastic polymers.

In contrast to ATH, MDH offers a much higher thermal stability with its temperature of decomposition at around 340°C. This enables compounding and processing at higher temperatures than permitted for ATH, and thus can result in increased throughput as desired. Use of MDH for fire-retardant wire and cable has been much limited compared to ATH, largely due to the higher cost of typical premium grades of MDH, which is a result of limited supply. The benefit of using MDH versus ATH has prompted continued industrial research and development, and as a result gradual commercial introduction of novel MDH materials suited for a range of wire and cable applications. These new MDH fire retardants are characterized by a number of attractive performance attributes and features as listed below:

- Unique particle size and size distribution combination allowing for optimized balance of compounding process and compound mechanical performance.
- Enhanced flame/smoke performance as shown by reduced heat and smoke production measured by cone calorimetry.
- Improved MDH powder incorporation and dispersion into the polymer, enabled by chemical modification of MDH surface, resulting in enhanced overall compound property and performance.
- Added FR efficacy achieved via using functionalized surface treatments.
- Improved economics of using the novel MDH as compared to conventional premium grades.

**J. M. Huber Corporation** has focused on developing these types of MDH products with continuing success in making available to FR cable compounders and cable producers a series of new MDH products with the above features.

Vertex® 100 and Vertex 90 product lines recently commercialized by Huber are examples of such new synthetically produced MDH fire retardants. This article will discuss the characteristics of Vertex MDH products and their performance benefits for use in formulating low-smoke and/or zero-halogen wire and cable compounds.

## Materials and Testing

Magnesium hydroxide materials were investigated in EVA and PVC formulations representative of commercial wire and cable compounds. A description of magnesium hydroxide materials used in this study is presented in **Table 1**.

The compounding was done on a **Brabender** lab mixer followed by sheeting out on a two-roll mill. The test specimens were compression molded. Unless otherwise noted, the EVA compounds described in this study were based on ethylene vinyl acetate co-polymer with 28% vinyl acetate content. Also unless otherwise noted, flexible PVC formulation used in this study contained 70 phr of metal hydroxide and 45 phr of plasticizer (TOTM). All material testing was performed according to following **ASTM** procedures per the following:

- *ASTM E1354*: Cone Calorimeter, 50 kW/m2.
- *ASTM E662*: NBS Smoke Chamber, flaming and nonflaming as indicated.
- *ASTM D2863*: Limiting Oxygen Index (LOI).
- *UL 94* vertical burn, 1/16" thickness.

Several aluminum hydroxide materials were also used in this study.

## Results and Discussion

*Effect of Particle Size Characteristics on Fire Retardancy of Magnesium Hydroxide in EVA.* Three grades of magnesium hydroxide (surface treated with vinylsilane) were used to

**Table 1. Magnesium hydroxide materials used in this study.**

| Material | Particle size, microns | Particle size distribution | Surface treatment |
|---|---|---|---|
| MDH-A | 5 | Broad | Vinylsilane |
| MDH-B | 3 | Intermediate | Vinylsilane |
| MDH-C | 1.5 | Uniform | Vinylsilane |
| Vertex 100 | 1.5 | Uniform | None |
| Vertex 100 SV | 1.5 | Uniform | Proprietary |
| Vertex 90 | 1.5 | Uniform | None |

Vertex is a registered trademark of J.M. Huber Corporation
MDH-A is a commercial MDH product
MDH-B and MDH-C are developmental MDH materials from J.M. Huber Corporation.

investigate the effect of particle size on FR performance (see **Table 1**) in EVA compound containing 64% MDH. Finer particle size clearly leads to noticeable reduction of the secondary peak of heat release (**Figure 1a**) and significant reduction of the secondary peak of smoke generation (**Figure 1b**).

The finer sized particles are generally characterized by higher surface area. It is the surface area of MDH that plays a very important role in suppressing both flame and smoke when the compound is ignited.

The formation of the secondary heat and smoke release peaks in cone calorimetry tests is believed to be associated with partial destruction of the insulation char formed when polymeric composition is burned, and is considered a negative effect as it contributes to increase of the total amount of smoke generated. If finer particle size magnesium hydroxide is used, a more effective char formation process takes place. At the moment when magnesium hydroxide is completely decomposed, a stronger char layer separates remaining polymer from oxygen and heat, and this reduces the formation of the secondary heat and smoke release peaks.

Magnesium hydroxide particle size also affects the compounding process and physical properties of the compound (**Table 2**). Finer particle size leads to an increase in tensile strength, i.e., making MDH more reinforcing.

A negative effect associated with MDH particle size going finer is increased compounding viscosity, which may lead to processing limitations. Appropriate surface treatment, selected specifically for particular polymer system and/or application, can be very effective in reduction of compounding viscosity and thus an increase in FR loading levels.

*Effect of Surface Treatment on Flame Retardancy of Magnesium Hydroxide Filled EVA.* Although particle size reduction is an effective way of improving FR efficiency of magnesium hydroxide, it has some limitations (production cost increase) and disadvantages (e.g., increase of compounding torque—**Table 2**).

Polymer formulators use several different approaches to achieve better FR performance of magnesium hydroxide filled compounds (besides particle size reduction). For example, zinc borate can be used as synergistic additive leading to improved overall FR performance.[1]

Surface treatment of magnesium hydroxide is often used to improve the compound's FR performance. Surface treatment helps to achieve better dispersion of metal hydroxide in the polymer indirectly affecting FR performance. Surface

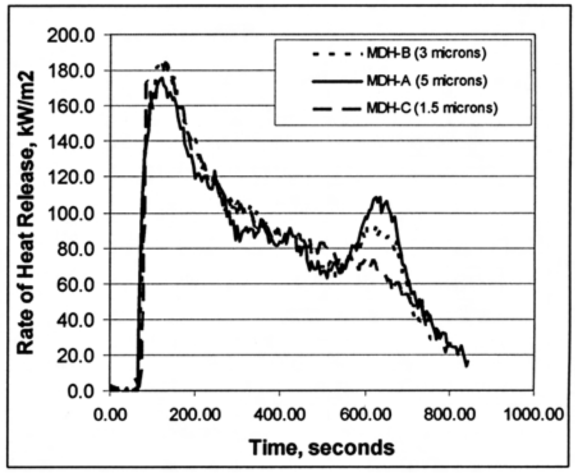


**Fig. 1a — Cone calorimetry results: effect of particle size on rate of heat release (EVA system, ~ 64% of magneseum hydroxide).**

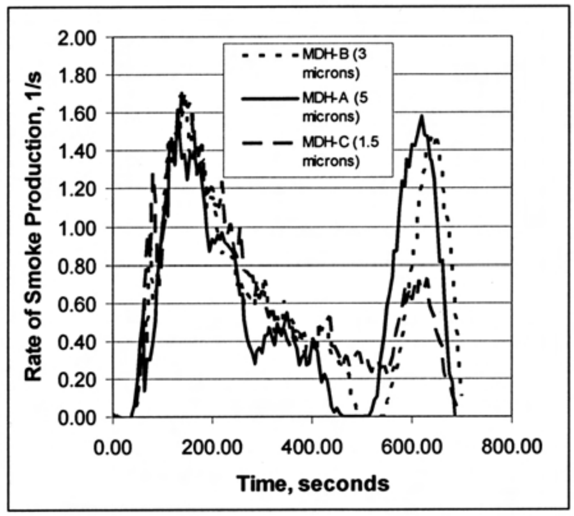


**Fig. 1b — Cone calorimetry results: effect of particle size on rate of smoke production (EVA system, ~ 64% of magneseum hydroxide).**

**Table 2. Effect of MDH particle characteristics on compounding performance and tensile properties (EVA system, ~ 64% of magnesium hydroxide).**

| Description | MDH-A | MDH-B | MDH-C |
|---|---|---|---|
| Particle size, microns | 5 | 3 | 1.5 |
| | | | |
| Compounding Torque (m g) | 7193 | 7841 | 8226 |
| | | | |
| Tensile strength (psi) | 1023 | 1090 | 1234 |
| Elongation (%) | 84 | 99 | 90 |

treatment also reduces compounding torque thus enabling higher loading levels.

J.M. Huber recently developed a new type of surface treated magnesium hydroxide with the surface treatment that directly enhances FR performance of MDH in EVA. The new grades of surface treated magnesium hydroxides are called Vertex 90 SV and Vertex 100 SV. **Figure 2a** and **Figure 2b** (on next page) show the effect of SV surface treatment on the rate of heat release and rate of smoke production.

**Continued...**

As shown, the new SV surface treatment (Vertex 100 SV) significantly reduces the rate of heat release including peak rate of heat release. Simultaneously, it reduces the rate of smoke generation.

As mentioned above, the formation of secondary heat and smoke release peaks is considered a negative effect. The SV surface treatment leads to extended burn time (secondary peak postponed) associated with dramatic reduction and delays of both secondary heat and smoke peaks.

The smoke reduction capacity of the SV surface treatment is confirmed by an almost 50% reduction of average specific extinction area (SEA), also measured by cone calorimeter (**Table 3**). Additional benefit of Vertex 90 SV and Vertex 100 SV products in EVA formulations is a significant increase of limiting oxygen index, LOI (**Table 3**).

Significantly enhanced FR performance of surface treated Vertex 100 SV compared to untreated Vertex 100 magnesium hydroxide creates an opportunity for reducing FR loading in EVA formulation.

**Figure 3** illustrates the effect of loading level reduction of Vertex 100 SV on smoke suppression efficiency measured by specific extinction area (SEA). One can see that formulation containing 60% of Vertex 100 SV shows smoke generation capacity similar to that containing 64% of untreated Vertex 100 (within experimental error).

**Table 4** illustrates the benefits of SV surface treatment on oxygen index of the MDH filled EVA compounds as well as on compounding rheological improvement. As shown, an EVA compound containing 56% by weight of Vertex 100 SV provides a two-unit increase in LOI and 38% reduction of compounding viscosity as compared to the EVA compound containing 64% of untreated MDH Vertex 100.

A concerted review of overall flame and smoke performance (**Figure 2a**, **Figure 2b** and **Figure 3**) and compounding rheological improvement (**Table 4**) clearly demonstrates a valuable benefit of the SV-treated Vertex MDH as a formulation tool to help achieve maximized compounding/extruding rates without trade-off in the FR performance.

Another benefit of the SV-treated Vertex MDH, a direct result of an ability to lower MDH loading level without compromising FR performance, is a significant boost to compounds' mechanical properties, as shown in **Table 5**. Such improvements in low-temperature brittleness and material ductility provide cable manufacturers with the opportunity of enhancing cable performance and cable downsizing.

*Comparison of ATH and MDH for Flexible PVC Smoke Suppression.* Some fire-retardant PVC compounds contain ATH for low-smoke performance. Like ATH, MDH is known as an effective smoke suppressant and is widely used in low-smoke polymer applications. A study was done to compare ATH with MDH on their respective effect on smoke suppression for a 70-phr metal hydroxide loaded PVC, and the results are in **Table 6**. It is clear that MDH outperforms ATH in more effectively reducing the compound smoke development when burned, as tested under nonflaming and flaming conditions. Vertex 100 MDH is a new product recently launched by J. M. Huber that has been engineered to provide the enhanced flame and smoke performance over common technical or even some premium grades of commercial magnesium hydroxide. This improved smoke performance by Vertex 100 is attributed to its unique particle characteristics. Such added performance benefits with Vertex 100 have also been successfully demonstrated in the polyolefin based flame retardant compound applications.

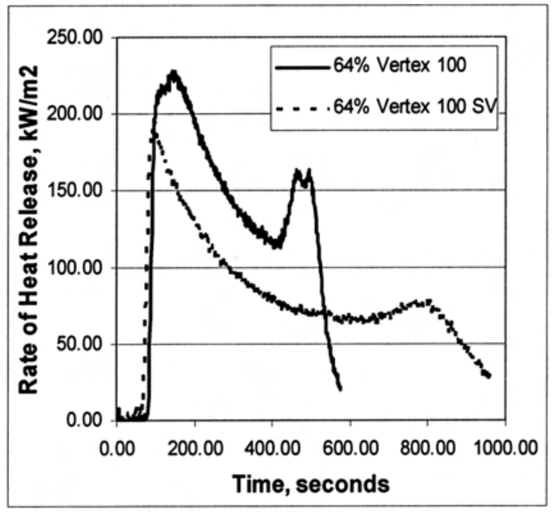


**Fig. 2a — Cone calorimetry results: effect of surface treatment on rate of heat release (EVA system, ~ 64% of magnesium hydroxide).**

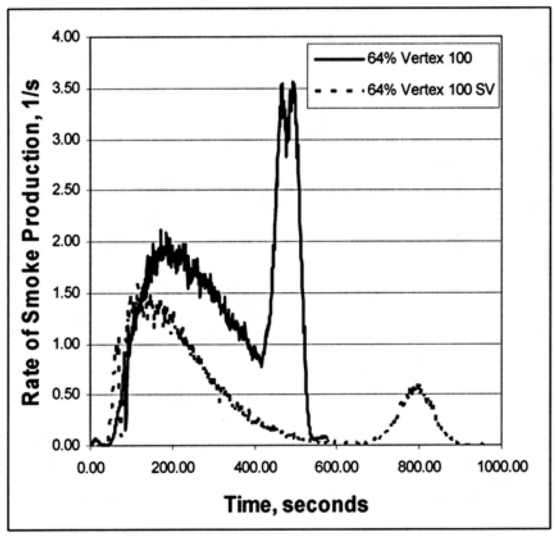


**Fig. 2b — Cone calorimetry results: effect of surface treatment on rate of smoke production (EVA system, ~ 64% of magnesium hydroxide).**

**Table 3. Effect of surface treatment on fire performance EVA-based compound containing magnesium hydroxide.**

| Magnesium Hydroxide | Average SEA, m2/kg | LOI, % |
|---|---|---|
| Vertex 100 (64%) | 240.1 | 38 |
| Vertex 100SV (64%) | 128.1 | 44 |

MDH also outperforms ATH in its ability to reduce total heat released (THR) during burning (**Table 7** and **Figure 5** on last page of this article). Compound containing Vertex 90 magnesium hydroxide shows significantly lower THR compared to compound containing untreated ground ATH Micral® 932 (with a median particle size of 2.5 microns). **Figure 4a** and **Figure 4b** show cone calorimetry results for compounds containing Vertex 90 and Micral 932. It is clear from the cone calorimetry data that magnesium hydroxide significantly shortens burn time thus reducing the amount of heat released.

The flexible PVC formulation used in this study contains 70 phr or approximately 22 wt. % of metal hydroxide. At this relatively low loading level, MDH significantly outperforms

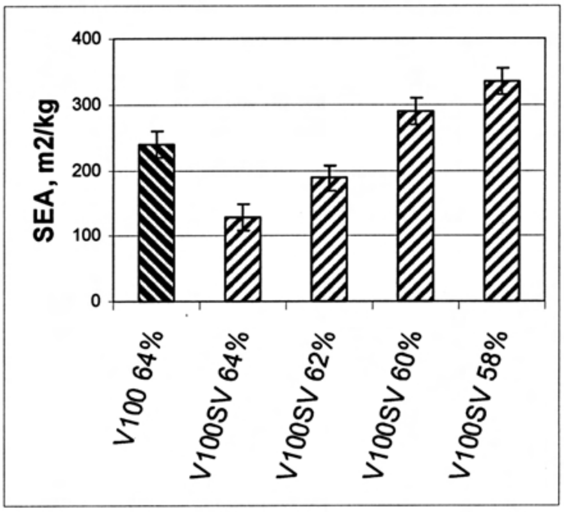


**Fig. 3 — Cone calorimetry results: effect of magnesium hydroxide loading level on rate of smoke production (EVA system, ~ 64% of MDH).**

**Table 4. Effect of loading level on LOI, compounding torque and mechanical properties of Vertex 100 SV in EVA-based formulation.**

| MDH type | MDH % | LOI % | Torque m g | Compounding viscosity reduction |
|---|---|---|---|---|
| Vertex 100 | 64 | 38 | 9515 | - |
| Vertex 100SV | 64 | 44 | 7293 | - 23% |
| Vertex 100SV | 60 | 41 | 6481 | - 32% |
| Vertex 100SV | 56 | 40 | 5859 | - 38% |

**Table 5. Effect of surface treatment on tensile properties of EVA-based compound containing magnesium hydroxide.**

| MDH | LTB | Tensile Strength psi | Elongation % |
|---|---|---|---|
| Vertex 100 64% | - 11º C | 1581 | 100 |
| Vertex 100SV 64% | - 10.5º C | 1105 | 134 |
| Vertex 100SV 60% | - 16.5º C | 1183 | 322 |
| Vertex 100SV 56% | - 21.0º C | 1372 | 444 |

ATH in terms of heat and smoke release suppression. One can expect that FR-related benefits of MDH over ATH will be even more pronounced at higher loading levels.

Both Vertex 90 and Vertex 100 MDH products are expected to perform well in flexible PVC formulations. These two products have similar particle characteristics (median particle size of 1.5 microns and uniform particle size distribution) and are available with a variety of surface treatments. As mentioned above, surface treatments may benefit polymer processing and compound's physical properties. In the case of flexible PVC, surface treatments can also improve compound color.

**Continued...**

**Table 6. Smoke properties comparison of ATH and MDH (70-phr of untreated metal hydroxide in f-PVC).**

| Description (in phr) | ATH (OE805) | MDH (Vertex 100) |
|---|---|---|
| PVC (K70) | 100 | 100 |
| Plasticizer (TOTM) | 45 | 45 |
| $CaCO_3$ | 20 | 20 |
| Stabilizer | 7 | 7 |
| Other FR synergists | 4 | 4 |
| Lubricants | 5 | 5 |
| | | |
| **NBS smoke density (Flaming)** | | |
| Ds @ 4 min. | 140.67 | 49.33 |
| Dmax | 632.00 | 469.67 |
| **NBS smoke density (Non-Flaming)** | | |
| Ds @ 4 min. | 52 | 27.33 |
| Dmax | 399.67 | 271.67 |

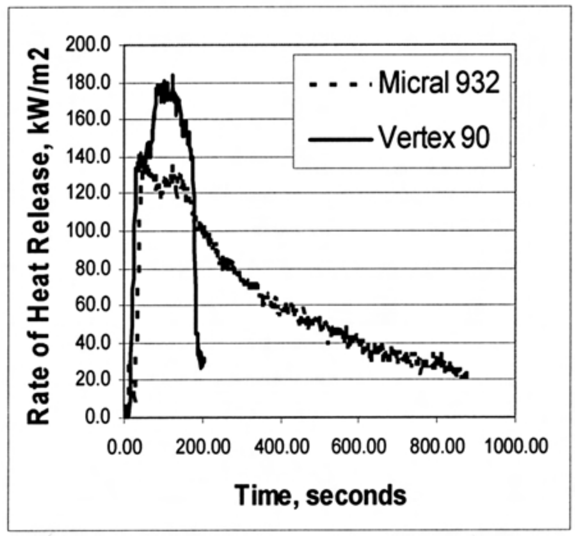


**Fig. 4a — Cone calorimetry results: effect of metal hydroxide type on rate of heat release (flexible PVC system, ~ 70 phr of metal hydroxide).**

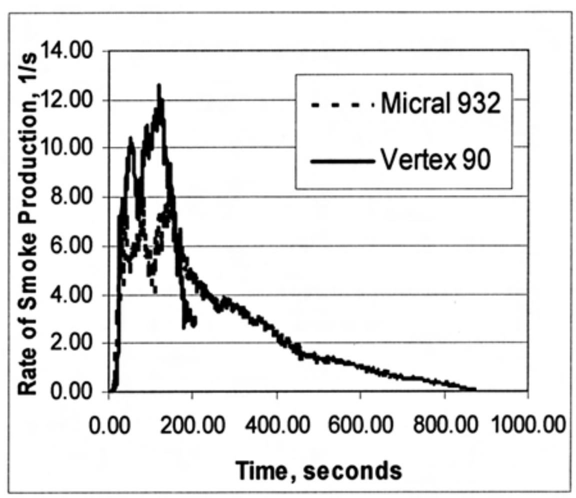


**Fig. 4b — Cone calorimetry results: effect of metal hydroxide type on rate of smoke production (flexible PVC system, ~ 70 phr of metal hydroxide).**

Table 7. FR properties of f-PVC compounds containing ATH.

| Property | Micral 932 | Vertex 90 |
|---|---|---|
| | | |
| LOI (%) | 37-38 | 36-37 |
| | | |
| UL-94 Vertical (1/16) | V-0 | V-0 |
| | | |
| Time to Sustained Ignition | 43.17 | 31.25 |
| Peak Rate of Heat Release | 138.35 | 182.7 |
| Time of Peak RHR | 106.5 | 101 |
| Total Heat Released | 55.95 | 21.8 |
| CO Yield | 0.1946 | 0.1344 |
| CO2 Yield | 1.0538 | 0.8006 |

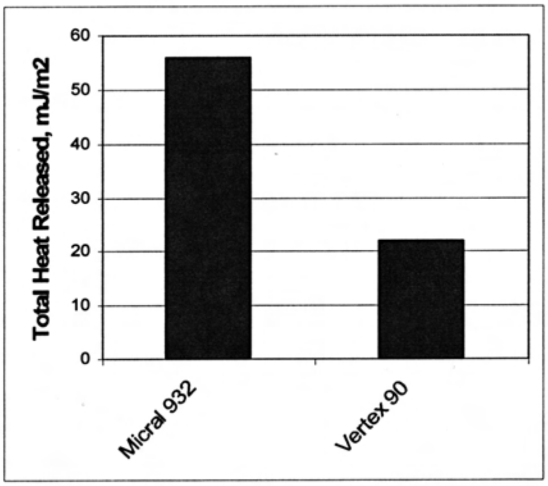


**Fig. 5 — Effect on metal hydroxide type on total heat released measured by cone calorimeter (flexible PVC system, ~ 70 phr of metal hydroxide).**

*Synergistic Effect of Metal Hydroxides With Calcium Carbonate.* An effort was also made to investigate whether the use of a combination of ATH and MDH would add a synergistic fire performance compared with using either ATH or MDH for a flexible PVC system. The results are given in **Table 8**.

For this particular compound composition, comparison of Samples 8-2 and 8-4 appears to show that Vertex 60 MDH under-performed Micral 9400 ATH in the smoke (measured by average specific extinction area) and inflammability (measured by LOI), although both yielding V0 ratings. Further, there appears to be a lack of fire performance synergies for a compound containing both ATH and MDH (Sample 8-3).

## Conclusions

Magnesium hydroxide particle size and particle size distribution have significant effect on FR efficiency in EVA. Finer particle size leads to significant reduction of secondary heat and smoke released peaks measured by cone calorimetry.

New SV surface treatments for MDH such as Vertex 90SV improve FR performance of MDH filled-EVA. By using Vertex 90SV or Vertex 100 SV, MDH loading level can be significantly reduced leading to better processing and improved physical properties of the compound.

Table 8. Smoke properties of f-PVC compounds containing untreated ATH or MDH. Effect of CaCO3, ATH vs. MDH combination.

| Description (ingredients in phr) | 10-1 | 10-2 | 10-3 | 10-4 |
|---|---|---|---|---|
| PVC (K70) | 100 | 100 | 100 | 100 |
| Plasticizer (TOTM) | 45 | 45 | 45 | 45 |
| Stabilizer | 7 | 7 | 7 | 7 |
| Lubricants | 5 | 5 | 5 | 5 |
| CaCO3 | 40 | 25 | 25 | 25 |
| FR synergist | 5 | 5 | 5 | 5 |
| AOM | 10 | 10 | 10 | 10 |
| Antimony oxide | 3 | 3 | 3 | 3 |
| ATH (Micral 9400) | 45 | 60 | 30 | - |
| MDH (Vertex 60) | - | - | 30 | 60 |
| | | | | |
| UL 94 Rating, 1/16" thickness | V-0 | V-0 | V-0 | V-0 |
| LOI, % O2 | 50 | 50 | 47 | 45 |
| Ave. SEA, m²/kg | 421.8 | 372.5 | 467.7 | 545.9 |

MDH significantly outperforms ATH as heat and smoke suppressant in flexible PVC formulations. At the same time, ATH-based flexible PVC formulations have higher LOI values. No FR performance synergy was found between ATH and MDH in flexible PVC formulations. WCTI

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***Reference:***
[1] *Shen, K.K.; Olson, E.; Amigouet, P.; Chen, T.; Recent advances on the use of metal hydroxide and borates as fire retardants in halogen-free polyolefins. Proceedings of Seventeenth Annual BCC Conference on Flame Retardancy, Stamford, Connecticut, May 2006.*

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***Company Profiles...***

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