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PRELIMINARY REPORT ON IGNITIBILITY OF COMBINED PRO-ECOLOGICAL WATERPROOFING AND FIRE RETARDANT COATINGS FOR PAPERBOARD IN ARCHITECTURAL APPLICATION

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Abstract
Both fire and water protection are crucial for the safety and usability of paper-based products applied as building components. The presented study investigates the possibility of combining environmentally-friendly fire retardants with oil- and wax-based waterproofing coatings on paperboard for architectural applications. The proposed impregnation technique can be used as protection for paper-based temporary and emergency structures, or as part of the protective system for building envelopes of permanent structures. The fire retardants selected for the tests were diammonium phosphate and a mixture of borax and boric acid in a 1:1 ratio. Single-flame ignitability tests were performed on the impregnated specimens to assess the fire performance of specimens with fire impregnation, waterproofing impregnation and both. A Life Cycle Assessment analysis was performed for fire-retardant paperboard. The study has shown that the application of layered fire and waterproofing treatments on paperboard components is possible and leads to a significant reduction in flammability compared to untreated and only waterproofed specimens.

Keywords: **Paper in architecture; Fire retardant; Paperboard; Ignitibility test; Life Cycle Assessment.**

1. INTRODUCTION

With the ongoing degradation of the natural environment, the demand for environmentally friendly and natural building materials is constantly increasing. One of these materials is paper – a bio-based, renewable, cellulosic material, suitable for recycling (up to seven times) and biodegradable [1]. As an exceptionally common material used in many areas of life, it is widely available, mass-produced and cheap. However, the most important disadvantage associated with paper is its sensitivity to water and fire. Like most cellulosic materials, paper is combustible and fire protection is crucial for its safe application in architecture.

Japanese architect Shigeru Ban, who used structures made of paper tubes, was a precursor of paper application in modern architecture. Paper has also been used in temporary and emergency architecture, including various shelters for victims of war or natural disasters [2–4], and even permanent buildings such as the Westborough Primary School [5] or Wikkelhouse [6]. Corrugated cardboard, honeycomb panels and cellulose fibre are used as thermal and acoustic insulation [7–9] as well as to form building envelopes and partitions [3,10], while various types of solid paperboard can be applied as protective and strengthening layers [5]. Furthermore, paper tubs and L-shapes are used as structural elements, e.g. columns, beams and rods [10].

1.1. Fire safety

One of the most crucial difficulties to overcome when designing with paper is fire safety – as with most bio-based building materials, paper is easily flammable. However, the combustibility of the material depends on the type of paper-based products used. While products composed of thin sheets of paper with air cavities in between, such as corrugated cardboard and honeycomb panels, ignite and spread fire quickly, thick layers of paperboard present certain fire protection properties. The outer layers of thick paperboard chars, forming a protective layer on the rest of the material – a similar mechanism can be observed in wood [1].

Despite this mechanism, additional fire retardant protection of paper-based building elements is necessary to ensure users safety. Although the legal regulation varies between countries, in most cases it is required that building elements are non-combustible or fire-resistant.

There are three main approaches to reducing the flammability of cellulosic, including paper-based elements.

- Material additives during production, e.g. phosphorus compounds, that provide effective long-term protection. However, the presence of retardants, e.g. inorganic salts, in the material may interfere with elements bonding and have an abrasive effect on cutting tools [11].
- Fire retardants applied to the end product, including immersion in water-based solutions and coatings, provide a uniform, seal coating on the surface of the final product. This technique usually requires less impregnating agent and facilitates recycling due to limited penetration into the material, but is more sensitive for air humidity changes and may increase the roughness of impregnated surfaces [12,13]. Some coatings can also provide water protection.
- Layering with other, fire-resistant materials, e.g. metal sheets or mineral boards facilitate the recycling process due to the possibility of separating materials. Moreover, it can provide additional protection against water and mechanical damage. However, finishing layers imply increased weight and the need for additional joins between materials.

In several paper-based designs and already completed buildings, a variety of fire retardant techniques has been used (see Table 1).

1.2. Fire retardants

The role of fire retardants (FR) is to increase the fire safety of the impregnated material by reducing its contribution to a possible fire. This effect can be achieved by delaying the moment of ignition of the material and reducing the combustion intensity and the spread of the fire. Several mechanisms of disruption of the combustion process at different stages can be distinguished.

- Formation of coatings that slow down the transport of heat to the protected material, delaying the moment of ignition;
- formation of a char layer on the surface of the material in the initial stages of combustion, which slows down heat transfer;
- emission of non-combustible gases at high-temperature conditions (e.g. water vapour);
- endothermic degradation of FR compounds, that reduces the temperature of the material.

Besides ensuring fire safety, FR should be harmless for people and the natural environment during normal use of the treated objects, as well as during combustion [16].

A large variety of chemical compounds can be applied as fire retardants, especially inorganic salts of phosphorus, boron, chlorine or sulfur. Furthermore,

Table 1.
Fire protection techniques applied in paper-based structures

Reference	Structure and authors	Paper-based material used	FR technique
[5]	Westborough Primary School Cardboard Building by Cottrell & Vermeulen Architecture	Honeycomb panels and paperboard	Layering – fibre-cement board (exterior) and FR cellulose pinboard (interior)
[14]	Corrugated Cardboard House by T. Konishi and M. Tamura	Corrugated cardboard	Coatings – urethane resin mixed with a combustion-resistant agent
[15]	CASTE wall system by Ozlem Ayan	Corrugated cardboard	Layering – steel sheets
-	Transportable Emergency Cardboard House gen 4 by Jerzy Łątka and Agata Jasiołek	Corrugated cardboard, honeycomb panels	Layering – steel sheets (exterior)
[4]	Paper House by BAMP! project team form TU Darmstadt	Paperboard, corrugated cardboard, honeycomb panels	Additives – FR paperboard (phosphorus compounds added during paperboard production)

organic substances, like urea and melamine can also be used [16]. In most commercial FR several compounds are combined to employ various retardancy mechanisms and achieve more effective protection. In the last years, the use of some FR has been either discouraged or prohibited due to their toxicity and harmful effects on people's health and the environment. The widely used halogenated FR (especially brominated FR) have been proven to emit toxic halogenated compounds during ignition, that accumulates e.g. in crop plants, penetrate the human body and cause carcinogenic effects [17]. Therefore, it is crucial to use FR that are not only effective but also safe. Besides experimental studies on the use of organic compounds like tannic acid [18], guanidines [19, 20] or plant extracts as FR, e.g. green coconut shell or banana pseudostem [21, 22], widely used groups of non-toxic FR are borates and phosphates.

Borates are one of the most efficient and well-studied FR for wood and other cellulosic materials, such as bamboo [17, 23] or paper [24]. They are also incorporated as a FR additive in the production of paper-based composite materials or laminates [25–27] and into commercial cellulose-fibre thermal insulation [28]. Borates can also act on these materials as a protection against insects, bacteria and fungi [29]. The two most widely-used FR compounds are boric acid and borax (sodium borate), both non-toxic and cheap. Both of them act by physical mechanism, forming a glassy protective coating on the impregnated surface, as well as chemical, by promoting a char formation and releasing water vapour at high temperature [17, 30, 31]. However, as described by Yu et al., borax presents better performance in slowing down the heat release while boric acid results in a lower amount of total heat released. Therefore, to achieve the best, synergic results the study suggested the use of both compounds in a ratio of 1:1 [23], which also enables solutions of higher concentration to be produced. The differences between the compounds are also noticeable in the degree of resistance to humidity. While boric acid is resistant to water vapour, changes in humidity may cause efflorescence on surfaces coated with borax [11]. Moreover, none of the borates has the ability to chemically bond with cellulose, which may have a negative effect on the durability of the protection, however, allows easy separation of the impregnating agent during the recycling process.

A wide range of Phosphorus-containing compounds can be used as a FR, including organic compounds (e.g. Trialkyl Phosphate or Phosphoramide) and inorganic ones [32, 33]. The most popular are inorganic salts of Ammonium Phosphate and Diammonium Phosphate. Like boron compounds, phosphates are non-toxic and soluble in water, however, they are more sensitive to changes in air humidity [11]. The main fire retardancy mechanism of phosphates in the condensed phase involves the formation of a non-combustible char layer on the impregnated surface [17]. Moreover, the retardants can also inhibit flame in the gas phase [34]. Like borates, inorganic phosphorus salts do not chemically bind to cellulose.

Most of the abovementioned FR are water-soluble, hygroscopic and sensitive to air humidity. Paper elements are also particularly sensitive to moisture, therefore it is especially important to protect impregnated paper from contact with water. This can be achieved by cladding the element with other materials, such as membranes and metal sheets or providing an additional layer of waterproof impregnation.

1.3. The aim of the research

The presented article investigates the use of environmentally-friendly inorganic boron- and phosphorus-based FR on paper-based building elements, as well as the combinations of these FR with biodegradable water impregnates. Although the mechanism of fire retardancy on cellulosic materials is already well known, there is no research available regarding additional water protection of the impregnated elements and their architecture-related applications. As water impregnates tend to increase the combustibility of the treated materials, while fire retardants usually increase their water absorbency, the combination of both types of protection is essential for its use in building elements. Therefore, the main objective of the research is (i) to verify the possibility of layering FR and oil-based waterproofing impregnantes on paperboard, (ii) to test their ignitability when exposed to a single flame, (iii) to compare the effects obtained using FR, water retardant and both, (iv) to observe changes in the impregnated surface over time, and (v) to assess the environmental impact of the FR. The results indicate potential new ways to protect paper-based building components in an environmentally-friendly and safe way.

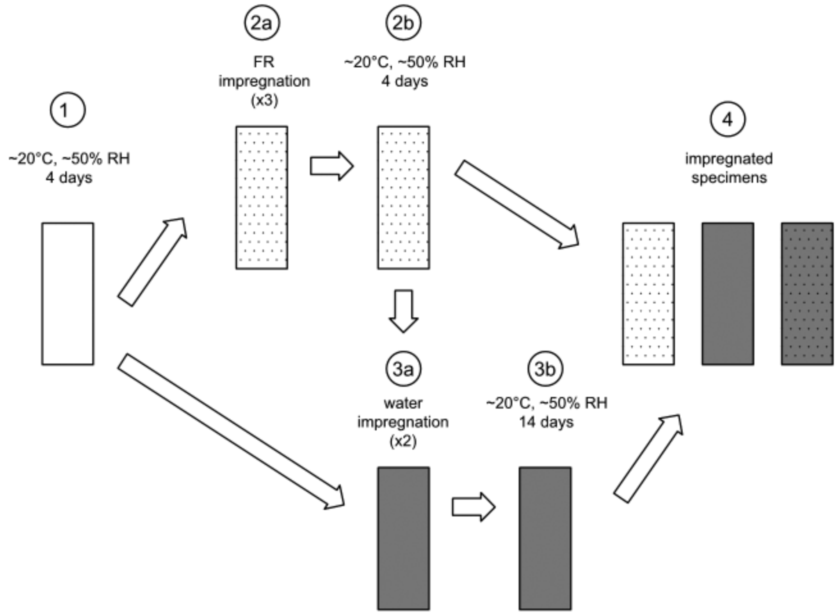

Figure 1.
Impregnation process

2. METHODOLOGY

The adopted methodology is based on single-flame material ignitibility test with specimens observation, followed by Life Cycle Assessment analysis. The experimental part of the research was divided into five stages.

- Bibliographical research and materials selection;
- specimens preparation and observation;
- ignitibility test;
- data analysis, including the calculation of charred areas;
- a year-long observation of specimens.

2.1. Materials

The tests were carried out on rectangular 90 × 250 mm pieces of three-layer glued paperboard made of 100% recycled paper, with an area density of 1845 gsm (Zing, EskaBoard paperboard, 3 mm thick).

The raw materials for the preparation of the FR solutions were obtained from Warchem Company, a local manufacturer of laboratory reagents from Poland. Three types of chemical compounds were used:

- borax (sodium borate) – $Na_2B_4O_7$ x $10H_2O$;
- boric acid – H_3BO_3;
- diammonium phosphate – $(NH_4)_2HPO_4$.

Water impregnation techniques were selected based on previous research regarding biodegradable paper impregnation [31]. The two types of coatings, that had presented the best performance in water-resistant tests were used in the presented research:

- a composite coating of linseed oil varnish (Dragon, Linseed oil varnish) and wood wax – a mixture of beeswax, plant-based and synthetic waxes (ICA Poland, Colorit, Paste wood wax);
- a homogenous layer of wood oil – a mixture of natural oils with the addition of solvents and waxes (Rust-Oleum, Timberex, Hard wax oil).

2.2. Specimens preparation

Four groups of paperboard specimens were tested:

- with only water impregnation (Group 1),
- with only fire impregnation (Group 2),
- and with both types of impregnation (Group 3),
- control specimens of paperboard with no coating (Group 0).

For each type, three identical specimens were prepared. Two FR retardant solutions were prepared by dissolving reagents in deionized water with a temperature of 50°C. The borates solution (BA-BX) was prepared from boric acid, borax and water in the ratio of 1:1:8, and phosphorates solution (DP) from diammonium phosphorate and water in the ratio of 3:7. The chosen concentrations result from the solubility of the used raw materials.

Table 2.
Types and designations of specimens

Specimen designation	Fire retardant used	Waterproofing impregnant used
0-X-X	-	-
1-X-LW	-	linseed oil varnish + wood wax
1-X-O	-	wood oil
2-B-X	borax + boric acid (BA-BX)	-
2-DP-X	diammonium phosphorate (DP)	-
3-B-LW	borax + boric acid (BA-BX)	linseed oil varnish + wood wax
3-DP-LW	diammonium phosphorate (DP)	linseed oil varnish + wood wax
3-B-O	borax + boric acid (BA-BX)	wood oil
3-DP-O	diammonium phosphorate (DP)	wood oil

The impregnation was done manually, using a brush. Firstly, fire-impregnates were applied in three layers and the specimens were air-dried for four days. The applied amounts allowed to achieve a saturation of approximately 200g/m^2 for DP and 167g/m^2 for BA-BX. Secondly, the water impregnants were applied in two layers, and the specimens were conditioned for another 14 days in normal conditions. Types of specimens are described in Table 2 and the impregnation process is illustrated in Figure 1.

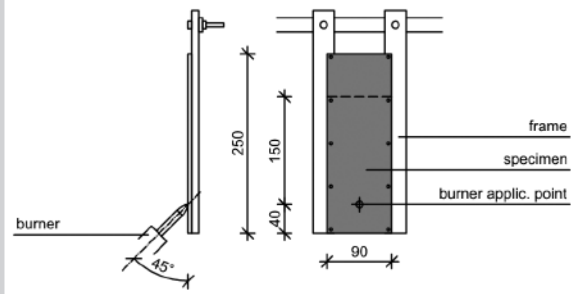

Figure 2.
Ignition test setup

2.3. Ignition tests and data analysis

The testing methodology was developed based on a single-flame source test described in ISO 11925-2 standard [35]. The tests were carried out in normal condition (approx.. 20°C, 50%RH) with a standardized flame of the propane-butane burner, corresponding to the size of the match flame. The specimens were mounted in a frame, and the flame was applied to their surface at an angle of 45 degrees, 40 mm above the bottom edge of the specimen (see Figure 2). After 30 s of exposition, the flame was extinguished, and the specimen was observed for another 30 s. After 60 seconds from the start the test was finished and the fire was extinguished if necessary. During the tests, specimens were observed in terms of ignition, fire maintenance and flames reaching 150 mm above the fire application point.

After the test, specimens were photographed and the ImageJ software was used to analyze the shapes and calculate the areas of the charred surfaces. Lastly, the specimens were left for a year-long observation for changes in appearance, such as colour change, unintentional interaction between impregnates or possible crystallization of flame retardants on the surface of the specimen.

2.4. LCA analysis of fire retardants

The Life Cycle Assessment analysis was performed in accordance with ISO 14040 and ISO 14044 standards [36, 37], using OpenLCA software with ecoinvent 3.8 database and ReCiPe LCIA method. The analysis was conducted for paperboards coated with BA-BX mixture, DP and uncoated, for a functional unite of 1 m^2. The analysis covers the production phase (cradle to gate approach) and the end-of-life phase, with a 90% recycling rate assumed for paperboard. The use phase was excluded as the application of FR paperboard in building elements is not fully addressed yet, therefore there is a lack of reliable data in this area.

3. RESULTS AND DISCUSSION

3.1. Ignition tests

Significant differences in the ignition process of the four tested groups of specimens were noticed (see Table 3). The unimpregnated paperboards in Group 0 showed ignition and incandescence requiring extinguishing at the end of the test. These were the only specimens in which burning occurred through the entire thickness of the material. Specimens from Group 1 (only water impregnation) ignited and main-

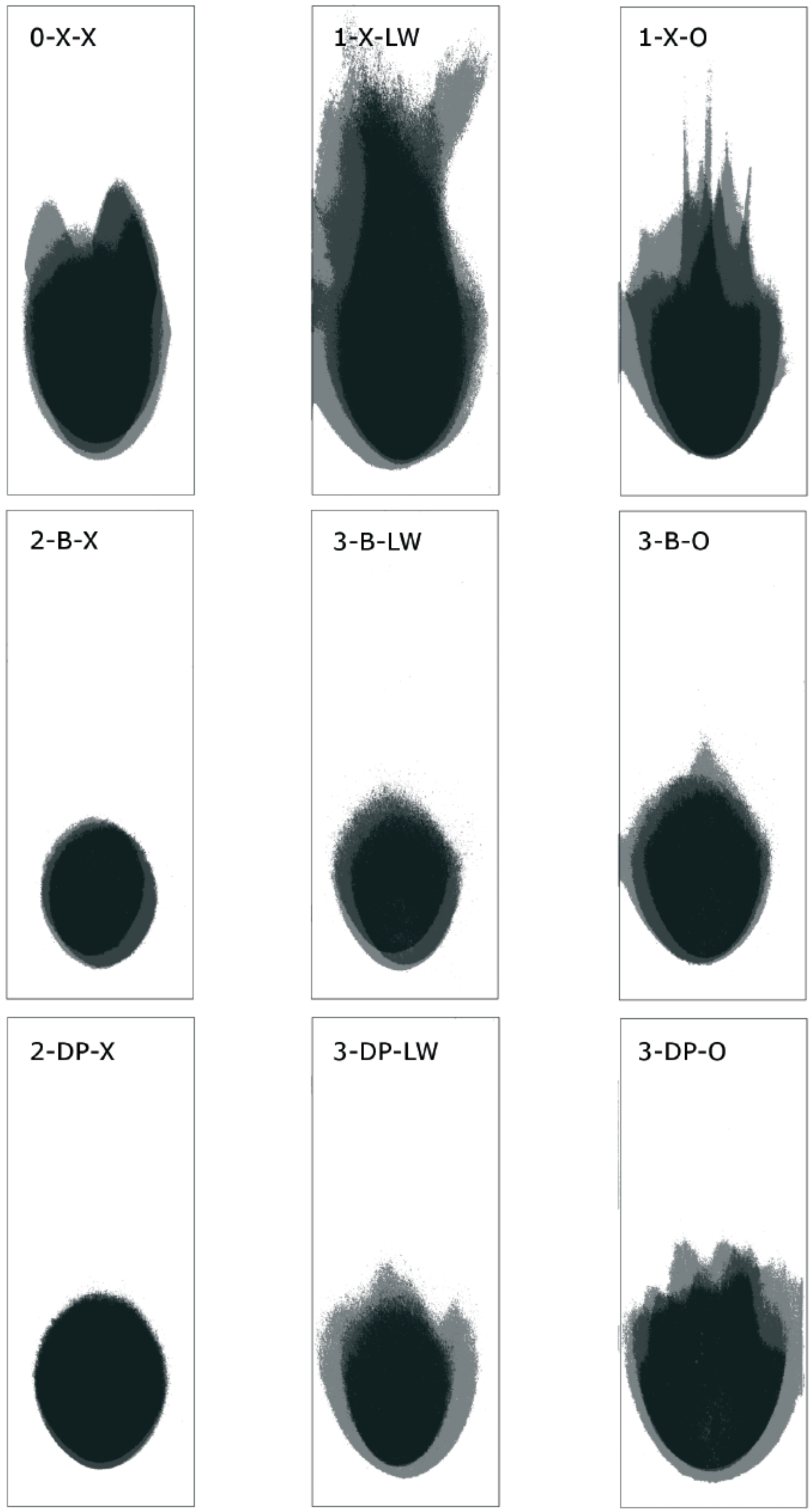

Figure 3.
Charred areas on the tested specimens – superimposed images from three trials for each type

Table 3.
Results of the ignition test

specimen	ignition	flame reaching 15 cm	maintaining the fire	Charred area (cm^2)
0-X-X	yes	no	yes	67.17
1-X-LW	yes	yes	yes	113.01
1-X-O	yes	no	yes	66.52
2-B-X	no	no	no	31.05
2-DP-X	no	no	no	45.71
3-B-LW	no	no	no	37.11
3-B-O	no	no	no	48.69
3-DP-LW	no	no	no	43.91
3-DP-O	yes	no	no	66.75

tained the fire, which spread across the surface of the paperboard, resulting in the combustion of a large area. Specimens impregnated with a combination of linseed oil varnish and wax (1-X-LW) were the only ones with flames reaching the height of 15 cm, and the ones with the largest charred area. As expected, specimens from Group 2 (only fire impregnation) presented full fire resistance with no ignition and an uniformed, oval-shaped charred area.

In Group 3 specimens, a significant reduction in flammability was observed compared to Group 1, although the results were slightly worse than in Group 2. None of the specimens maintained fire when the flame was removed, although a small ignition (not reaching a height of 15 cm) did occur for the phosphate-coated specimens (3-DP-O). The spread of the flames was noticeably smaller than in Group 1, which was also reflected in a limited charred area (see Figure 3).

3.2. Surface observation

No problems during the impregnation process were observed – precoating with fire retardants did not hinder water impregnation – and no changes in the appearance of the specimens occurred shortly after the impregnation. However, after the year-long exposure to light and changing air humidity, significant changes were noticed in some of the specimens (see

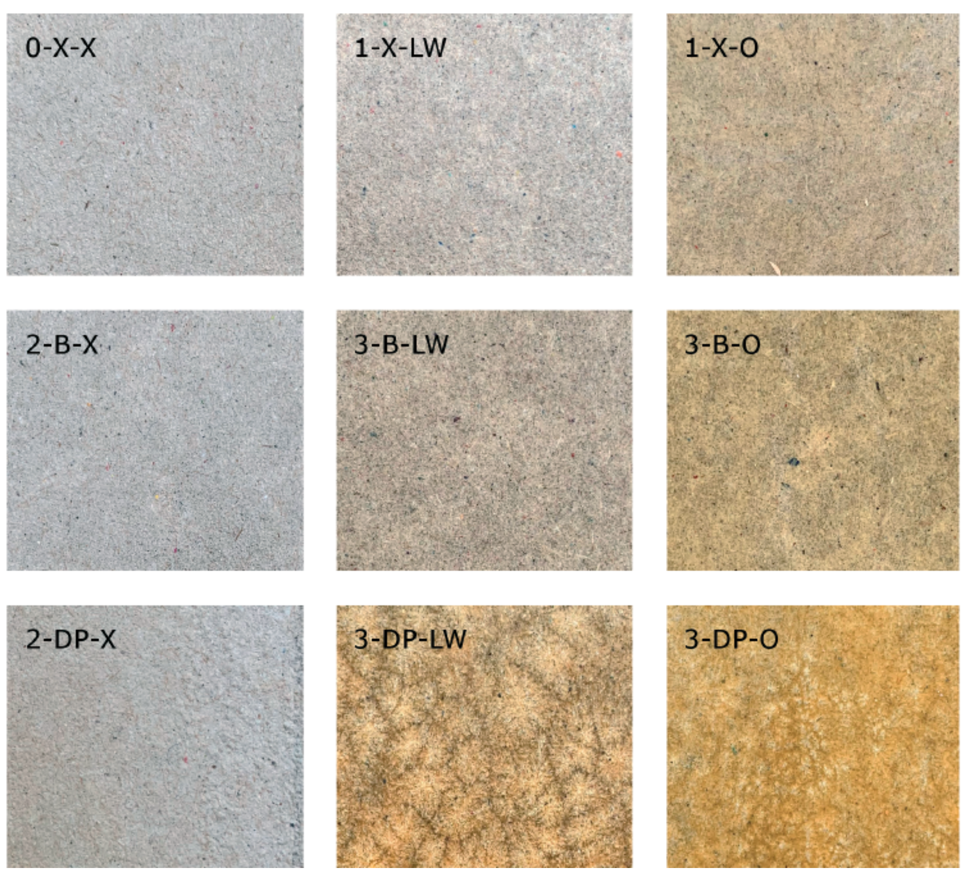

Figure 4.
Surfaces of the specimens after a year-long observation period

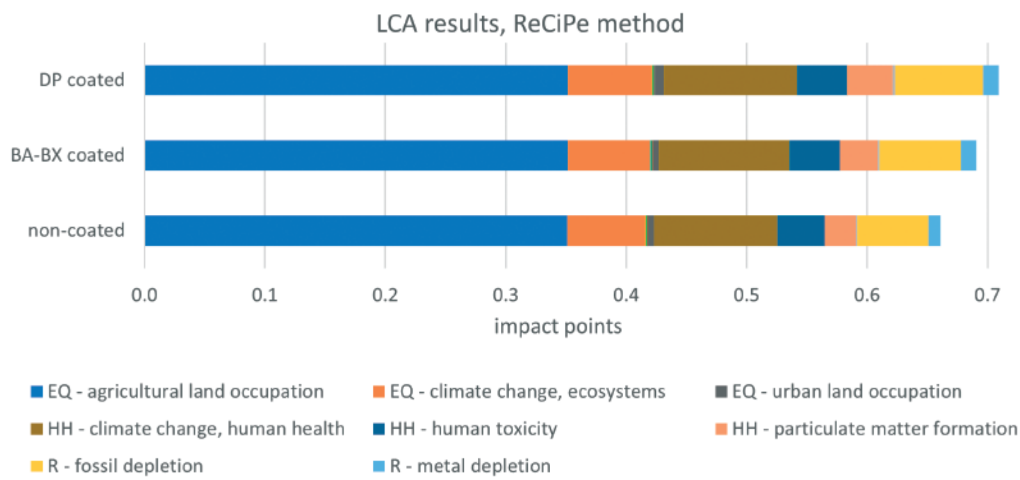

Figure 5.
LCA results for FR paperboards.
Note: for legibility, only the most important impact categories have been included in the description

Table 4.
Normalized environmental impact of 1 m^2 of paperboard

Area of protection	Midpoint impact category	Unit	Type of specimen		
			non-coated	BA-BX coated	DP coated
Environment quality	agricultural land occupation	10^{-1}	3.51	3.51	3.52
	climate change, ecosystems	10^{-2}	6.47	6.83	6.98
	freshwater ecotoxicity	10^{-5}	5.27	5.88	6.09
	freshwater eutrophication	10^{-5}	9.66	1.04	1.04
	marine ecotoxicity	10^{-6}	9.43	1.08	1.09
	natural land transformation	10^{-3}	1.28	1.51	1.60
	terrestrial acidification	10^{-4}	1.18	1.40	2.43
	terrestrial ecotoxicity	10^{-4}	5.12	5.21	5.59
	urban land occupation	10^{-3}	4.85	5.11	7.40
Human health	climate change, human health	10^{-1}	1.02	1.08	1.10
	human toxicity	10^{-2}	3.96	4.20	4.16
	ionising radiation	10^{-5}	9.17	9.65	1.01
	ozone depletion	10^{-5}	1.01	1.10	1.18
	particulate matter formation	10^{-2}	2.55	3.15	3.78
	photochemical oxidant formation	10^{-3}	1.08	1.29	1.90
Resources	fossil depletion	10^{-2}	5.93	6.76	7.29
	metal depletion	10^{-2}	1.02	1.28	1.29

Figure 5). On all of the paperboards impregnated with DP efflorescences are visible, while BA-BX specimens remain free from such changes. Moreover, all the specimens with water impregnation have developed a yellow tinge, thaw was especially visible on paperboards with wood oil treatment and DP pre-coating.

3.3. Life Cycle Assessment

The conducted LCA analysis showed a limited impact of both discussed FR techniques on the paperboard component. The BA-BX coating increased the overall environmental impact of the element by 4% in relation to uncoated specimen, and the DP coating – by 7% (see Figure 5). In each case, the highest impact (approx. 50%) was associated with agricultural land occupation, due to the use of land for the cultivation of cellulosic plants. The most significant changes between LCA scenarios were observed in the areas of "climate change, human health", "particulate matter formation" and "fossil depletion" (see Table 4).

3.4. Application in building elements

The results obtained, confirmed the validity of using inorganic FR while coating the cardboard with oil- and wax-based waterproofing agents. Coating of borates mixture was more effective in inhibiting ignition than diammonium phosphorate solution, and also more resistant to coating degradation over time. Moreover, it presented a slightly lower environmental impact in the LCA analysis. In the case of water impregnates, wood oil was less combustible than a mixture of linseed varnish and wax. The former, according to previous studies, is also more effective in protecting against water [38]. Therefore, on the basis of the data obtained, the best effect in terms of both water and fire protection and durability of the coating can be achieved by coating paperboard with a mixture of boric acid-borax and wood oil (testing specimen 3-B-O).

The presented technique can be applied to building elements that require moderate water and fire protection. It should be noted that coated paperboard cannot form a building element on its own, but it needs to be combined in sandwich elements with different materials that provide structural stability, e.g. paper honeycomb panels, and corrugated cardboard. In such composition coated paperboard can significantly increase fire, microbes and mechanical resistance of the elements, as well as its acoustic properties if needed. Depending on the type of composite, it can be used as building envelope, building partition, or structural element of small buildings.

The most common application of the proposed technology is in temporary and emergency structures. Paper-based elements can be sustainable alternatives for conventional materials, as their limited life span is more suited for temporary architecture. Still, regarding their life span, temporary architecture has to present a certain level of fire resistance, to ensure users' safety. The proposed impregnation is a pro-ecological alternative to traditional varnishes and foils used in temporary buildings, in which it may act as a single layer of protection. Moreover, the proposed coating can be a part of a double protection system for the envelopes of permanent buildings, in combination with non-combustible finishing materials such as metal sheets or mineral boards. The presented technique can also be used as additional protection between internal layers of the paper-based building envelope, hindering fire penetration into the material in case of fire [39]. Furthermore, paper-based elements are already used in lightweight building partition systems, where incorporation of coating may reduce the need of using finishing materials with a higher environmental burden (e.g. fire retardant plastic).

Architectural boards that are currently used in applications similar to those proposed for paper-based ones are various types of wood-based products (e.g. soft fibreboard), thin metal sheets, plastic panels (e.g. low-density polyethene or polylactic acid) or laminates, such as HPL (that also includes paper) or glass fibre-resin laminate. Most of these existing products present higher mechanical strength but, on the other hand, significantly higher environmental impact. Therefore, product selection should be tailored to the requirements of the specific application.

4. SUMMARY AND CONCLUSIONS

This article describes a new environmentally-friendly technique of combined protective coating for paperboard – with boron and phosphorus compounds as fire retardants and biodegradable oil- and wax-based agents as water agents. Results obtained in ignitability tests confirmed the validity of this technique - precoating with flame retardant reduced the naturally high combustibility of oil-based impregnates and double-coated specimens showed a significant reduction in ignitability in comparison with uncoated ones. The use of a combination of borax and boric acid is recommended due to its high flame retardancy (despite the lower concentration of the flame retardant), lower environmental impact and higher stability in changing air humidity.

In spite of the promising results, the application of the proposed technique to building components requires further research. It is necessary to verify the possible influence of the precoating with FR on the effectiveness of the waterproofing agents, and the behaviour of the impregnated elements over a longer period of time. The described research is a stage in a longer project focusing on paper-based building envelopes. In the next steps, the design architectural board will be combined with various cores and tested on a larger scale.

Nevertheless, the proposed solution offers an alternative to traditional protective coatings for paper-based building partition and envelope elements. It could be particularly useful for emergency and temporary architecture, where fire protection is sometimes omitted, and additional impregnation could significantly increase the safety of these structures.

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