



Durability of intumescent coatings and recommendations for test concepts for a working life of more than 10 years

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ABSTRACT

It is an essential requirement for all building products to ensure durability of their fire safety. Throughout the working life of products, intumescent coatings are aged by certain climatic factors. To predict a lifetime of several years, generally the behaviour of the intumescent coating is extrapolated based on accelerated artificial ageing. The established German and European procedures to assess the durability assume a working life of at least 10 years. For a longer period, additional evidence is required; yet the procedure and the specifications to justify this are not described. In addition to addressing this formal lack, from a scientific point of view it is necessary to investigate the degradation of intumescent coatings in detail and to propose a reliable test concept to assess durability for more than 10 years. This paper summarises the existing knowledge about the ageing of intumescent coatings. The results of various demanding weathering approaches are presented for two intumescent coatings tested in a joint research project. Moreover, formulations with a reduced amount of functionally relevant components were analysed to gain insight into the associated effects. Derived from these research results and knowledge, recommendations are proposed to assess the durability of intumescent coatings for more than 10 years based on a combination of verifications.

1. Introduction

Intumescent coatings [1], also known as reactive fire protection systems, are used in structural fire design to increase the fire resistance of steel structures [2–4]. The application of these coatings according to required fire resistance makes it possible to fulfil the architectural design requirements, while the low coating thickness results in marginal additional loads for the structure. When exposed to elevated temperature in case of fire, the intumescent coating applied to the steel member expands to multiple times the initial thickness to form a thermally protective char [5,6]. This char considerably reduces the heating of the underlying steel member and thus delays the deterioration of the mechanical steel properties caused by high temperatures. Thereby, the fire resistance of the steel member is improved.

The potential ageing of intumescent coatings is caused by certain climatic factors. Depending on the conditions at the application site, the intumescent coating is exposed to changes in temperature, humidity [7], rain [8], freezing and thawing [9,10], acid polluted environment [11],

and/or ultra-violet radiation in varying degrees of intensity [12,13]. This exposure may result in decomposition of the components required for intumescence, e.g., migration of components [7] in the presence of water [12], or physical failure of the coatings [14] such as blisters [8], cracks [11], ablation [13], and erosion [10]. A loss of relevant components must be expected as a result of leaching out or blooming, as well as from the decomposition of the polymeric binder [15] and thus reduce the thermal protective performance of the intumescent coating.

In Germany, to test and assess the durability of intumescent coatings, the national procedure for granting national approval (abZ or aBG) and the European procedure for granting a European Technical Assessment (ETA) are applicable. The two procedures are based on different fundamental approaches. Thus, they are not directly comparable and must be distinguished from one another, especially with regard to interpreting the findings obtained. The European procedure is based on accelerated artificial (short-term) weathering tests, while the German procedure applies both accelerated artificial (short-term) and natural (long-term) weathering, whereby the long-term tests extend the validity

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period of the national approval granted by the results of the short-term tests. Detailed information about the two procedures is given in Chapter 2.1. Regardless of which procedure is used to prove durability, the regulations are based on specified assumptions regarding environmental influences.

Recently, the durability of intumescent coatings was investigated within a comprehensive research project [16] accompanied by associated industry partners, e.g., manufacturers of intumescent coatings. The main conclusions of the research project are presented in this paper. Data from short- and long-term tests of commercial products approved in the last 30 years were analysed. In addition, based on different approaches to the artificial ageing of specimens, a comprehensive experimental programme was carried out to address the thermal protective performance of one water-based and one epoxy-based intumescent coating, both without topcoat. These intumescent coatings tested are not commercial products, but two representative guide formulations; see Fig. 1. Moreover, numerical simulations were also performed to estimate the reduced thermal protective performance of aged intumescent coatings [16]. Finally, a proposal for a test concept to prove the durability of intumescent coatings for a working life of more than ten years was proposed.

2. Review of procedures

2.1. German procedure

The German procedure to assess the durability of intumescent coatings is described in Ref. [17] and requires fire tests after short- and long-term weathering. In both cases, steel plates with intumescent coating are tested. The applied weathering scenarios differentiate between indoor and outdoor application. The long-term specimens for indoor use are usually stored on premises of the test laboratory. The outdoor weathering takes place at special outdoor areas of the individual test laboratory, assuming comparable exposure and weathering conditions at the different testing sites in Germany. The short-term weathering tests are carried out first; when the product passes the corresponding fire tests, it receives a temporary national approval for 2 years. The short-term specimens are exposed to defined weathering cycles in a climate chamber (see Appendix A.3) and fire-tested afterwards using the standard temperature/time curve [18]. The thermal performance of the weathered specimens is compared to the

performance of the specimens in the initial state, i.e., without weathering. If the weathered specimens show ‘significantly lower performance’, national approval is not granted, or, in the case of long-term tests, not extended.

The specimens for the long-term weathering tests are gradually assessed as extension of the approval after 2 years approaches, and also 3 and 5 years thereafter. Therefore, at the time of testing the specimens have been stored for 2 years (1st extension), 5 years (2nd extension), or 10 years (3rd extension). Durability tests beyond 10 years are not considered for further extensions of national approval unless facts become known that indicate deteriorated performance of the intumescent coating. The German approach to approving the durability of intumescent coatings assumes that if no degradation takes place in the first 10 years of working life, it will probably also not happen thereafter.

2.2. European procedure

At European level, the durability of intumescent coatings is assessed based on the regulations given in the European Assessment Document (EAD) 350402-00-1106 [19], where exclusively accelerated artificial (short-term) weathering tests are applied. The artificial weathering ensures that test laboratories all over Europe generate the same results. The weathering scenarios (see Appendix A.3) to be applied in the short-term weathering are designed to apply similar environmental conditions at the application site of the intumescent coating, i.e., type Z₂ (internal conditions with humidity lower than 85 %), type Z₁ (internal conditions with humidity equal to or higher than 85 %), type Y (semi-exposed), and type X (directly exposed). In the specified order, the stress level increases from indoor use with low temperature changes and low changes in humidity to outdoor use with high temperature changes including freezing and changes in humidity as well as exposure to ultra-violet radiation and spray water. According to EAD 350402-00-1106, effects from contamination of the environment must be taken into account by additional considerations in the context of issuing an ETA.

The durability of the intumescent coating is assessed by comparing the thermal protective performance of coated steel plates with and without accelerated artificial (short-term) weathering. As a comparative parameter, the time to reach a steel temperature of 500 °C (t_{500}) is used. The specimens are tested in small-scale fire tests using the standard temperature/time curve according to EN 1363-1 [18]. Durability is

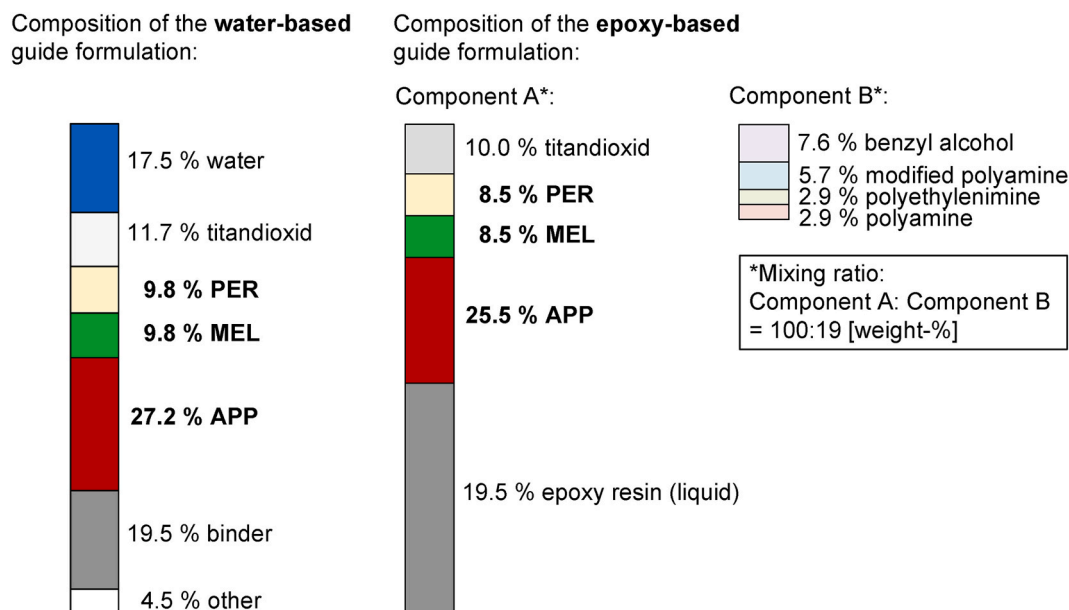


Fig. 1. Chemical compositions of the water-based and epoxy-based guide formulations (APP = ammonium polyphosphate, PER = pentaerythritol, MEL = melamine).

proven when the average t_{500} of the weathered specimens is not less than $0.85 \times t_{500}$ of the specimens without weathering (initial tests). Moreover, the t_{500} of each weathered specimen must not be less than $0.8 \times t_{500}$ of the initial tests.

The test and assessment methods in EAD 350402-00-1106 take into account a working life of 10 years for the intended use of the intumescent coating. It should be noted that the working life assumption is intended to prove for economical use of the products with regard to the expected lifetime of a structure. It is neither a guaranteed minimum value nor an absolute upper limit with regard to the durability of the product. It is possible to increase the estimated working life to 25 years if the manufacturer offers sufficient documented evidence to demonstrate to the Technical Assessment Body (TAB) the safe use of the product for this period under the given environmental conditions. However, no suitable testing and assessment concept for this proof is given in EAD 350402-00-1106 [19]. Therefore, it is up to the TAB to decide whether the information provided by the manufacturer is sufficient.

3. State of the art

In the last decade several research projects have addressed the durability of intumescent coatings, including different types of natural [9,13] and artificial [7,8] exposure scenarios, and enhancing formulations improve durability by methods such as using microencapsulated APP [20], an APP with higher degree of polymerization [21], or inorganic additives which reduce the permeation of water [22,23], and by exchanging specific components such as expandable graphite (EG) instead of APP [24] or different polymeric binders [25]. Moreover, manufacturers of intumescent coatings constantly work to improve the durability of their products. A selection of these investigations and their main conclusions are presented below.

In 2013 Jimenez et al. [26] published investigations on small-scale steel plates coated with an epoxy-based intumescent coating. The specimens were fire-tested after complete immersion in different types of water. The results showed that the thermal protective performance degraded due to the water exposure. Salt water led to a complete loss of thermal protective performance. Using X-ray mapping, a leaching out of functionally relevant components was identified as a main degradation effect. In a subsequent research work, specimens with a topcoat were exposed to salt water. In the fire test no significant loss of the thermal protective performance was observed [27].

Investigations on the accelerated artificial (short-term) weathering of small-scale steel plates coated with two different solvent-based intumescent coatings were published by Wang, LL. et al. [28,29]. The type Z₁ use scenario according to EAD 350402-00-1106 [19] was applied up to two times. The fire tests showed that the expansion of the char as well as the thermal protective performance were reduced with increasing exposure time. Furthermore, the intensity of the degradation depended on the given product. In an additional research work, the effect of different topcoats with varying thicknesses was analysed. As a result, a significant reduction in char thickness and thus deterioration of the thermal protective performance was avoided by using an appropriate topcoat.

In a previous research project at BAM [30] the degradation of water-based intumescent coatings was analysed based on accelerated artificial (short-term) weathering applied to small-scale specimens and corresponding fire tests [31,32]. To gain a better understanding of the weathering and ageing phenomena, the decomposition of the individual components as well as their chemical and physical interactions were investigated. The essential chemical components for intumescence, i.e., ammonium polyphosphate (APP), pentaerythritol (PER), titanium dioxide (TiO₂), melamine (MEL), and the binder, were exposed separately to changes in humidity and temperature as well as to ultra-violet radiation. For APP, MEL, and the binder the greatest effect on the durability and the corresponding degradation of the thermal protective performance of the intumescent coating was determined. The main

mechanisms were driven by moisture, which is therefore considered as the dominant influencing factor. The findings of this research were used to improve the initial formulation of the intumescent coating. The APP was replaced by encapsulated APP, which delayed the reaction of MEL and APP, and PER was replaced with di-PER, improving the durability of the intumescent coating.

More comprehensive reviews on the durability of intumescent coatings have been published elsewhere [33,34]. A detailed description of the state of the art can also be found in monographies such as [35]. Moreover, manufacturers have dedicated massive research efforts to improving the durability of intumescent coatings and published information about their products with respect to achieving a working life of more than 10 years [36]. However, to assess the long-term stability of intumescent coatings, some intrinsic challenges are identified. The simple physicochemical models of the kinetics of degradation are rather uncertain when extrapolated to longer time periods. The ageing of the coatings regenerates the relevant kinetics, changing key parameters such as the properties of the surface or the polarity of the chemical components. Furthermore, a scientific and systematic description of the degradation of fire protective performance is required. The correlation between the chemical ageing of the components and the resulting fire protection falls short when it comes to details or an unambiguous mathematical description. Congruously, there is a need for clarification as to whether the already established short-term weathering procedure is suitable to assess the durability of intumescent coatings for more than 10 years.

4. Results and discussion

4.1. Analysis of artificial short-term and natural long-term tests

The German approval procedure [17] requires proof of durability through short-term and long-term tests. Thus, a database has been accumulated over the last decades, which allows a systematic analysis of the durability of intumescent coatings both under artificially accelerated (short-term) ageing as well as under natural (long-term) climatic conditions. In cooperation with several manufacturers of intumescent coatings, which are associated partners in the research project [16], a total of eight test datasets from approved commercial products were analysed. All analysed products passed the durability tests and have been granted extended approval. For data protection reasons the data were anonymized. The datasets include different product types, i.e., water-, solvent- and epoxy-based intumescent coatings, which are used for both interior and exterior applications. The fire tests were carried out in accordance with DIN 4102-8 [37] using steel plates 500 mm × 500 mm × 5 mm in size with an intumescent coating applied on the front side. The analysis of the available data showed no significant reduction in the thermal protective performance. Thus, no time-dependent degradation occurred. As an example, the comparison of test data from short- and long-term tests of a solvent-based intumescent coating for indoor application is given in Fig. 2. The deviations between the specimens are within the expected scatter of fire tests. The measured steel temperature curves show no significant differences between specimens with and without weathering. For two datasets, information regarding the dry film thickness (DFT) was available before and after weathering, which showed an increase in the DFT with exposure time. However, this did not result in any change to the thermal protective performance of the intumescent coating.

The analysed data showed that the thermal protective performance obtained in the accelerated artificial (short-term) tests does not differ significantly from the results of the long-term natural tests conducted over a period of 10 years. However, the amount of available data used for the comparison was small and included products that have successfully fulfilled durability requirements and therefore obtained approval. Test data of intumescent coatings that have passed the accelerated artificial (short-term) tests but failed the long-term natural tests were

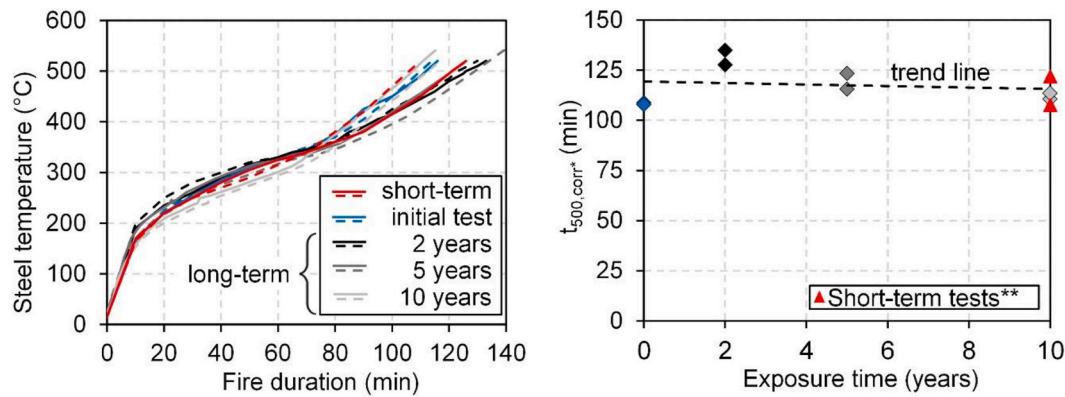


Fig. 2. Summary of results of test data for assessing the durability of a commercial product from the German approval procedure [17]

The data shown are for a solvent-based intumescent coating with a tested DFT of 2600 μ m

* The scatter in the actual DFT of the specimens was taken into account in the assessment, i.e., analogue to the procedure according to EN 13381-8 [38], Annex D

** In line with the European procedure [19], a working life of 10 years was also assumed when the short-term tests were passed according to the German procedure [17].

not available. In summary, the analysis of eight natural (long-term) weathered products did not show any significant reduction in thermal protective performance.

4.2. Specimens with a reduced amount of functionally relevant components

The ageing of intumescent coatings induced by weather results in a decrease in the reactivity or concentration of functionally relevant chemical components. The ageing rate and thus the concentration of the aged products as a function of the environmental impact is unknown. From the literature [26,33] it is already known that moisture as well as exposure to water result in a migration of functionally relevant components to the surface of the intumescent coating. Consequently, these components are no longer available for a chemical reaction in case of fire, such that the thermal protective performance deteriorates. To

address these degradation mechanisms and to gain insight into the correlation between the functionally relevant chemical components and the thermal protective performance of intumescent coatings, ageing was simulated by selectively reducing the amount of individual chemical components. Thus, the ageing-related change in the chemical structure of the intumescent coating was incorporated directly into the production process of the specimens, thus eliminating the need for time-consuming weathering. Based on the guide formulations of two intumescent coatings (Var. 1), a large number of small-scale specimens with a reduced amount of functionally relevant components (Var. 2 to 13) were produced and subsequently fire-tested simultaneously; see Table 1. The approach of reducing the amount of functionally relevant components was carried out on both a water-based and an epoxy-based intumescent coating. Details about the manufacturing of the specimens (steel plates 75 mm \times 75 mm \times 5 mm in size coated with a DFT of 1.0 mm), the fire testing, and thermogravimetric analysis (TGA) are described in the

Table 1

Test programme for fire tests on specimens with a reduced amount of functionally relevant components.

Water-based intumescent coating*				Epoxy-based intumescent coating			
Variant	APP	MEL	PER	Variant	APP	MEL	PER
1	100 %	100 %	100 %	1	100 %	100 %	100 %
2	90 %	100 %	100 %	2	95 %	100 %	100 %
3	80 %	100 %	100 %	3	90 %	100 %	100 %
4	70 %	100 %	100 %	4	85 %	100 %	100 %
5	100 %	90 %	100 %	5	100 %	95 %	100 %
6	100 %	80 %	100 %	6	100 %	90 %	100 %
7	100 %	70 %	100 %	7	100 %	85 %	100 %
8	100 %	100 %	90 %	8	100 %	100 %	95 %
9	100 %	100 %	80 %	9	100 %	100 %	90 %
10	100 %	100 %	70 %	10	100 %	100 %	85 %
11	90 %	90 %	90 %	11	95 %	95 %	95 %
12	80 %	80 %	80 %	12	90 %	90 %	90 %
13	70 %	70 %	70 %	13	85 %	85 %	85 %

Note: The values in the table represent the concentration of the chemical components used for the different variants of the intumescent coating. Water-based and epoxy-based intumescent coatings were tested, with a total number of 27 specimens each. Variant 1 was tested three times and all other variants were tested twice.

* Due to workability, in all variants the water content was reduced by 20 % compared to the guide formulation.

supplementary information; see Appendix A.1, A.2 and A.4.

The fire tests conducted in the 1 m³-furnace showed that even with a major reduction of functionally relevant components the intumescent coating still reacted well and formed a thermally protective char. In the case of the water-based intumescent coating the reduction of the components APP, PER and MEL changed the thermal protective performance; see Figure A.11 in the supplementary information. The simultaneous reduction of all three components resulted in significant deterioration and thus a faster increase of the steel temperatures compared to the guide formulation. The specimens with a reduction of one component showed different results; e.g., the thermal protective performance usually decreased for APP and PER and increased for MEL. This third result is probably due to the fact that the selected guide formulation does not represent a formulation optimised for thermal protective performance, so that changes in the chemical composition of the intumescent coating can have a positive effect. The epoxy-based intumescent coating showed small differences in thermal protection performance between the specimens with the guide formulation and those with a reduced amount of functionally relevant components; see Figure A.11 in the supplementary information. This may result from the weaker concentration of the selected component or from improved chemical stability.

The results of the fire tests are in line with those of the TGA. For the water-based intumescent coating, the thermal decomposition differed significantly from the guide formulation due to the changed concentration of the functionally relevant components. In contrast, the modification in the concentrations of the epoxy-based coating hardly changed the results of the TGA.

In summary, for both investigated intumescent coatings, i.e., water-based and epoxy-based, no direct correlation between the reduction of functionally relevant components and the thermal protective performance was identified. Most likely, the selected components in the intumescent coatings are available in such excess that, despite the reduction, a stable and thermally protective char can still be formed in case of fire. The information regarding the chemical composition of an intumescent coating serves primarily to identify the product. Due to the complex reactions and processes taking place during the foaming of an intumescent coating, components in excess, as well as the natural scatter of fire tests, no direct correlation was observed between the changed chemical composition and the thermal protective performance. Furthermore, with the approach of reducing the amount of functionally relevant components it is hardly possible to consider the physical changes resulting from weathering, such as pore enlargement, blistering, crack formation, or delamination. This structural degradation of the microstructure of an intumescent coating is a major reason for the deterioration in thermal protective performance and durability. In addition, the degradation of the binder, which affects the viscosity during the foaming process of the intumescent coating, needs to be considered.

4.3. Multiple weathering approach

4.3.1. Water-based intumescent coating

To analyse the durability of the water-based intumescent coating for

a period of more than 10 years, the duration of the accelerated artificial (short-term) weathering tests was significantly extended. In this multiple weathering-period approach, the specimens were weathered in a climate chamber under similar conditions as type Z₁, at up to six times the exposure duration given in EAD 350402-00-1106 [19]. The test programme included four test series; an initial state without weathering and accelerated artificial (short-term) weathering cycles of 1 x Z₁, 3 x Z₁, and 6 x Z₁. The accelerated artificial (short-term) weathering tests were carried out on small-scale steel plates (75 mm × 75 mm × 5 mm) coated with a DFT of 1.0 mm. All specimens had a primer applied on all sides and an intumescent coating applied to the front side (75 mm × 75 mm), but no topcoat. The test programme consisted of 30 specimens with water-based intumescent coating.

After about three days of weathering, the first blisters appeared on the surface of some specimens. Several blisters had already burst at the end of the first complete weathering cycle (1 x Z₁). After three (3 x Z₁) and six (6 x Z₁) complete cycles of weathering, the condition of the intumescent coating had deteriorated to such an extent that cracks or blisters formed in some cases (Fig. 3). Furthermore, an increase in weathering duration resulted in an increase of the DFT, due to moisture-related swelling and shrinking processes and the resulting enlargement of the pores, as well as partial delamination of the intumescent coating. After completion of the weathering cycles, no significant weight loss was determined for the weathered specimens as compared to the specimens in the initial state.

Based on the results of the TGA, a change in thermal decomposition was already determined after the first weathering cycle (1 x Z₁). All weathered specimens showed an additional decomposition stage between 50 °C and 100 °C due to the release of water. The decomposition stage between 200 °C and 250 °C in the initial state was changed due to the weathering exposure. The reaction between APP and PER is considered to be the reason for this change. The changes in the TGA occurred in both nitrogen and artificial air atmosphere. More detailed results are given in the supplementary information; see Appendix A.2.

To assess the thermal protective performance of the water-based intumescent coating in the initial state and after weathering, fire tests were carried out in the 1 m³-furnace as well as in the electric furnace with 1 L volume on specimens (75 mm × 75 mm × 5 mm) with a DFT of 1.0 mm. The two furnaces and the experimental test setup are described in detail in Appendix A.4 and A.5 of the supplementary information. The electric furnace was also used to analyse the expansion behaviour of the intumescent coating during the heating process. For this purpose, specimens with a diameter of 30 mm, a steel plate thickness of 0.75 mm, and an intumescent coating applied with a DFT of 0.5 mm were used. The specimens were also exposed to accelerated artificial (short-term) weathering. The main results are summarised below.

In the fire tests both in the electric furnace and 1 m³-furnace, the thermal protective performance of the weathered specimens decreased significantly compared to the specimens in the initial state (Fig. 4). It is important to mention that the fire tests conducted in the electric furnace and 1 m³-furnace have a different test set-up and different actual fire exposure, so that no direct comparison of the results is possible. The fire tests with the electric furnace show that an extension of the weathering exposure time resulted in further deterioration of the thermal protective

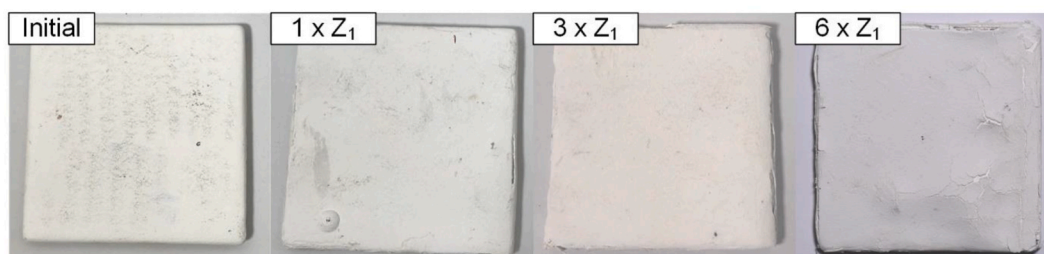


Fig. 3. Example of the water-based specimens in the initial state and after performing the accelerated artificial (short-term) weathering cycles.

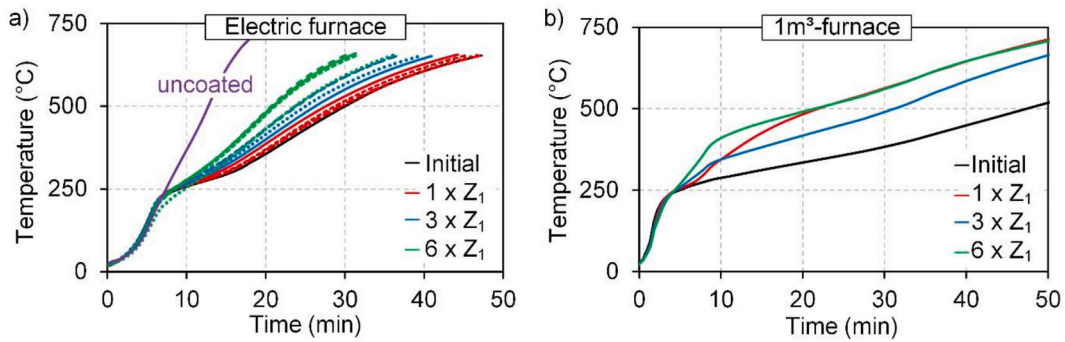


Fig. 4. Steel temperature/time curves obtained from the fire tests in the (a) electric furnace and (b) 1 m³-furnace carried out for the water-based intumescent coating. Note: Due to the electric heating, in the first 5 min of the electric furnace tests the gas temperature was lower than the standard temperature/time curve. The dashed lines represent repeated measurements.

Because of a defective furnace thermocouple, during the first 10 min of the fire test in the 1 m³-furnace the gas temperature was significantly higher than the standard temperature/time curve; see Appendix A.8.

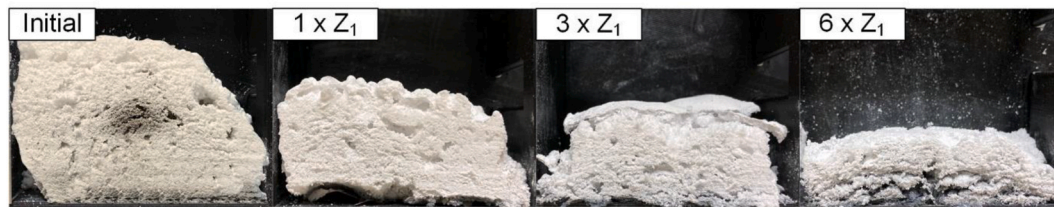


Fig. 5. Exemplary sectional views of the char of the water-based intumescent coating after fire tests in the electric furnace.

performance of the intumescent coating. The deterioration of the thermal properties is due to the change in the morphology of the char. For instance, the fire tests in the electric furnace show a decrease in char thickness with increasing duration of the accelerated artificial (short-term) weathering (Fig. 5). This effect was also measured in the expansion tests. In addition, a change in the char was observed in the fire tests due to the accelerated artificial (short-term) weathering, i.e., an increase in the number of large pores, and in some cases, reduced char thickness.

4.3.2. Epoxy-based intumescent coating

Similar to the water-based intumescent coating, the multiple weathering-period approach was used to assess the epoxy-based guide formulation. However, the short-term weathering conditions applied were defined in accordance with type X of EAD 350402-00-1106 [19]. A total of four test series was conducted: initial state without weathering, and accelerated artificial (short-term) weathering cycles of 1 x X, 3 x X, and 6 x X. The accelerated artificial (short-term) weathering tests were carried out on small-scale steel plates (75 mm × 75 mm × 5 mm) coated with a DFT of 1.0 mm. All specimens had a primer applied on all sides and an intumescent coating applied to the front side (75 mm × 75 mm), but no topcoat. The test programme consisted of 38 specimens with epoxy-based intumescent coating. Detailed information on the experimental investigations is also provided in the supplementary

information; see Appendix A.2 to A.8. The following text summarises the conducted tests and the obtained results of the epoxy-based intumescent coating.

After the first day of weathering, the specimens already showed obvious yellowing (Fig. 6). After further weathering the colour darkened and changed to a shade of brown. During the artificial accelerated weathering thin net-like cracks appeared on the surface of the intumescent coating. Yet, none of the weathered specimens, even those that had been weathered for a duration of six times the type X condition, showed any wide cracks, blisters or delamination of the intumescent coating. Furthermore, no loss of weight or DFT was detected.

The results of the performed TGA showed no differences in thermal decomposition between the initial state of the epoxy-based intumescent coating and those that were exposed to the accelerated artificial (short-term) weathering.

The thermal protective performance of the epoxy-based intumescent coating in the initial state and after weathering was assessed by fire tests in the 1 m³-furnace and the DIN 4102-8 furnace. For this purpose, specimens with dimensions of 75 mm × 75 mm × 5 mm and a DFT of 1.0 mm were tested. In addition, the expansion behaviour of the char was analysed in fire tests based on specimens with a diameter of 30 mm, a steel plate thickness of 0.75 mm, and an intumescent coating applied with a DFT of 0.5 mm. The steel temperature/time curves obtained in

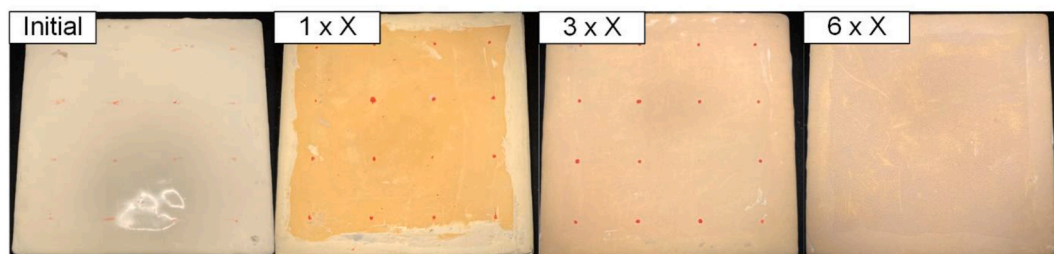


Fig. 6. Example of the epoxy-based specimens in the initial state and after performing the short-term weathering cycles.

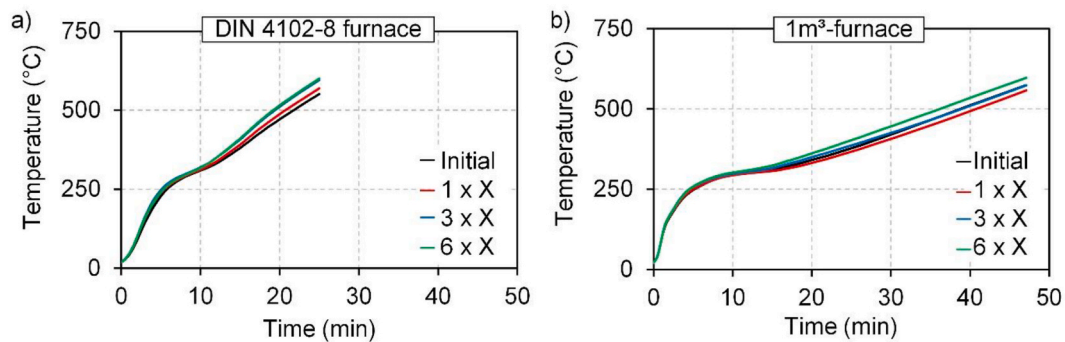


Fig. 7. Steel temperature/time curves obtained from the fire tests carried out for the epoxy-based intumescent coating: (a) DIN 4102-8 furnace, (b) 1 m³-furnace.

the fire tests showed no significant differences between the initial state specimens and those exposed to the artificial accelerated weathering (Fig. 7). Thus, the epoxy-based intumescent coating had a high durability and was able to withstand the challenging exposure conditions multiple times. The change in colour, which occurred due to the artificial accelerated weathering, did not affect the thermal properties of the epoxy-based intumescent coating in the event of fire. Furthermore, the morphology of the char remained unchanged. This applies to the char structure and thickness. In contrast to the tested water-based product, the epoxy-based intumescent coating formed a char consisting mainly of a thin crust-like top layer and a large underlying cavity.

4.4. Summary of the experimental investigations

Different approaches for artificial ageing were conducted, i.e., reducing the amount of functionally relevant components, and extending the accelerated artificial (short-term) weathering tests. In the experimental investigations, guide formulations of one water-based and one epoxy-based intumescent coating were used, and demanding weathering conditions were selected. The effects of the artificial ageing were assessed based on visual inspections, thermo-analytical methods, and fire tests. The results allow insight into the degradation of intumescent coatings.

Based on the investigations carried out with the water-based guide formulation, an extension of the weathering period resulted in increased degradation of the intumescent coating. The degradation was detectable both visually after the accelerated artificial (short-term) weathering tests by means of blisters and cracks, as well as after the fire tests, respectively, in terms of reduced thermal protective performance and changes in char morphology. In addition to the duration of exposure, it must be noted that the weathering scenario and the absence of a topcoat had a decisive influence on the degradation. The selected weathering scenario of type Z₁ was a tough challenge for the water-based intumescent coating due to the high humidity in combination with the omitted topcoat. In particular, the swelling caused by the high humidity and the subsequent shrinkage during the drying phase facilitate the formation of blisters, cracks and may result in partial delamination of the intumescent coating.

Except for the colour change and thin net-like cracks, which are both probably due to minor changes in the chemical structure, the extension of the weathering duration did not result in any dramatic physical or chemical degradation of the tested epoxy-based intumescent coating. Thus, the thermal protective performance was not affected. This is remarkable, since no topcoat was used, and the type X weathering scenario with the most challenging conditions was applied according to EAD 350402-00-1106 [19].

Regarding the two investigated intumescent coatings, it can be assumed that if no significant physical, chemical or thermal degradation occurs after the first complete weathering cycle, such effects are not to be expected for an extended exposure time. Furthermore, the

degradation is significantly influenced by the selected weathering scenario and intumescent coating, e.g., water-based intumescent coatings are vulnerable to moisture-based weathering scenarios.

5. Proposal for a test concept to assess a working life of more than 10 years

Based on the results of the experimental investigations and the state of the art, recommendations for a test and assessment concept covering a period of more than 10 years are proposed. The regulations already established in EAD 350402-00-1106 [19], which aim for a working life of 10 years, serve as a starting point. Thus, the proposal focuses on the assessment as part of the approval procedure for intumescent coatings. As the assessment via accelerated artificial (short-term) weathering uses extrapolation, the extension of the extrapolated period results in higher uncertainties. A reliable extension of the working life from 10 to 25 years cannot be achieved simply by increasing the duration of the short-term tests linearly by a factor of 2.5. Therefore, an extended test and assessment concept with additional and appropriate approaches is required. In the following, several possibilities are presented:

Extension of the short-term artificial accelerated (short-term) weathering duration by a factor that is higher than the intended working life divided by 10 years, e.g., a factor of 3 for an intended working life of 25 years.

A narrow assessment criterion for the tolerable reduction of the thermal protective performance of the weathered intumescent coating, e.g., the difference between the mean value for t_{500} of the weathered compared to the initial specimens can be reduced to 10%, instead of the 15 % given in EAD 350402-00-1106 [19]. However, it must be noted that the results of fire tests have a natural scatter, which cannot be avoided.

Use of more challenging use scenarios, e.g., applying cyclic weathering according to EAD 350402-00-1106 [19] with more demanding environmental conditions. According to EAD 350402-00-1106, a more extensive use scenario (e.g., type X) covers all less demanding use scenarios (e.g., type Y, Z₂, Z₁). Necessarily, if the intended use scenario is less demanding than tested, a longer working life can be assumed [36].

Application of advanced use scenarios with more challenging conditions than specified in EAD 350402-00-1106 [19], e.g., a particular weathering simulation targeting specific factors or covering specific damage mechanisms.

Verification of long-term durability for more than 10 years based on steel constructions with aged intumescent coating. This approach is possible only if the chemical composition of the product has not changed. For new products, this approach is not suitable.

These possible approaches should not be considered exhaustive and may be complemented by other suitable ideas. In general, to extrapolate the working life for a duration of more than 10 years, it is particularly important to combine several individual approaches. For instance, a

robust and yet practicable test concept could consist of extending the accelerated artificial (short-term) weathering duration combined with applying a more challenging weather scenario and, if possible, a narrower assessment criterion. In Germany, based on these approaches, first products have already obtained an ETA confirming a working life of 25 years [36]. In addition to the selected test concept, as for all other building products, it is necessary to ensure that the intumescent coating applied on the steel structure remains intact over the entire working life. Therefore, regular inspections are mandatory. As already described in EAD 350402-00-1106 [19] for a working life of 10 years, the extended test concept does not cover incorrectly selected environmental conditions, nor improper application or special phenomena, such as mechanical or water damage.

To conclude, since the European assessment method for testing durability is based on an extrapolation of the results from accelerated artificial (short-term) weathering tests, the uncertainties are expected to increase with a longer working life. To address this and to achieve high reliability, it is particularly important to combine several individual approaches. In addition, it is necessary to ensure that the intumescent coating applied on the steel structure remains in an intact condition during the entire working life.

6. Conclusion

The following statements refer to the two guide formulations tested. In principle, it is possible to use the findings for other intumescent coatings as well, yet the differences in chemical composition must be accounted for. The main conclusions derived from the state of the art, supported and updated by the conducted research [16] are the following:

- I. Intumescent coatings are affected by ageing and weathering, respectively. As a result, their thermal protective performance can deteriorate or, in extreme cases, disappear entirely. The durability of intumescent coatings is significantly influenced by the product itself, i.e., the layer structure and chemical composition, as well as by the acting environmental conditions, i.e., the level of weathering and duration. For example, water-based products are usually designed for indoor durability requirements. Due to their water solubility, they are susceptible to environmental conditions with high humidity or spray water, especially if no topcoat is used. For internal conditions with a humidity of less than 85 %, water-based intumescent coatings have already obtained an ETA for a working life of 25 years [36].
- II. The ageing of intumescent coatings is complex. The associated changes affect not only their chemical composition, e.g., transformation or the leaching out of individual components, but also the physical and structural properties of the intumescent coating, e.g., blister or crack formation. Thus, the degradation of the thermal protective performance results from a complex interrelationship of chemical and physical ageing of the intumescent coating, specific to each individual product. Consequently, it is very difficult to describe the degradation and to link it directly with the actual thermal protective performance of the intumescent coating. Furthermore, statements for a specific product usually cannot be generalised, i.e., transferred to other products.
- III. The thermal protective performance over a product's working life is affected by individual ageing effects, e.g., no significant degradation may occur up to a boundary value in the change of the chemical composition. In addition, the interaction of ageing effects needs to be considered, e.g., accelerated water absorption as a result of cracks in the intumescent coating. Discontinuously occurring phenomena may occur, e.g., delamination of the coating. As a result, degradation cannot be assumed as a linear function over time, so that a linear extrapolation may not be appropriate.

- IV. Based on the current knowledge, it is rather unlikely that degradation and its corresponding effect on the thermal protective performance of an unknown aged intumescent coating can be assessed using an approach with reduced complexity. Thus, merely analysing the chemical ageing of key components via spectroscopic or thermo-analytical measurements will not be sufficient. For instance, a significant change in chemical composition may occur before thermal protective performance is affected. On the other hand, blistering, cracks, or partial delamination may not change the chemical composition, but already significantly reduce the thermal protective performance.
- V. If there is clear visible degradation of the intumescent coating, e.g., colour changes, blistering, cracking or partial delamination, the thermal protective performance is often reduced. However, this is only an indication and not a general consequence. For example, the epoxy-based product investigated showed visual changes without a reduction in thermal protective performance. On the other hand, a visually unchanged intumescent coating is not a guarantee for intact thermal protective performance [30].
- VI. Commercial intumescent coatings with a valid approval or ETA have proven their durability for a working life of at least 10 years. This requires the correct selection of the environmental conditions, method of application and maintenance of the product. The application of an appropriate topcoat is a very effective method to improve the durability of an intumescent coating [29].

Although the accelerated artificial (short-term) weathering procedure given in EAD 350402-00-1106 [19] is not an exact projection of a real ageing of 10 years, there is no principal shortcoming to this established assessment method. Accelerated artificial (short-term) weathering is based on defined and controlled conditions and provides comparable, repeatable, and reproducible results. Thus, from both a formal and scientific position the accelerated artificial (short-term) tests can be used as a practical starting point for a testing concept to assess the durability of intumescent coatings for a working life of more than 10 years.

CRediT authorship contribution statement

Dustin Häßler: Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Resources, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. **Maximilian Mund:** Writing – review & editing, Writing – original draft, Visualization, Validation, Resources, Methodology, Investigation, Formal analysis, Data curation. **Lars-Hendrik Daus:** Writing – original draft, Visualization, Methodology, Investigation, Formal analysis, Data curation. **Sascha Hothan:** Writing – review & editing, Writing – original draft, Supervision, Project administration, Methodology, Funding acquisition, Conceptualization. **Peter Schaumann:** Writing – review & editing, Writing – original draft, Supervision, Project administration, Methodology, Funding acquisition, Conceptualization. **Bernhard Schartel:** Writing – review & editing, Writing – original draft, Supervision, Project administration, Methodology, Funding acquisition, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

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