UDC 667.6:532.135:691.175
DOI: 10.15587/2706-5448.2025.334121

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MODELING THE RHEOLOGY OF COMMERCIAL REACTIVE FIRE-RETARDANT COATING MATERIALS FOR STEEL

Fire protection of steel load-bearing building structures by surface-treating them with reactive fire-retardant coating materials is a crucial factor in ensuring fire safety on national scale. Both, the quality and operational characteristics of such materials, which are the subject of this study, require continuous improvement to reduce the cost of fire protection, as it can constitute a large portion of the overall construction expenditure. The aim of this study was to determine optimal rheological parameters for commercial reactive fire-retardant coating materials that ensure that the material can be applied with the maximum wet coating thickness.

To achieve this aim, the dynamic viscosity (η) was measured using Brookfield viscometer for a set of commercial reactive fire-retardant coating materials that provide fire resistance of at least R120 for steel load-bearing structures. The dependence of viscosity on shear rate (γ) in the range of (2.09–52.25) s^{-1} was modeled using the Casson equation. This allowed for the determination of the main rheological parameters of the studied materials – shear stress (τ, Pa), yield stress (τ_0, Pa), and viscosity at high shear rates (η_∞, Pa · s), which contribute to material's applicability.

With the use of the empirical and calculational data, the approximate viscosity of water-based intumescent coating materials necessary to produce defect-free layer of wet coating on studied surfaces was determined. It was measured by Brookfield viscometer with No. 7 spindle at rotational speeds (30–50) rpm at 20°C, and should preferably be: (30–15) Pa · s, (at 1 mm wet coating thickness); (50–25) Pa · s, (at 1.5 mm wet coating thickness), (80–50) Pa · s, (at 2.0 mm wet coating thickness). These levels of viscosity prevent sedimentation and sagging of the coating during material's application and can serve as reference markers for optimization of industrially manufactured intumescent fire-retardant products.

The obtained results can serve as practical recommendations for manufacturers seeking to improve the rheology of reactive fire-retardant materials in order to increase the wet coating thickness per layer.

Keywords: *fire protection of steel, fire-retardant coating, dynamic viscosity of paint, rheological profile, coating thickness.*

Received: 27.03.2025
Received in revised form: 02.06.2025
Accepted: 23.06.2025
Published: 29.08.2025

How to cite
Vakhitova, L., Drizhd, V., Kalafat, K., Vakhitov, R., Taran, N., Bessarabov, V. (2025). Modeling the rheology of commercial reactive fire-retardant coating materials for steel. Technology Audit and Production Reserves, 4 (3 (84)), 6–11. https://doi.org/10.15587/2706-5448.2025.334121

1. Introduction

In recent years, the global fire protection market displays a noticeable increase in demand for intumescent (reactive) coatings [1]. These materials are used for steel structural elements (columns, beams, metal frames, etc.) to prevent their deformation or loss of load-bearing capacity [2, 3]. According to the reports [4], the global market for reactive coatings has marked up at 975 million USD in 2020 and is projected to reach 1.1 billion USD in 2026 while striking a compound annual growth rate (CAGR) of 3.5% (Fig. 1). Europe, according to [4], is the largest consumer and producer of the intumescent fire-retardant materials, estimated at 490 million USD in 2026, with a CAGR of 3% between 2023 and 2026.

Commercially available reactive fire-retardant coating materials mainly consist of a non-optional intumescent triad: ammonium polyphosphate (APP), melamine (MA), and pentaerythritol (PE) [5, 6]. In addition to these essential components, they will include the following ingredients – pigment (most often, titanium dioxide TiO_2), binding polymer, additional flame retardants and fillers, rheological and reinforcing additives, etc. [1, 7]. Thin-layer coatings are indispensable in mass construction involving steel, which is rather popular for implementing diverse architectural forms or modern design solutions [8]. Nevertheless, the application of reactive fire protection for steel has a significant limitation associated with the high cost of fire-retardant treatment. Over the past 10 years, in response to the constant increase in regulatory requirements for the fire resistance of load-bearing structures, the fire-retardant efficiency of intumescent coatings has nearly tripled. The fire resistance rate (R) has increased from a maximum of 90 minutes to around 300 minutes. Simultaneously, the average thickness of the fire-retardant coating on the steel structure has increases from approximately 2–3 mm (R90) to 10–15 mm (R300). This significantly increases the cost of applying fire-retardant coating on-site, due to the number of layers required to achieve the necessary dry film thickness, which now exceeds 10.

It is possible to reduce the number of layers to be applied for a fire-retardant coating material by improving its rheology. That would allow it to be applied in thicker layers, exceeding 0.7–1.2 mm, which is clearly stipulated in technical documentation supplied by manufacturers. [7] Unfortunately, published sources provide little to none in terms of systematic scientific information on the practical applicability of the rheology of intumescent coating materials [8–10].

Typically, processes and operational characteristics related to commercial coating materials – production, storage, application, etc. –

can be described using shear rate (γ, s^{-1}), shear stress (τ, Pa), yield stress (τ_0, Pa), zero-shear viscosity (η_0, Pa · s), viscosity at given shear rate (η, Pa · s), and at high shear rates (η_∞, Pa · s) [11]. Several studies [12, 13] have examined the correlation between the rheological characteristics of coating materials and various other parameters, e. g. stability of pigmented coating materials during storage and application, their leveling and sagging, splash resistance.

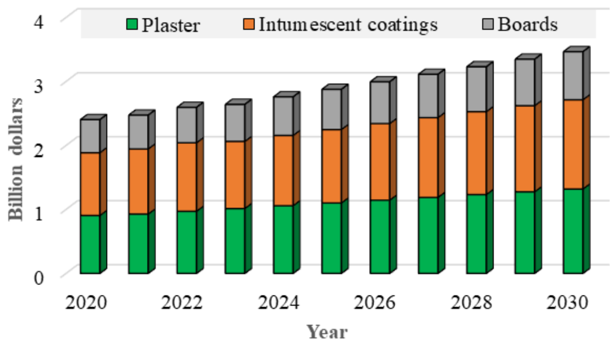

Fig. 1. Market growth dynamic for passive fire-retardant materials in 2020s–2030 [4]

The empirical theological data accumulated for a broad selection of thin-film coatings [14, 15] allows for a generalization [16] of the optimal average values of τ_0, η_0, and η_∞ that would be optimal at particular stages of material's life-cycle – its production, storage, and application (Table 1).

The results presented in the Table 1 can serve as practical recommendations for manufacturers of coating materials and relate to thin-film coatings which contain up to 45% non-volatiles and, in some cases, exhibit Newtonian viscosity [17]. The fire-retardant, highly filled coating materials (70–75% non-volatiles), which are the subject of this study, predominantly demonstrate rheology [7] characteristic of non-Newtonian fluids [18]. However, due to the complete absence of similar viscosity-related data regarding the intumescent coating materials, it is currently impossible to predict optimal rheological parameters for those.

Therefore, *the aim of this research* is to determine the optimal rheological parameters of commercial intumescent fire-retardant coating materials that ensure that coating is applied at maximum possible wet coating thickness.

Table 1

Rheology of thin-layer coating materials [16]

Process	Shear rate γ, s^{-1}	Viscosity, Pa · s	Shear stress, Pa
Storage (low-to-zero shear)	0.1	$\eta_0 > 50$	$\tau_0 > 1$
Applying with brush, no dripping	0	$\eta_\infty > 2.5$	$\tau_0 > 1$
Mechanized application	10^4	$\eta_\infty \sim 0.1–0.3$	$\tau > 0.25$
Drying with leveling and minimal sag	1	$\eta_0 \sim 5–10$	$\tau > 0.25$

To achieve this aim, the following objectives have been set:
– to investigate the dependence of dynamic viscosity (measured by Brookfield viscometer) on shear rate for commercial fire-retardant materials available in Ukraine;
– to design a mathematical model for optimal calculation of the rheological profiles in order to obtain the rheological parameters of the studied materials;
– to develop practical recommendations regarding the rheological parameters of intumescent fire-retardant coating materials that would make it possible to increase coating thickness per one pass during its application.

Achieving the set aim will enable manufacturers of passive fire-retardant materials to adjust the rheology of their products in order to reduce the costs of fire protection at construction sites.

2. Materials and Methods

2.1. Object and hypothesis of research

The object of this research are intumescent fire-retardant coating materials produced by well-known global brands that have been imported into Ukraine over the past five years, as well as the Ammokote MW-120 produced in Ukraine (Table 2). All the studied materials provide R120 fire resistance.

All the materials listed in Table 2 are categorized as water-based intumescent fire-retardant products, which have practically identical compositions, as confirmed by infrared (IR) spectroscopy [5]. The table also presents the calculated number of coating layers (N) required to achieve fire resistance of R120.

The main hypothesis of the research is that optimizing the rheological properties of reactive fire-retardant paints will significantly reduce the number of applications passes while simultaneously ensuring maximum wet film thickness. Implementing this hypothesis is expected to lead to a decrease in costs for the fire protection treatment of steel structures and to increase their competitiveness in the fire protection market.

Table 2

Properties of studied intumescent coating materials[1]

Coating	Non-volatiles content, %	Coating thickness, (mm)[2]	L, (mm)[3]	N[4], equal or over
Ammokote MW-120 (Covlar group LLC, Ukraine)	70 ± 2	5.20	1.20	6
Hensotherm 421 KS (Rudolf Hensel GmbH, Germany)	72 ± 3	4.47	0.70	9
Interchar 1120 (International Paint Ltd, UK)	68 ± 3	4.79	1.00	7
Nullifire – SC801 (Tremco illbruck Trading as Nullifire, UK)	69 ± 2	5.20	1.20	7
Steelguard 651 (PPG Coatings Europe BV, Netherlands)	70 ± 3	5.79	1.00	8
Promapaint SC3 (Enterprise No. F/033, Promat Sp. A, Italy)	71 ± 3	5.54	1.02	7

Notes: [1] Produced from publicly available technical documentation (Ukraine). [2] Dry coating thickness to obtain R120 fire resistance for A_m / V = 200 m^{-1}. [3] Maximum possible single-layer wet coating thickness. [4] Number of layers necessary to obtain R120 fire resistance

2.2. Dynamic viscosity measurements

The measurement of dynamic viscosity was performed using Brookfield DV-III rotational rheometer, as described in [7]. All measurements were performed using spindle 7 (sp N7) at 20°C in a metal cylindrical vessel with a capacity of 1000 cm^3 (diameter 9.5 cm, height 15 cm). The rheometer used for the measurements provides a maximum spindle rotational speed of 250 rpm, which corresponds to a shear rate (γ) of 52.25 s^{-1}. The minimum shear rate (γ) achievable is 0.209 s^{-1}.

The dynamic viscosity η (Pa · s) measured is represented by the average of three independent measurements, with an error margin not exceeding 5%. The mathematical processing of the results was carried out using the least squares method.

2.3. Solid components sedimentation

500 g of each fire-retardant material (Table 2) was placed into a separate plastic container and stored for 6 months in a dark place

at a temperature of 20 ± 5°C. After the storage period was complete, the height of the clear liquid l_1 (mm) that had accumulated above the surface of the settled material was measured.

2.4. Coating sag after application

Coating sag after application was determined visually [7]. Using a metal applicator, a layer of paint with a thickness of 1.0–2.0 mm was applied onto two black-and-white cardboards. Those were then immediately positioned vertically, and after 5 hours, the length of the maximum distance traveled by the applied material on the surface of the cardboard – (l_2, mm the average value obtained from two samples) was measured.

3. Results and Discussions

3.1. Rheology of fire-retardant intumescent coating materials

The rheological profiles of the materials studied (example – Nullifire SC 801, Steelguard 651, Fig. 2) confirm their non-Newtonian behavior with varying degrees of thixotropy and syneresis (size of the hysteresis loop). The label "forward" on the graphs (Fig. 2) corresponds to changes in the composition's viscosity as the rotational speed increases, while "backward" refers to viscosity changes as the rotational speed decreases. The viscosities for all samples decrease with increasing shear rate, and the size of the hysteresis loop between the two graphs ("forward" and "backward") indicates the thixotropic properties of the fire-retardant coating materials.

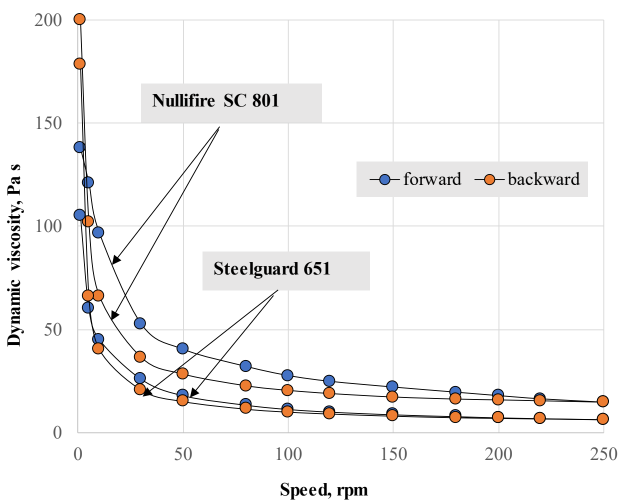

Fig. 2. Dynamic viscosity of Nullifire SC 801 and Steelguard 651 fire-retardant materials

Fig. 3 shows the dynamic viscosity profiles of the water-based coating materials (Table 2), which demonstrate significant variability of rheological behavior, particularly at low shear rates. For example, the dynamic viscosity exhibited by the samples at a shear rate $\gamma = 0.209\ s^{-1}$ ranges from 105 Pa · s for Steelguard 651 to 456 Pa · s for Promapaint SC3.

Currently, it is not possible to compare the viscosity of the compositions (Table 2) obtained in this study with any commercial ones. The technical documentation provided by the respective manufacturers generally does not contain information about the rheological properties of the products. Studies on the viscosity of reactive fire-retardant materials of any composition are absent in the published scientific sources.

Studied materials have been available on the Ukrainian market for the past 5 years and have solid reputation among contractors commercially applying these materials to steel structures. According to the feedback received, the utmost effectiveness in terms of applicability (no runs, sagging, or splattering) and applicational efficiency (wet coating thickness up to 2 mm per one pass) is provided by Promapaint SC3 and Ammokote MW-120. The thinnest defect-free coating was achieved with Steelguard 651 (wet coating thickness 0.7–0.8 mm) and Hensotherm 421 KS (wet coating thickness about 1 mm). These results correlate with the experimentally measured dynamic viscosity of the intumescent fire-retardant materials (Fig. 3).

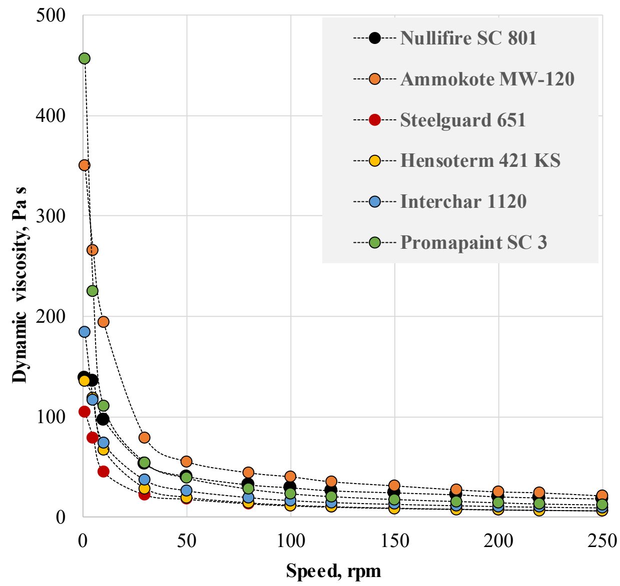

Fig. 3. Dynamic viscosity of fire-retardant intumescent materials

The next step in the study focused on addressing the main research objective – establishing and confirming the relationship between the measured rheology of the coating material before application and its processing characteristics during application. Through mathematical modeling, the key rheological parameters of the studied samples were determined: shear stress (τ, Pa), yield stress (τ_0, Pa), and viscosity at high shear rates (η_∞, Pa · s), which ensure optimal application.

3.2. Use of Casson model in the estimation of rheological parameters

There are several well-known mathematical models describing the flow behavior of non-Newtonian fluids, such as Bingham model [19], Herschel-Bulkley model [20], Casson model [21], and others. The latter (Casson model) is considered the most suitable one for studying paints and coating materials products, industrial polymer-based fluids, synthetic lubricants, and food products [22]. Casson equation used in this model [23] is expressed as follows

$$\eta^{1/2} = \eta_\infty^{1/2} + \frac{\tau_0^{1/2}}{\gamma^{1/2}}. \quad (1)$$

Shear stress τ_0 at shear rate $\gamma \to 0$ is determined according to equation (1), although the necessary parameters can only be measured on the Brookfield viscometer used in this experiment with a significant margin of error. In addition to that, the parameter η_∞ is also calculated, as it can only be reliably measured using ICI-type viscometers [24] at high shear rates ($\gamma > 1000\ s^{-1}$). All the calculations involved in this study were done using viscosity values obtained at spindle rotation of 20–250 rpm, where the measurement error is minimal [22]. Fig. 4 shows the plots of the $\eta^{1/2}$ versus $\gamma^{-1/2}$ for all the studied fire-retardant coating materials (Table 2).

The results of data processing presented in Fig. 4, are provided in Table 3 as a set of rheological parameters for each material. Dynamic viscosity measured at 5 rpm (which corresponds to $\gamma = 1.045\ s^{-1}$) was taken as η_0 (Table 3). At lower spindle rotation rates, measurements on

Brookfield DV-III viscometer cannot be performed reliably, due to high degree of data variation [25].

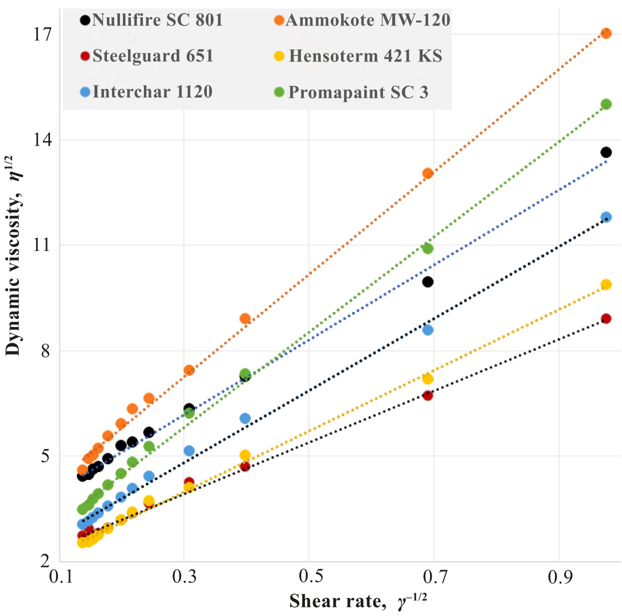

Fig. 4. Linearized viscosity data plots for the studied fire-retardant materials

Table 3

Rheological parameters of studied fire-retardant coating materials

Coating material	η_0, Pa · s	η_∞, Pa · s	τ_0, Pa	R^2	ρ, g/cm^3	*d*, mm
Steelguard 651	89.4 ± 7.6	1.39 ± 0.07	96.8 ± 5.1	0.999	1.37	0.701
Hensoterm 421 KS	128 ± 11	1.21 ± 0.08	138±7	0.996	1.39	1.01
Nullifire SC 801	135 ± 12	5.90 ± 0.28	303±11	0.982	1.35	2.28
Interchar 1120	116 ± 9	4.67 ± 0.11	311±9	0.996	1.39	2.33
Promapaint SC 3	256 ± 21	8.76 ± 0.21	378±14	0.992	1.33	2.92
Ammokote MW-120	265 ± 19	6.25 ± 0.40	364±16	0.994	1.34	2.77

Correlation and regression analysis performed on the data from Fig. 4 revealed high values of regression and correlation factors (R^2 = 0.982–0.999) for all of the presented linearized plots of $\eta^{1/2}$ versus $\gamma^{-1/2}$.

The yield stress (τ_0, Pa) presented in Table 3 is a critical parameter which characterizes the quality of coating material during its application on metal surfaces. It is especially important to estimate the sag, which generally increases with the thickness of the coating layer. For vertically oriented surfaces, the maximum allowable thickness of a single wet coating layer (*d*, mm) is calculated as [7]

$$d = \frac{\tau_0}{\rho \cdot g}, \quad (2)$$

where ρ (g/cm^3) – material's density; g = 9.81 m/s^2 – gravitational constant.

The wet coating thicknesses (*d*, mm) that can be applied in a single layer, as calculated by formula (2), are presented in Table 3. Based on these results, the studied materials can be nominally divided into three groups:

– those with a single wet layer thickness of about 1 mm (Steelguard 651, Hensoterm 421 KS);
– those that can be applied with a single wet layer thickness of about 2 mm (Nullifire SC 801, Interchar 1120);
– those with permeable single wet layer thickness exceeding 2 mm (Promapaint SC3, Ammokote MW-120).

This classification coincides with the empirical results provided by Ukrainian contractors specializing in commercial application of fire-retardant coatings at construction sites, who have extensive experience in using the studied materials. Naturally, these calculated values of thickness, *d*, should be understood and used as indicative tendencies only. In real-world scenarios, the wet coating thickness may be limited by other factors such as the coating's tendency to cracking and crater formation, desirable drying time, application method, geometrical complexity of steel structure and its height, etc.

3.3. Practical recommendations regarding the optimal rheology of intumescent fire-retardant coating materials

Manufacturers of intumescent fire-retardant coating materials face two main challenges in determining the optimal rheological parameters of their products:

– what dynamic viscosity indicators should the paint have to ensure storage without sedimentation and defect-free application of a coating when applying at maximum single-layer thickness using airless spray equipment;
– how to quickly and cost-effectively measure dynamic viscosity of the product to assess its quality post-production.

Table 4 presents the results of experimental studies on assessing sedimentation and sag during coating application, which were conducted according to methods 2.2–2.3 for the investigated commercial coating materials.

To validate the experimental data obtained in laboratory setting, full-scale application of the studied materials onto steel surface was carried out. The maximum wet coating thickness (d_1, mm) that could be applied in one pass without sagging or dripping, and forming a defect-free coating (no cracking or crater formation during drying), was determined. Coatings were applied on vertically positioned 100 × 100 cm steel plates, using Graco Mark V high-pressure airless spraying unit. The obtained values of d_1 are presented in Table 4.

Based on the results in Table 4, it can be concluded that sedimentation of solid components was not observed in any of the studied materials, and the measured values of sag (l_2, mm) are consistent with the calculated permissible maximum thicknesses (*d*, mm), presented in Table 3. A comparison of the number of required coating layers (N_1) calculated using the d_1 (Table 4) with the corresponding indicator *N* from Table 1 was carried out. The results show that increasing the dynamic viscosity of the paint up to the recommended values will reduce the number of applied layers by up to 40%. This can significantly lower the cost of the fire protection treatment.

Table 4

Sedimentation and sagging of studied fire-retardant materials during their application

Coating material	Sedimentation, l_1, mm	Sag, l_2, mm – Wet coating thickness 1.0 mm	1.5 mm	2.0 mm	2.5 mm	d_1, mm	N_1[3], equal or over
Steelguard 651	0	0	15	56	74	1.0	8
Hensoterm 421 KS	0	0	5	22	51	1.2	5
Nullifire SC 801	0	0	0	0[1]	12	1.5	5
Interchar 1120	0	0	0	0[1]	14	1.5	5
Promapaint SC3	0	0	0	0	0	2.5	3
Ammokote MW-120	0	0	0	0	0	2.5	3

Notes: [1]Cracking was observed. [2]Airless spraying: pressure on the supplied material – 180–220 bar; inner diameter of the hoses – 8–10 mm. [3]Calculated using d_1 parameter

The conducted computational and experimental studies allow the determination of the average limiting values of η_0, τ_0 and η_∞, which characterize optimal rheology of intumescent fire-retardant coating materials (Table 5).

Table 5

Rheological parameters of fire-retardant coating materials to be applied at maximum permissible single-layer wet coating thicknesses[1]

Process in the life-cycle of a coating	Shear rate γ, s^{-1}	Viscosity, Pa · s	Shear stress, Pa
Storage (low to no shear)	0.1	$\eta_0 > 90$	$\tau_0 > 100$
Mechanized application at coating thickness of 1 mm 1.5 mm 2.0 mm	10^4	 $\eta_\infty > 1.2$ $\eta_\infty > 4.5$ $\eta_\infty > 6.0$	 $\tau_0 > 100$ $\tau_0 > 300$ $\tau_0 > 350$
Minimal sag at 2.0 mm wet coating thickness	1	$\eta_0 \sim 200$	$\tau_0 > 300$

Note: [1]data measured on Brookfield viscometer with Sp7 spindle, at 20°C

In order to control the quality of an intumescent fire-retardant coating material in post-production, it is sufficient to measure its dynamic viscosity using Brookfield viscometer at a fixed spindle rotational speed. Table 6 presents the correlation between said measured values and the recommended maximum wet coating thickness at which materials can be applied.

Table 6

Dependence of wet coating thickness on the recommended dynamic viscosity values

Wet coating thickness, mm	Dynamic viscosity, η, Pa · s	
	30 rpm	50 rpm
1	20–30	15–20
1.5	35–50	25–40
2.0	60–80	50–60

It should be clarified that the dynamic viscosity values presented in Table 6 are approximate value ranges. However, the fact that they were obtained through the analysis of the rheological properties of commercial fire-retardant materials from globally recognized manufacturers, and not the experimental batches of newly-developed formulations, greatly enhances the applied value of the results. Not only is this study the first one to apply mathematical modeling to calculate the rheological parameters of intumescent fire-retardant coating materials, but a simple and effective algorithm for controlling material's rheology was offered to manufacturers of passive fire protection products. This can help with quality control and enable the production of high-quality material that can be applied to metal surfaces with a wet coating thickness of 1.0–2.0 mm. And it is precisely in these recommendations that the practical significance of this study lies. By applying those, a significant reduce of the cost of fire protection of load-bearing structures can be achieved.

Under the martial law conditions, this fact is especially important. Reactive intumescent paints that provide high fire resistance are the material of choice for the reconstruction of critical infrastructure and the construction of shelter structures for the energy and defense industries. Improving the properties of these materials by reducing both cost and time required for application is of critical importance for the rapid restoration of damaged infrastructure.

One limitation of this study is the lack of comparative data on the application of the tested fire-retardant coatings in industrial conditions. To obtain more accurate rheological data (Tables 5 and 6), further experimental studies are scheduled, this time involving a larger number of test subjects and more extensive tests on the application of fire-retardant intumescent coatings on large-scale steel structural elements.

4. Conclusions

This study presents a systematic investigation of the variation in dynamic viscosity of commercial intumescent fire-retardant materials, measured using a Brookfield viscometer within the shear rates of $\gamma = 0.209–52.25\ s^{-1}$. It was shown that the examined materials exhibit non-Newtonian flow with varying degrees of thixotropy and syneresis. The viscosities of samples produced from similar types of fire-retardant materials differ by a factor of 4–5 at shear rates $\gamma = 2.09–20.90\ s^{-1}$.

By applying mathematical modeling using the Casson equation, with high correlation factors (R^2 = 0.982–0.999), the key rheological parameters of the materials – shear stress, yield stress, and viscosity at high shear rates were determined. Comparison of the calculated parameters with the experimentally measured values of sedimentation during storage and sag of the coatings during their application made it possible to identify optimal rheological characteristics that result in defect-free application of the materials at various wet coating thicknesses.

Both, the calculational and experimental results obtained can be utilized as practical guide for improving the rheology of intumescent fire-retardant coating materials to increase the single-layer wet coating thickness. Preliminary calculations indicate that increasing the dynamic viscosity of the fire-retardant composition to the recommended values could reduce the necessary number of coating layers by up to 40%. This would significantly lower the cost and shorten the time required to finish works associated with fire protection of building structures.

Conflict of interest

The authors declare that they have no conflict of interest in relation to this research, including financial, personal, authorship, or any other nature that could affect the research and its results presented in this article.

Financing

Budget financing of the NAS of Ukraine.

Data availability

The manuscript has no associated data.

Use of artificial intelligence

The authors confirm that they did not use artificial intelligence technologies when creating the presented work.

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