Intumescent Coatings – Truly Functional Coatings That Save Lives – Latest Developments

Christophe Baude, Maurille Sécher, Cécile Fonteneau, Alan Fream
OMNOVA Solutions, Performance Chemicals

In recent years, thin film intumescent coatings have become the preferred choice for the protection of structural steel and have enjoyed significant growth on a global basis. They provide cost-effective passive fire protection to structural steel, while maintaining the aesthetic qualities of the steel, a quality being demanded by more and more architects and engineers.

However, due to the ever increasing complexity of building design, the sophistication of the design and application of intumescent coatings has also increased.

This paper gives an overview of the current state of the art in the thin film intumescent coatings market and of progress that has been made particularly in the water-based sector, in terms of improvement of the main weakness of waterborne intumescents, namely that of water sensitivity.

Introduction[1,2]

Fires in high rise buildings, like the one in The Address Downtown Hotel tower in Dubai on December 31, 2015, are a constant reminder that fire safety in new construction is of paramount importance.

The fire, which broke out in the 63-storey luxury hotel less than three hours before Dubai's New Year's Eve fireworks spectacular, was just the latest in a string of building blazes in Dubai last year. The fire could not have happened at a worse time, viewed by millions on TV and causing a public relations disaster for the city. Although more than 2,000 people were affected by the blaze, fortunately there were no deaths or serious injuries. Nonetheless, the fire adds to the approximately one percent of world GDP that literally goes up in smoke every year as a result of devastating fires.

While steel itself does not burn, it loses its strength and load-bearing capacity when exposed to temperatures above 500 °C. Thus, in a steel-framed structure, such as a multi-storey building, fire protection is a necessity to protect human lives during the unfortunate event of a fire. Today most buildings and structures have some degree of fire protection because, in most countries, fire protection is a requirement of National Building Codes. The amount of protection required is usually related to the height or number of storeys of the building and the density of occupancy.
Considerable progress has been made in recent years in improvements in efficiency of intumescent coatings for protection of structural steel. However, as far as water-based intumescent coatings are concerned, little progress has been made in reducing water sensitivity, their “Achilles heel”. Consequently waterborne products are limited to service in interior conditions.

The poor water resistance of these coatings is primarily related to the water sensitivity of the binders used to formulate them, which is in turn related to their chemistry.

The development of a new polymer, specifically with the aim of producing a binder for use in the formulation of waterborne intumescent coatings that can achieve up to 2 hours fire resistance, but with significantly improved water resistance, which will allow the coatings to be used in semi-exposed and exposed environmental conditions, is the subject of this paper.

Intumescent coatings: What are they? How do they work? [3,4,5,6]

Intumescent coatings react under the influence of fire and swell in a controlled manner to many times their original thickness, producing an insulating carbonaceous char or foam that protects the substrate from the effects of the fire.

Intumescent coatings, unlike other forms of passive fire protection (PFP) such as boards and sprayed cementitious systems, can be used to provide a decorative and protective finish that does not detract from the original appearance of the exposed steelwork. This is clearly evident in new airports, shopping malls, hotels, sports stadiums, etc, where, with increasing frequency today, thanks to the unobtrusive nature of this form of PFP, intumescent coatings enable architects to fully exploit the creative design possibilities of the steel itself.

The basic technology of intumescent coatings has changed little for the past 40 years. Having said that, our knowledge of the chemistry has increased significantly, and the technology of the key ingredients has evolved, and continues to evolve, to keep pace with the needs of the market.

Intumescence is generally accomplished with a minimum of three components: a source of carbon (typically pentaerythritol or dipentaerythritol, PER or DIPER), a blowing agent (typically melamine, MEL, and solid chlorinated paraffin, CP), and a source of mineral acid catalyst (typically ammonium polyphosphate, APP). When an intumescent coating is subjected to heat, a series of chemical reactions occurs: the APP decomposes to produce phosphoric acid; the phosphoric acid causes dehydration of the PER or DIPER to produce a carbon char; the blowing agent decomposes, releasing non-flammable gases which cause the carbon char to foam, thus producing a meringue-like structure which is a highly effective insulator against heat.

The importance of the resin binder in intumescence [7,8,9]

There is no doubt about the importance of the binder in an intumescent coating. Indeed, in the CEPE Guidance to a quality control fire test regime for intumescent coatings, the probability of effect on fire protection is stated as “certain,” and the fire test level required if the binder is changed in a formulation is “5” (the highest).

The role of the binder has been studied in much depth in recent years. Besides providing the properties typically required for most coatings applications, it also must degrade over the correct temperature range with other ingredients in the formulation, so that the chemical reactions that produce intumescence can take place. Furthermore, it must have a melt viscosity that is not too low, which would cause the molten coating to slump during intumescence, nor too high, which would prevent the expansion of the foam. The molten polymer must have a certain viscoelasticity, comparable to that of bubblegum, to contain, within the molten matrix, gases released during degradation of the ingredients, thus producing the foamed char.

European Standards for fire protection coatings [10,11,12,13]

In Europe, the performance of an intumescent (or reactive coating as it is often referred) in terms of its resistance to a cellulosic fire is determined in accordance with CEN fire resistance test methods, which are currently EN 13381-6, EN 13381-8 and prEN 13381-9. (EN13381 = Test methods for determining the contribution to the fire resistance of structural members.) The fire stability of a structural steel element protected with an intumescent or reactive coating is rated in minutes.

The performance of fire protective reactive coating systems, under service conditions, must not deteriorate during their assumed intended working life so as to affect significantly the performance of the products, especially the protective effects in case of fire. Because the EN 13381 standards do not cover this aspect, several years ago European Technical Approval Guidelines (ETAGs) were established by EOTA (European Organisation for Technical Approvals) under the Construction Products Directive - 89/106/EEC - (CPD) upon mandates of the European Commission.

Under the current requirements of the Construction Products Regulation 2011 (CPR), which has replaced the CPD, reactive coatings for structural steel in cellulosic-fire situations are subject to a European Technical Assessment (ETA). That assessment forms the basis of the manufacturer’s voluntary CE product marking based on a “certificate of conformity” provided by a “notified certification body.” The certificate endorses ongoing compliance of the product with its ETA. ETAG018-2 gives guidance for the Attestation of Conformity of “Reactive Coatings for Fire Protection of Steel Elements”. The Construction Products Regulation in Europe indicates that products with CE marking coming from a harmonized EN standard are obligatory in Member States.

The New European Standard EN 16623-2015, intended for adoption by manufacturers on a voluntary basis, supports the existing mandatory process and reflects agreed-upon best practices. It covers the characterization of reactive coatings intended for the fire protection of structural steel and other ferrous metal substrates, in end-use conditions during cellulosic fires. Working groups within CEPE are now lobbying the EU commission and CEN to convert this voluntary standard into a mandatory harmonised hEN, in order to establish a level playing field for reactive coatings for cellulosic fire situations. A finalised version, however, may take several years. So, in the interim, CE marking for reactive coatings is today a quality assurance for users rather than a strict obligation.

One important element of EN 16623-2015 concerns ‘durability’. Categories related to exposure conditions within the standard, which is a refinement of those listed in ETAG018-2, are:

Type	Exposure Description
X	Intended for all conditions (internal, semi-exposed and exposed)
Y	Intended for internal and semi-exposed conditions. Semi-exposed includes temperatures below zero, but no exposure to rain and limited exposure to UV.
W/Y	Temporary full external for a maximum of 6 months, then semi-external.
W/Z1	Temporary full external for a maximum of 6 months, then internal with high humidity.
W/Z2	Temporary full external for a maximum of 6 months, then internal with controlled environment.
Z1	Intended for internal conditions (excluding temperatures below zero) with high humidity.
Z2	Intended for internal conditions (excluding temperatures below zero) with humidity classes other than Z1.

Table1: Use categories as defined in EN 16623

Waterborne intumescent coatings

Today, in the cellulosic fire segment of the market, both solvent- and waterborne intumescent products exist. Although solvent-borne intumescents are compliant with current European VOC legislation, market demand has been increasing for a high performance, durable, waterborne product, especially for on-site application, where solvent odour and VOC emissions can be concerns. However, waterborne intumescents have a number of well known weaknesses, not least of which is poor ‘durability’, related to their high sensitivity to humidity and water, which is a serious weakness that prevents them replacing solvent-borne coatings to a large extent. Therefore today, waterborne intumescents are limited to Z1 / Z2 environments.

Next-generation waterborne intumescent coatings must seek to improve upon the existing offerings, with a wider application window, faster drying in thick films which will increase the maximum application thickness per coat, better in-can storage stability (especially @ 5 °C), improved freeze-thaw resistance, and better overcoating performance (avoiding resolubilisation of the intumescent coating by waterborne topcoats).

Current binder technology for waterborne intumescent coatings

The water sensitivity of these coatings is primarily related to the water sensitivity of the binders used to formulate them: most commonly VA, VA/vinyl versatate (VV), or VA/E/VV or vinyl acrylic polymers, which are subject to hydrolysis, although providing the right characteristics in terms of stability with the commonly used intumescent ingredients and thermoplasticity / melt flow behaviour, essential for formation of stable insulating char.

The high water sensitivity of current waterborne intumescent coatings can be clearly demonstrated by a simple immersion test in water. After less than one half hour, the coating swells, softens and blisters (and also its intumescent performance is considerably reduced because of loss of water soluble intumescent ingredients). A solvent-borne intumescent on the other hand will resist more than 5 hours, with no blistering or loss of intumescent performance.

To the average person, this weakness may not appear to be important because many intumescent coatings are designed for service in dry, interior environments (Z1/Z2).

However, although a reactive coating system may be intended for internal use only, the construction process may result in the coating system being subjected to exposed conditions for a period before the building envelope is closed. In this case, either special provisions need to be made to protect temporarily the exposed reactive coatings, or the reactive coating needs to be evaluated as if it were to be used for exposed applications (type X).

Design of a new binder for waterborne intumescent coatings [14,15,16]

The extensive use of vinyl acetate copolymers for waterborne intumescents is linked to its relatively low cost and to the fact that it is an excellent polymer for intumescence in terms of its thermal degradation and char forming characteristics. PVAc contains oxygen, so it's a natural char forming polymer. During the intumescence process, PVAc has a natural tendency to dehydrate and form double bonds, which lead consequently to the char, the reaction product of a chemical process resulting in incomplete combustion of a solid.

Many copolymers of vinyl acetate with vinyl versatate (VV) monomers have been developed in recent years, to reduce the water sensitivity by steric protection of the vulnerable vinyl acetate group from hydrolysis. Although water resistance of such copolymers can be improved compared to polyvinyl acetates, the thermal degradation characteristics are affected and intumescent properties are not as good. In any case, even if water resistance is improved, it is not at the same level of performance as that of styrene/acrylic copolymers, such as PLIOLITE® resins, which have been used with a long and successful track record in solvent-borne intumescent coatings, and which are well known for their excellent resistance to water. The advantage of a waterborne styrene / acrylic copolymer, therefore, is potentially to enable the formulation of durable water-based reactive coatings.

A new binder has been developed that has a polymeric structure that is designed to mimic the same thermal degradation of solvent-borne PLIOLITE® resin. Degradation profiles of the new binder (referenced as HYDRO PLIOLITE® 211) and PLIOLITE resin have been compared by Thermo Gravimetric Analysis.

As can be seen in Figure 1 below, the two binders begin to decompose in the same temperature range both for the binder alone and in blend with solid chlorinated paraffin. Moreover, decomposition occurs in the same range of decomposition of the standard intumescent ingredients such as PER, MEL and APP, to enable intumescence of the coating.

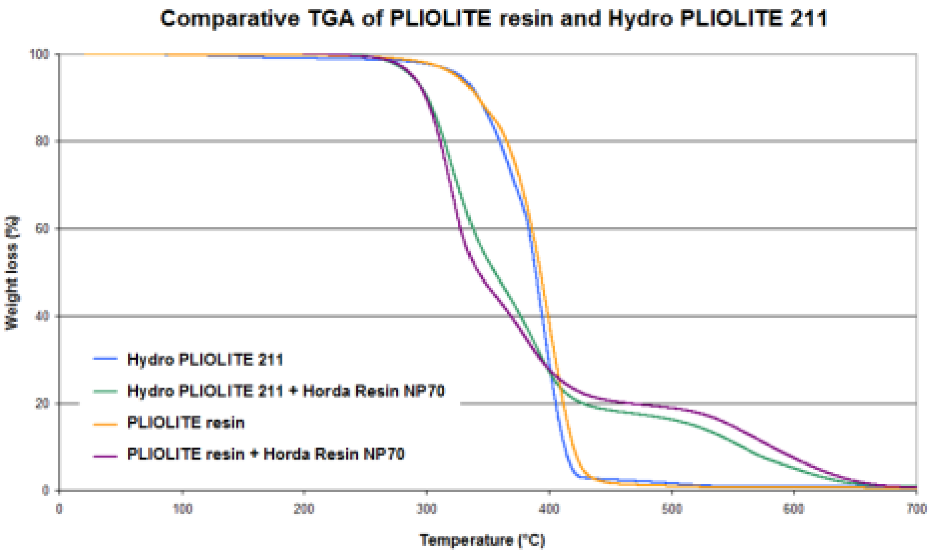


Figure 1: Thermal Gravimetric Analysis (TGA) curves of new waterborne binder vs best-in-class solvent-borne binder (Pliolite)

New binder for waterborne intumescent coatings

The new binder (HYDRO PLIOLITE 211) developed specifically for the formulation of waterborne intumescent coatings, is a styrene-acrylic emulsion copolymer, designed without the use of APEO surfactants and formaldehyde, that fully conforms to the current REACH requirements. The monomer composition has been adjusted to obtain the same good melt characteristics, and synergy with other standard intumescent ingredients (APP/PER/MEL), of PLIOLITE solvent-borne resins, which results in

an excellent and uniform char consistency. Char structure, as described by Wang et al., is one of the main parameters of effective insulation. They demonstrated that a honeycomb char structure with uniform, dense and regular cells, without micro-cracks or large voids, provides the best insulation properties.
Typical physical properties of the new binder are presented in Table 2 below:

HYDRO PLIOLITE 211 - Typical Properties	
Particle size	**150 nm**
Tg	**30.0 °C**
MFFT	**25.5 °C**
Solids content	**50.0% by wt.**
pH	**8.8**
Viscosity (Brookfield)	**< 400 mPa.s**

Table 2: Typical properties of new binder (HYDRO PLIOLITE 211)

Experimental

Testing protocol for evaluation

As far as intumescent coatings are concerned, fire resistance is the most critical test to be carried out. Although assessment of intumescent coatings for purposes of certification is carried out in large-scale furnaces with columns and beams on which paint is applied at different thicknesses, the first step in the evaluation of fire resistance is an indicative test on a steel plate in a small-scale laboratory furnace. This type of test also is used for assessment of primer compatibility and durability tests. This kind of evaluation can give much important information on the efficiency of the insulation, char consistency and char thickness.

Fire resistance of intumescent paints in small scale furnace

Cellulosic fire resistance tests on structural steel use a standard heating regime that follows the ISO834 curve, where the test furnace attains a temperature of around 950 °C after 60 minutes. An uncoated steel section placed in the furnace gradually heats up, the lag between the furnace temperature and the temperature of the steel being related to the heat capacity, or massivity of the steel, which is expressed as the section factor (Hp/A m^{-1}) This is the ratio of the exposed perimeter of the steel section to the cross-sectional area of the steel: the more massive the steel section, the lower the Hp/A, the more heat it can absorb, and the longer it takes to attain the 'failure' temperature (typically > 500 °C). In other words, the higher inherent fire resistance the steel section has, the less fire protection it requires.
When a steel section coated with intumescent is exposed to the same furnace conditions, the steel heats up, but once the coating has intumesced to form a protective insulating layer, the rate at which the temperature of the steel increases is considerably slowed (seen as an inflection on the temperature / time curve of the steel section) until, eventually, it reaches the failure temperature. The time it takes to reach the failure temperature is the fire resistance time of the coating.

For the small scale furnace tests, coatings are applied at a dry film thickness of 1 mm, on 5 mm thick steel panels of dimensions 200 x 300 mm. The temperature at the back, uncoated side of the panel is recorded by attaching 2 K-thermocouples. The furnace is heated by means of 2 propane burners, under computer control. The heating regime of the furnace is designed to follow the ISO 834 temperature / time curve for cellulosic fire. The insulation ability (fire resistance) of the intumescent coating is assessed as the time for the 2 thermocouples to reach 500 °C. Thickness and consistency of the resulting char are also reported.

Small-scale, propane-fired, laboratory test furnace

Durability Test

Accelerated aging tests for evaluation of durability are carried out in accordance with the procedures described in ETAG 018-2 and EN 16623-2015. For durability conditions X and W, painted panels are exposed to UV and water cycles carried out in a Q-UV cabinet following the conditions of EN ISO 16474-3:2013, Table 4, Cycle 2 with a cycle of 5 hours of exposure to UV-A and 50 °C, followed by 1 hour of water spray.

For Y conditions, painted panels are exposed to accelerated aging comprising temperature and humidity changes as described in Table 3 below. The test is carried out in a climatic chamber that offers temperature and humidity control during 14 days, thus 2 times the following cycle:

Day	Time			
	6 hours	6 hours	6 hours	6 hours
1 + 2	20 °C ± 3 °C 95% ± 5% rh	70 °C ± 3 °C 20% ± 5% rh	20 °C ± 3 °C 95% ± 5% rh	70 °C ± 3 °C 20% ± 5% rh
3 + 4	20 °C ± 3 °C 95% ± 5% rh	30 °C ± 3 °C 40% ± 5% rh	40 °C ± 3 °C 95% ± 5% rh	30 °C ± 3 °C 40% ± 5% rh
5 + 6 + 7	- 20 °C ± 3 °C	40 °C ± 3 °C 95% ± 5% rh	- 20 °C ± 3 °C	40 °C ± 3 °C 95% ± 5% rh

Table 3: temperature / humidity cycles for accelerated aging

Water soak test

Coatings are applied to R36 steel Q panels at a thickness of 1 mm. After 1 week of drying at ambient temperature, the panels are half-immersed in water for 3 hours. The coatings are visually assessed for signs of deterioration and then fire tested in the small-scale furnace during 10 minutes.

Formulation approach for new waterborne binder [17]

Two formulations were developed with the new binder (HYDRO PLIOLITE 211), using either pentaerythritol (PER) which exhibits important water solubility (5.25% according to typical value from the manufacturer) but which gives optimised char expansion, or dipentaerythritol (DIPER), which exhibits low water solubility (0.22% according to typical value from the manufacturer) and allows to optimise water resistance. These are referred to as:

WB IC 01 : Standard formulation (using PER)

- Designed to provide excellent fire protection at competitive cost for durability categories Z1, Z2 and Y

WB IC 02 : Premium formulation (using DIPER)

- Designed to provide excellent water resistance and good fire protection for durability categories Z1, Z2, Y, W-Y and X (depending on topcoat used)

These 2 formulations are based on the following recipe structure.

Product	%
Water	14-20
Defoamer	0,1-0,4
Dispersing agent	1-2
Titanium dioxide	6-10
Melamine	8-12
Pentaerythritol and/or Dipentaerythritol	8-12
Ammonium polyphosphate	25-30
Plasticizer	2-5
Coalescent	2-5
Associative Thickener 1	0,1-1,0
Associative Thickener 2	0,1-1,0
HYDRO PLIOLITE 211	12-20
Cosolvent	1-4

Table 4: Recipe structure for 'standard' and 'premium' water-based intumescent formulations

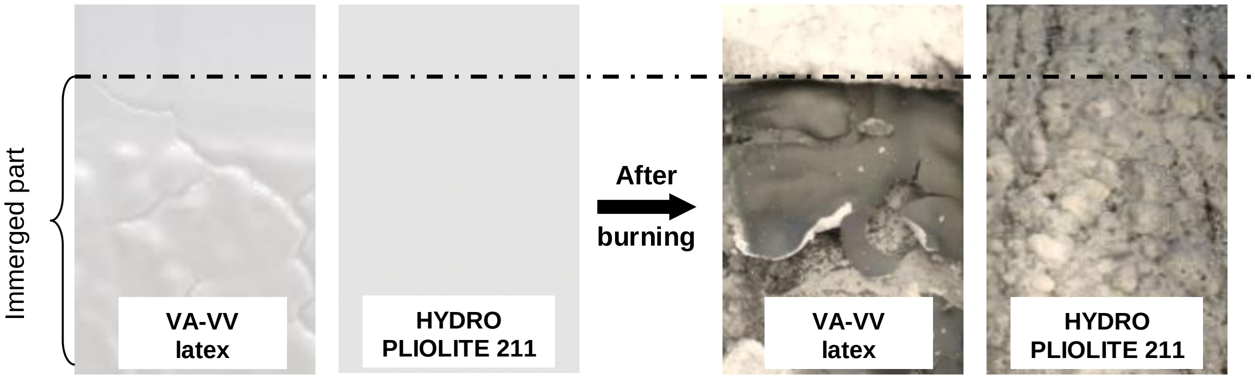
Results and Discussion

In the first part of the study, HYDRO PLIOLITE 211 was compared to a VA/VV latex recommended for intumescent paint in formulation WB IC 02 at the same binder level. Water soak test, exposure to W and Y conditions were assessed comparatively for the 2 technologies.
In the second part of the study, fire resistance of WB IC 01 based on HYDRO PLIOLITE 211 was compared to commercial intumescent coatings, both solvent-borne and waterborne systems.

<u>Comparative evaluation of new binder (HYDRO PLIOLITE 211) with commercial VA-VV latex.</u>

- **Water soak resistance**

Panels painted with intumescent paints were half-immersed during 3 hours. After drying, painted panels were submitted to ISO 834 curve during 10 minutes.



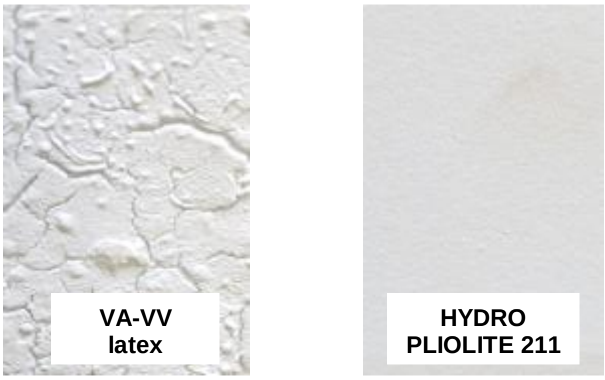
Effect of water soak on dry films

Effect of water soak on intumescent properties

The photos above demonstrate quite clearly the superior water resistance of HYDRO PLIOLITE 211 compared to the commercial VA/VV latex as the paint film of the HYDRO PLIOLITE 211 formulation was not visually affected by water immersion, while that of the VA/VV latex showed serious blistering and loss of adhesion. Furthermore, the coating based on HYDRO PLIOLITE 211maintained its intumescent properties, while that based on the VA/VV latex showed no longer any expansion on the part that was immerged.

- **W conditions exposure**

Q-panels painted with the 2 paints were exposed to W conditions in Q-UV during 5 days. Panels were visually assessed after the accelerated aging cycles.



Effect of accelerated aging (W conditions) on dry films

Whereas the coating WB IC 02 based on HYDRO PLIOLITE 211 exhibited no defect, the same formulation with VA-VV latex showed cracking and blistering due to water absorption and high sensitivity of this binder.

- **Y conditions exposure**

Coatings were applied at a dry film thickness of 1 mm on steel panel for assessment in the small scale furnace. After 1 week of drying at ambient temperature, and 1 week at 70 °C, the panels were submitted to Y conditions cycling during 2 weeks. After 3 days recovery at ambient temperature, fire resistance was assessed. Results are presented in Table 5:

	VA/VV latex	HYDRO PLIOLITE 211
Before Y conditions		
• Char thickness • Time to 500 °C • Char aspect	2 cm 41 min Bad/large voids	4 cm 53 min Uniform and crispy
After Y conditions		
• Char thickness • Time to 500 °C • Char aspect	1 cm 37 min Bad/poor expansion	2.5 cm 49 min Uniform and crispy

Table 5: Fire test results, before and after accelerated exposure (Y conditions)

Both paints fulfilled the requirement for Y conditions as fire resistance after exposure is within 85% of the fire resistance of the non-exposed panel.
Although formulation with VA/VV latex and pentaerythritol shows normally good intumescence properties and fire resistance, the formulation using dipentaerythritol, which is used for durable and exposed coatings, showed a poor quality char with low expansion and large voids.

The formulation with HYDRO PLIOLITE 211 gives good fire resistance even after Y exposure, confirming, therefore, the good suitability of this product for durable intumescent coatings. The temperature / time curves representing the fire resistance of the coatings can be seen in Figure 2 below.

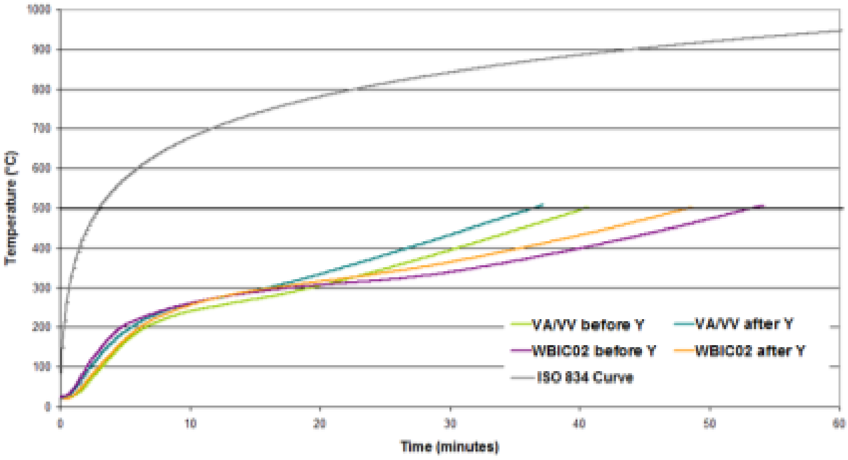


Figure 2 : Time / temperature curves of WB IC 02 vs VA/VV binder, before and after accelerated exposure (Y conditions)

Comparative evaluation of HYDRO PLIOLITE 211 formulation with commercial products

The HYDRO PLIOLITE 211 formulation WB IC 01 was compared to commercial waterborne and solvent-borne intumescent coatings obtained from the industrial market. Fire resistance was assessed on steel panels coated with 1 mm of intumescent coating. Results are presented in Table 6 below:

Paint	Time to 500 °C	Char height
WB IC 01	63 minutes	6 cm
WB Commercial A	58 minutes	6 cm
WB Commercial B	55 minutes	4 cm
SB Commercial C	53 minutes	4 cm
SB Commercial D	63 minutes	4 cm

Table 6: Fire test results for new binder vs commercial wb and sb intumescent coatings

The formulation WB IC 01 based on HYDRO PLIOLITE 211 demonstrates very good expansion and fire resistance, similar to or better than that of best-in-class intumescent coatings.

Char structure, after fire testing, of intumescent formulation based on new binder HYDRO PLIOLITE 211, compared to commercial waterborne and solvent-borne intumescent coatings

Moreover, the char structure produced by WB IC 01 is very consistent: uniform with small cells and crispy in texture, which is, as discussed previously, the ideal structure for thermal insulation. Despite very good fire resistance and expansion, the chars formed by conventional waterborne intumescent coatings exhibit a softer, more powdery aspect, which is not an ideal consistency as far as best ‘stickability’ is concerned, because the environment within a large scale test furnace as used for fire assessments can present a lot of turbulence.

Conclusion

Structural steel design is a vital component of modern architecture. Steel is a versatile building material, and its high load-bearing capacity facilitates structures with wide spans that integrate perfectly into building designs and accentuate specific features of such designs. Building regulations dictate that consideration be given to safety aspects of the construction, which sometimes may appear to conflict with the modern, ornate style of architecture. Intumescent, or reactive fire protection coating systems are perfectly suited to resolving this conflict: through their corrosion and fire protection, they provide a cost-effective solution by fulfilling all the technical requirements and at the same time accentuating the aesthetic appearance of a steel structure.
Water-based intumescent coatings based on vinyl-acetate polymers or copolymers exhibit good intumescence properties thanks to their thermal degradation behaviour but demonstrate poor water resistance linked to their chemistry.

PLIOLITE® type resins have proven their efficiency in the production of durable and effective solvent-borne reactive fire protection coatings because of their unique manufacturing process and polymer composition and morphology. Based on the same chemistry and know-how, a new water-based binder has been developed to meet best-in-class product fire resistance requirements that meets the demands of the new durability standards of European regulations.

The superior water resistance of the new binder HYDRO PLIOLITE 211, compared to current existing water-based technology, has been demonstrated using water soak and durability tests according to ETAG 018 / EN 16623 in this study. The better compatibility with intumescent raw materials for durable intumescent coatings has also been evidenced in a typical water-based formulation.

Finally, one further advantage of the new binder compared to existing technologies is that intumescent formulations developed using it exhibit excellent viscosity stability at low temperature (5 °C) without having to resort to the use of special grades of the common intumescent raw materials.

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