

The effectiveness of impregnation of building materials of cellulose origin and its impact on selected properties

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Abstract. One of the biggest challenges facing a designer of paper structures is its low resistance to moisture and water. Paper is a hydrophilic material that absorbs moisture from the outside. This causes the hydrogen bonds between the cellulose molecules to loosen and as a result a rapid decrease in strength parameters. In order to be able to use paper as a construction material, there is a need to select and evaluate the effectiveness of the appropriate impregnant, as well as to know its impact on the mechanical properties of the impregnated paper. The paper analyzes the effect of the use of various impregnations, including wood oil, yacht lacquer, and fire-retardant agent impregnation, on the tensile strength of several types of cellulose-derived materials, e.g. corrugated board, solid board, paper cores, and honeycomb board. The effectiveness of the impregnation was also assessed using the method of measuring the contact angle of the reference and impregnated surfaces.

Keywords: cardboard; impregnations; tensile strength; contact angle.

1. INTRODUCTION

Along with the changing situation in the world, disasters, wars, and other unexpected events on the one hand, and tendencies towards ecological awareness and sustainable development on the other hand, the situation in the construction market is changing. New, unusual construction materials or materials known for years, although used mainly in other industries, are used. Paper is an example of such a material. It turns out that it can be successfully used in architectural and construction applications. Today, examples of paper architecture can be found all over the world, starting with Japan and the designs of the precursor of paper architecture – Shigeru Ban [1–3]. According to numerous studies presented in the literature, paper-based products can be valuable building materials due to the mechanical parameters [4,5], and above all, for ecological and economic reasons [6]. These products are used, e.g. as construction elements [7,8] or walls [9–13].

One of the challenges in using paper is its sensitivity to humidity and the presence of water. The problem arises from the fact that as the moisture of the material increases, the hydrogen bonds between the cellulose fibers are weakened, which in turn means a rapid decrease in the material strength parameters. The mechanical strength of paper decreases by approximately 10% for every 1% increase in material moisture level [6]. The optimum paper moisture content is around 5–7% [14,15].

In order to be able to use paper as a construction material, there is a need to minimize strength losses due to environmental

factors. Impregnation methods that serve to protect paper from moisture take various forms: spray impregnation, impregnation consisting of covering the paper with varnishes, lacquers, and oils (e.g. paraffin used previously by Shigeru Ban), impregnation by lamination with PE foils (e.g. used by researchers from TU Delft), immersion impregnation, impregnation by sticking a protective layer, impregnation by covering the paper with traditional waterproof materials, e.g. metal, chemical modification of the structure (e.g. used in Japan). The choice of an impregnant can affect the subsequent recyclability of the material, which should be considered [6]. For example, impregnation with polyethylene or polypropylene practically makes it impossible to reuse and is a source of environmentally hazardous waste [4]. While choosing the appropriate impregnation, it is worth considering the one that not only increases moisture resistance but also does not deteriorate other properties, including fire resistance or durability. From this point of view, it can be said that choosing the right impregnation is crucial in the use of paper in construction.

To date, there is little research in the literature on the protection of paper structures, e.g. [16,17]. In [17] various, essentially biodegradable protection agents were tested, such as wax, paraffin, wood oil, wood varnish, or PGG wall emulsions. An interesting observation is that the impregnation technique also affects its usability. The edges of the samples tend to absorb more moisture. It is worth using this knowledge while designing to avoid contact of such edges with water.

The conducted tests allow for the following conclusions: Impregnations based on oil and wax act as protection against moisture, also acrylic varnishes in tests show similar impregnating properties and the best results were obtained when using a composition of various agents, e.g. oil and wax. It should be noted that oil-based impregnations are the safest for the environment.

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On the other hand, a helpful solution is to use cladding made of traditional materials – cement boards, wooden cladding, or sheet metal cladding.

However, the current knowledge base does not facilitate answering the question of which impregnation system is the best – hence the need to conduct research as in this work to approximate the answer to this question. Therefore, research was conducted to compare the effect of impregnation of paper-based materials samples on their tensile strength. The answers obtained will be helpful in the search for effective waterproofing and fireproofing agents, the impact of which will not affect negatively strength parameters.

In the presented research, it was decided to test the effect of impregnating agents on tensile strength, as a representative of the strength parameter. This is an important parameter because individual structural elements in a potential building (planned to build a housing unit from materials of cellulosic origin and components of these materials) will work under loads causing tensile stresses. In the case of bending, to which structural elements will be subjected, tension occurs in the extreme fibers, so this is an important case. These loads were considered to be an important possible strength case that may occur when elements made of these materials are used as structural parts. Tensile strength was selected for the preliminary strength tests presented in this article as a representative feature, and in the future, it is planned, also as part of this ongoing project, to conduct broader tests, considering other strength characteristics of the material.

2. MATERIALS AND METHODS

2.1. Materials for tests

For the tests, four types of samples from a specific range of paper used in construction were prepared:

- Cardboard (solid cardboard) – thickness of 4 mm.
- Corrugated cardboard – wall thickness of 4 mm, 5 layers, EB flute.
- Paper tubes – outer diameter of 60 mm, wall thickness of 4 mm.
- Honeycomb – thickness of 25 mm.

Each of these sample series was then grouped into four categories according to the applied impregnation:

- Representative samples – no impregnation (5 pieces).
- Samples impregnated with impregnation No. 1 – wood oil Bird Brand Complete Decking Oil (5 pieces).
- Samples impregnated with impregnation No. 2 – yacht lacquer Sadolin Yacht (5 pieces).
- Samples impregnated with impregnation No. 3 – fire-retardant agent Burnblock (5 pieces).

Consequently, combinations of 80 samples were obtained to be tested.

2.2. Samples preparation, impregnation, and storage

In the case of corrugated cardboard, solid cardboard, and honeycomb, samples of an oar shape with a length of 300 mm and widths of 30 mm and 50 mm, respectively, were prepared

(Fig. 1). In the case of paper tubes, samples 300 mm long and 50 mm wide were cut out of tubes.

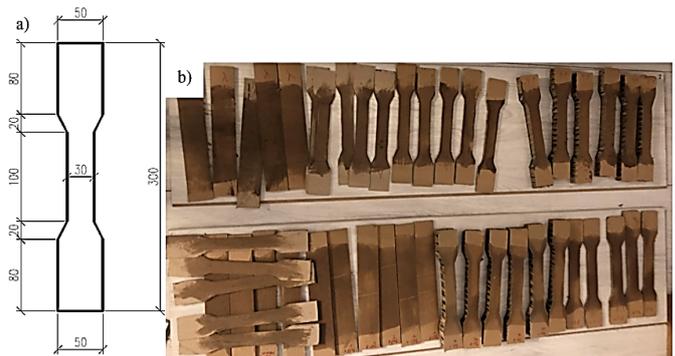


Fig. 1. (a) Sketch of the sample (dimensions in mm); (b) the samples after impregnation

The samples were impregnated at room temperature in a shaded place with low air humidity. The samples were covered with agents in accordance with the manufacturers' guidelines and analogously to how it was done in the case of testing the effectiveness of impregnations in the studies described in the literature. The impregnation was applied with a paintbrush by putting a layer of the agent and leaving it to dry. After 24 hours the operation was repeated, and a second protective layer was applied.

Both samples and impregnations were stored for a long time, at least 28 days, in dry conditions and at room temperature. However, the conditions in which the samples were not thoroughly examined, because the research was aimed at comparing the results with each other, therefore the focus was on the same preparation of the samples and standardization of the environment in which they were stored. The impregnations were stored in the same conditions (room temperature and low humidity) as these are the recommendations of the manufacturers of these agents.

In order to perform a tension test on the honeycomb, steel plates with a cut-out hole were glued to the wider part of the samples with quick-drying epoxy glue on both sides, which were then joined with a screw. A third plate was threaded through this screw, which was placed in the jaws of the testing machine.

2.3. Strength tests – tension tests

The tests were conducted at the Faculty of Civil Engineering at Wrocław University of Science and Technology with the use of a standard hydraulic press. The testing methods were proposed based on the wood testing methods according to the standard procedures based on the wood-cellulose products (paper-based products) analogy. The samples were placed in a testing machine and a tensile load was applied at a rate of 2 mm/min until failure. The results: tensile force, sample deformation (elongation), and the ultimate force were recorded using a computer set and photographic documentation was prepared. Test setup with samples of various series is presented in Fig. 2.

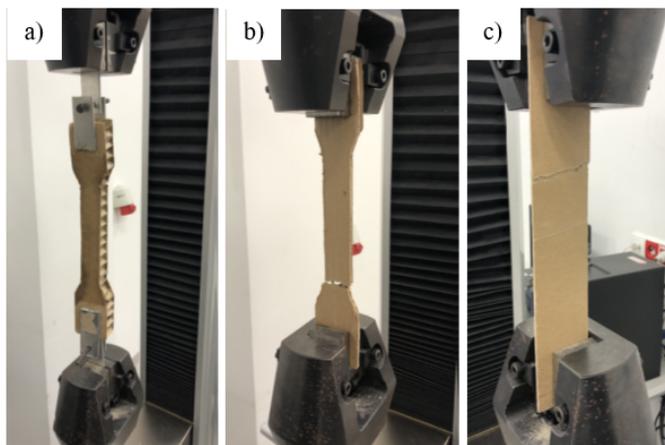


Fig. 2. View of the tension test setup: (a) honeycomb; (b) corrugated cardboard; (c) piece cut out of paper tube

2.4. Contact angle method

For the measurements of the contact angle, one sample of each type of cardboard coated with wood oil, yacht lacquer, fire-retardant agent, and without impregnation was selected. Measurements were made using a self-constructed test stand (Fig. 3), which consisted of a leveling table, a white screen, a lamp, and an adjustable arm with a syringe holder.

A cardboard sample was placed on a measuring table, the lamp was set appropriately so as to obtain a dark shape of a drop on a light background. Then the syringe filled with demineralized water was positioned so that the tip of the needle was just above the drop. A medical needle with a diameter of 0.6 mm was used. The drop was applied by gradually tightening the screw pressing on the syringe. The picture of the drop was taken 5 s after the drop came into contact with the cardboard. The photos were taken using a smartphone placed on a tripod with an additional macro lens with $\times 20$ magnification. Then, the photos of the drops were analyzed in the ImageJ program using a plugin dedicated to the analysis of the contact angle.

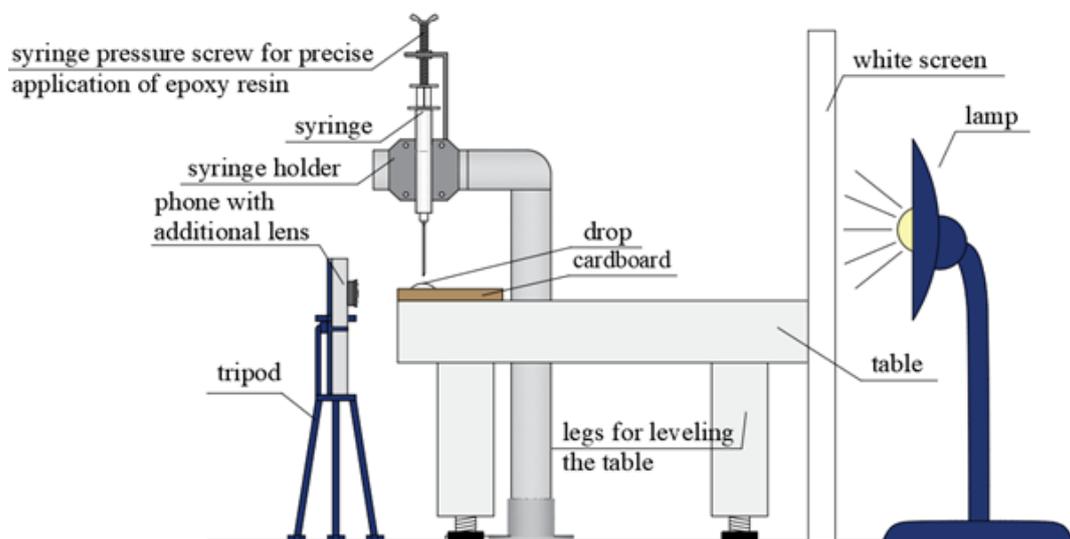


Fig. 3. Sketch of the test stand for contact angle measurements

3. RESULTS AND DISCUSSION

3.1. Strength tests

The results of tensile strength tests for individual groups of samples presented in Figs. 4–7 are shown below in tabular form in Tables 1–4. The mean destructive force for individual impregnation methods was calculated, as well as the effect of the method on the change (increase or decrease) of the obtained destructive force for a series of samples. Below there are also photo-documented images from tests along with the failure images of the samples as well as observations from the tests and attempts to discuss the obtained results.

Cardboard

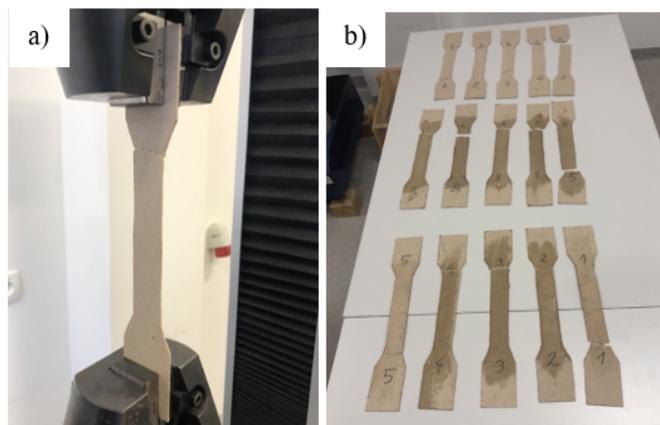


Fig. 4. (a) View of failure moment of cardboard sample; (b) cardboard samples after tension tests

As research shows, in the case of solid cardboard, there is an increase in destructive force, especially in the case of the application of yacht oil as impregnation, where the increase was 34.5 %. The strength increases for samples with the use of other impregnations – yacht lacquer and fire-retardant agent were significant as well – above 20% in each case.

Table 1
Results of the strength tests for cardboard samples

Sample series	Values of ultimate forces [kN]	Mean value of destructive force [kN]	Standard deviation [kN]	Variation coefficient [%]	Effect (change in the mean value of ultimate force) [%]
No impregnation	0.664	0.734	0.081	11.0	-
	0.778				
	0.802				
	0.796				
	0.630				
Wood oil	0.766	0.892	0.109	12.2	+21.5
	1.020				
	0.988				
	0.870				
	0.816				
Yacht lacquer	0.916	0.987	0.06	6.2	+34.0
	1.022				
	1.074				
	0.972				
	0.953				
Fire-retardant agent	0.914	0.910	0.06	7.1	+24.0
	0.821				
	0.874				
	0.972				
	0.969				

Table 2
Results of the strength tests for corrugated cardboard samples

Sample series	Values of ultimate forces [kN]	Mean value of destructive force [kN]	Standard deviation [kN]	Variation coefficient [%]	Effect (change in the mean value of ultimate force) [%]
No impregnation	0.416	0.349	0.04	11.3	-
	0.326				
	0.340				
	0.346				
	0.316				
Wood oil	0.354	0.310	0.03	11.1	-11.0
	0.340				
	0.298				
	0.278				
	0.282				
Yacht lacquer	0.360	0.358	0.02	6.1	+2.5
	0.330				
	0.374				
	0.382				
	0.342				
Fire-retardant agent	0.340	0.316	0.02	7.7	-9.5
	0.304				
	0.344				
	0.294				
	0.296				

The reason for such an increase may be the fact that the impregnate penetrating the structure of solid cardboard – a thin structure (thinner than in the case of, e.g. corrugated cardboard), affects the bonds between cellulose particles, strengthening them, which leads to a change in the value of destructive force.

Corrugated cardboard

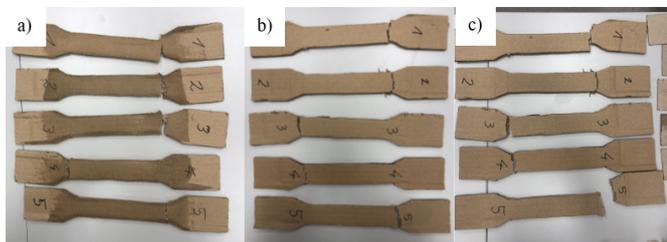


Fig. 5. Corrugated cardboard samples after tension tests

The conducted study shows that the effect of impregnation on the strength value in the case of corrugated cardboard is rather small. Wood oil and fire-retardant impregnators decrease the destructive force. Yacht oil, on the other hand, showed a 2.5% increase in destructive force. This percentage is so insignificant that it can be concluded that the impregnation did not affect the

value of the ultimate force and the value differs slightly, which may result from the scattering of the results of the obtained values.

Paper tubes

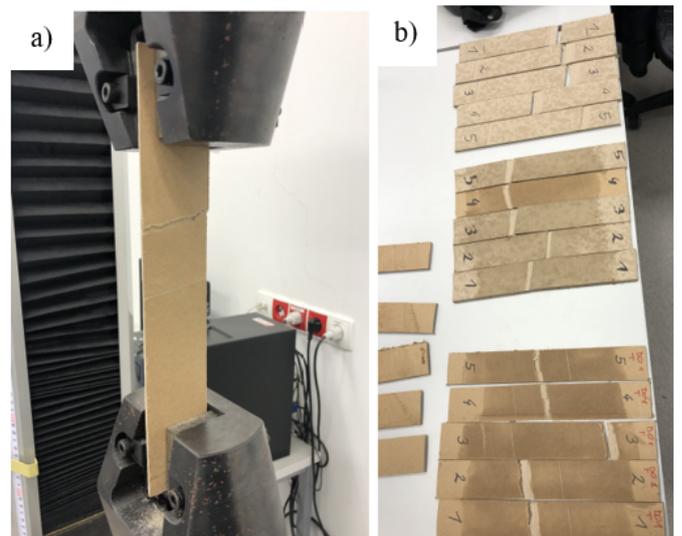


Fig. 6. (a) View of failure moment of paper tube sample; (b) paper tube samples after tension tests

Table 3

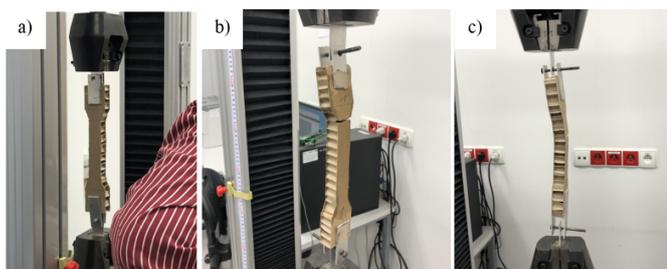
Results of the strength tests for paper tube samples

Sample series	Values of ultimate forces [kN]	Mean value of destructive force [kN]	Standard deviation [kN]	Variation coefficient [%]	Effect (change in the mean value of ultimate force) [%]
No impregnation	2.694	2.660	0.04	1.5	-
	2.704				
	2.660				
	2.638				
	2.604				
Wood oil	2.742	2.724	0.08	2.8	+2.4
	2.761				
	2.811				
	2.699				
	2.609				
Yacht lacquer	2.842	2.784	0.06	2.1	+4.7
	2.810				
	2.814				
	2.694				
	2.762				
Fire-retardant agent	2.710	2.708	0.05	1.8	+1.8
	2.754				
	2.658				
	2.658				
	2.760				

The observation is again confirmed: the fire-retardant impregnation does not affect the result of destructive force (less than 2% of change). Wood oil has a slightly positive effect on this value. The biggest difference is observed when using yacht oil, however, it is still an increase of less than 5%.

The results of the honeycomb test show that the effect of the fire-retardant agent is the least significant and at the same time negative, as in the case of the other samples. A rather surprising result, however, is an incredibly significant increase (over 100%) in critical force when using yacht oil or wood oil. In this case, it can be assumed that the agent penetrated the deeper layers of the honeycomb and affected the stiffness of the walls in the middle of the section, which resulted in an increase in strength.

Honeycomb

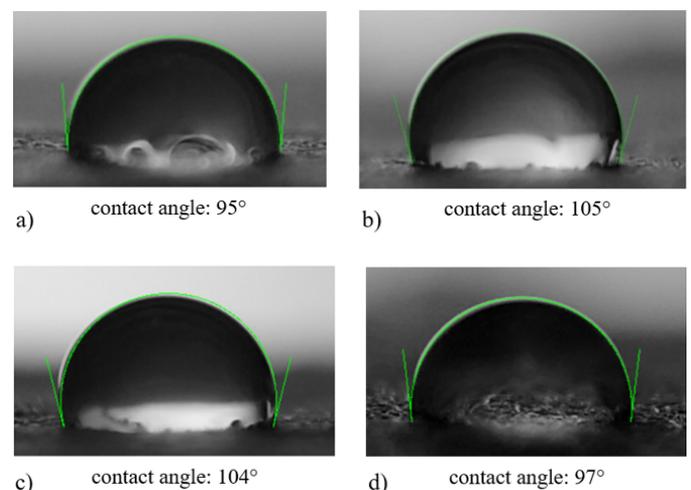
**Fig. 7.** Views of honeycomb sample during tension test**Table 4**

Results of the strength tests for honeycomb samples

Sample series	Values of ultimate forces [kN]	Mean value of destructive force [kN]	Standard deviation [kN]	Variation coefficient [%]	Effect (change in the mean value of ultimate force) [%]
No impregnation	0.228	0.255	0.04	17.0	-
	0.288				
	0.194				
	0.298				
	0.266				
Wood oil	0.822	0.716	0.09	13.2	+180.0
	0.716				
	0.592				
	0.732				
Yacht lacquer	0.758	0.730	0.02	2.7	+186.5
	0.728				
	0.716				
	0.718				
Fire-retardant agent	0.230	0.219	0.07	31.0	-14.0
	0.160				
	0.311				
	0.176				

3.2. Contact angle tests

Sample photos of the droplet are shown in Fig. 8, and all the results of the contact angle tests are summarized in Table 5. The smallest value of the contact angle was obtained for the reference sample ($95^\circ \pm 2.3^\circ$). Covering the cardboard with the impregnation increased the contact angle and thus the hydrophobicity of the surface by 2% for fire-retardant agent impregnation

**Fig. 8.** Sample views of water contact angle measurements on samples (a) not impregnated and covered; (b) wood oil; (c) yacht lacquer; (d) fire-retardant agent

(average result $97^\circ \pm 1.9^\circ$), 6% for yacht lacquer (average result $102^\circ \pm 2.3^\circ$) and 10% for wood oil (average score $105^\circ \pm 1.8^\circ$).

Table 5

Results of contact angle measurements of cardboard

Sample series	Values of ultimate forces [kN]	Mean value of destructive force [kN]	Standard deviation [kN]	Variation coefficient [%]	Effect (change in the mean value of ultimate force) [%]
No impregnation	95	95	2.3	2.45	–
	94				
	94				
	94				
	95				
	100				
Wood oil	104	105	1.8	1.70	+10
	108				
	106				
	103				
	105				
	104				
Yacht lacquer	99	102	2.3	2.22	+6
	99				
	104				
	101				
	102				
	104				
Fire-retardant agent	96	97	1.9	2.00	+2
	99				
	95				
	97				
	100				
	96				

When making drops, even for the reference sample, water drops did not soak into the cardboard. Drops of water soaked into the cardboard only after being rubbed with a finger. This is due to the cardboard manufacturing process. Typically, the paper is first heated to approximately 200°C and corrugated, then glue is applied to the corrugations, and the top flat layers of the paper are glued to it. This adhesive pre-impregnates and hydrophobizes the paper, resulting in an average contact angle of 95° for the reference samples. When the contact angle exceeds 90° , the surfaces are considered hydrophobic. Therefore, the difference between the contact angle for the reference and impregnated samples is not spectacular. Nevertheless, the impregnants used to improve the hydrophobicity of the cardboard surface, of which better results were obtained for the yacht lacquer and wood oil impregnation, which coincides with the strength results.

Comparing the obtained results with those presented in the literature, when it comes to the effectiveness of impregnation of materials of cellulose origin against the negative effects of water and moisture, an analogy can be noticed in the results presented, for example, in [17]. The authors of this paper showed the effectiveness of using some other impregnation agents and the best effect was obtained by using composite coatings consisting of both oils and waxes – Timberex hard wax oil, which could be somehow compared to the wood oil, which obtained the best results when it comes to the effectiveness of the impregnation in this study, however, they are not the same agents.

4. SUMMARY AND CONCLUSIONS

The study presents a series of tensile test results for four different types of paper assortments that appear in architecture. In addition, contact angle measurements were made for one type of cardboard. The impregnation agents were tested, which in earlier studies showed a positive effect on paper protection against the most troublesome environmental factors to which paper in construction may be exposed – water and fire. It was important to compare only those samples that showed promising protective properties.

The paper aimed to compare the destructive force and contact angle of comparative samples – non-impregnated with impregnated samples.

Several research outcomes show that the agents used during the test do not affect (or affect to a very small extent in individual cases) the reduction of the destructive force and in the case of honeycomb and waterproof oils there is even a significantly increased value of the destructive force.

To sum up, specifically:

The results for cardboard show that the effect of impregnation improves the tensile strength of the material – the ultimate force value increases by 21% when using wood oil, by 34% when using yacht oil, and 24% when using a fire-retardant agent. It can be assumed that these agents could penetrate the structure of the material and strengthen the bonds between cellulose molecules which in turn increased in strength. A solid board is a thin structure. Therefore, the penetration of impregnating agents is proportionally deeper than in the case of corrugated boards. Hence, it is assumed that corrugated board does not have such an impact and solid board does.

In the case of corrugated cardboard, the obtained results indicate that the impregnation methods are of little importance in the value of the destructive force. Wood oil slightly (11%) reduces the mean force value, and the use of yacht oil very slightly increases this value (2.5%). The use of a fire-retardant agent slightly reduced the destructive force (9.5%). It can be assumed that the fluctuations of these values may not be related to the effect of the impregnation but simply to the random nature of the distribution of the ultimate force. Conclusion: The corrugated board retains its properties after impregnation.

In the case of paper tubes, the value of destructive force slightly differs when using different impregnants. These differences are so small (up to 5%) that it can be said with a high

degree of probability that they result from the randomness of the force value, not from physical changes in the material characteristics that translate into strength.

On the other hand, honeycomb shows a high increase in tensile strength when using liquid impregnation agents. These are almost 3-fold increases (over 180%). It can be assumed that this increase is due to the penetration of the impregnating agent which stiffens the walls in the middle of the cross-section of the honeycomb and this stiffening has a key impact on the value of the destructive force. This conclusion may be very interesting and is a possible starting point for further research, also on methods of strengthening honeycomb structures. Interestingly, the fire-retardant agent impregnation no longer shows such an effect – in this case, the mean force value was analogous to that of the reference sample (difference of 14%). This indicates that the stiffening of the walls is caused specifically by compounds found in wood and yacht oils – both agents have a similar, but not identical composition. Chemical analysis of the indicated impregnations could answer the question of what exactly causes this increase in material strength.

On the other hand, contact angle measurements showed that impregnating agents increase the contact angle (by 2–10% depending on the impregnant), and thus reduce the wettability of the cardboard, which may have a positive effect on its strength. Wood oil has the best hydrophobic properties, followed by yacht lacquer.

Considering the above, the effect of impregnation has no or positive effect on the strength of the material and positively affects the hydrophobicity of the cardboard, therefore all impregnating agents can be used in the construction. It can be concluded that the tested agents are safe to apply for building structures.

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