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Influence of modification of aged spruce wood by *in situ* polymerization with ethyl methacrylate and methyl acrylate and paraloid B72 on color and water absorption

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Abstract: *Influence of modification of aged spruce wood by in situ polymerization with ethyl methacrylate and methyl acrylate and paraloid B72 on color and water absorption.* Wood is hydrophilic in nature, which creates problems such as dimensional instability and susceptibility to biological degradation. By using chemical modification, wood can be refined into new products with improved properties. Such a combination of wood and polymer is called a wood-polymer composite (WPC). The presented work compares the modification of aged wood by two methods. *In situ* polymerization with ethyl methacrylate and methyl acrylate with modification with Paraloid B72 solution. The determinants of the modifications carried out are percentage weight gain (WPG), color change and water absorption. Unmodified wood aged and unaged were the control samples. Also unaged and modified wood samples were prepared to compare results. The greatest change in color intensity and total color difference after the aging process was recorded for samples modified with Paraloid B72, and after UV light treatment - modified by the *in situ* polymerization. The *in situ*-modified samples had the lowest water absorption among aged samples, and Paraloid B72-modified samples had the lowest water absorption among unaged samples. The highest WPG was obtained from the aged samples modified by the *in situ* polymerization.

Keywords: spruce wood, *in situ* polymerization, ethyl methacrylate, methyl acrylate, paraloid B72, color, water absorption

INTRODUCTION

Wood is composed mainly of cellulose, hemicelluloses and lignin and also a large number of compounds in the form of organic extractives and inorganic minerals (ash) (Krzysik 1957, Rowell 2005). The hydrophilic nature of wood caused by hydroxyl groups of its structural and non-structural components creates problems such as dimensional instability or susceptibility to biological degradation. Through the use of chemical modification, wood can be upgraded to new products with improved properties (Prosiński 1984, Hill 2007, Belgacem 2008).

Any combination of wood and polymer is considered a wood-polymer composite (WPC). It is a wide range of materials including both - solid wood filled with polymer and polymer filled with wood fibers (Ellis 2000).

This work focused on wood filled with polymer. The idea of introducing polymers into wood and carrying out the modification process is to fill the cell lumen (Marchwicka 2012). Wood filled with polymer can be obtained in two ways – by impregnating wood with polymer or by impregnating wood with monomers and conducting polymerization inside wood. The second method is called *in situ* polymerization.

The process of impregnating solid wood with polymer can be carried out using e.g. acrylic resins, which include Paraloids. They are colorless, show good adhesion to the substrate, have high mechanical properties, are insoluble in water and are theoretically reversible. They are also characterized by high resistance to light, moisture and microorganisms. Paraloid B72 is a copolymer of ethyl methacrylate (70%) and methyl acrylate (30%) with a degree of polymerization of about 900. It is soluble in organic solvents such as – aromatic hydrocarbons, esters, ketones, ethylene glycol esters and others (Ciabach 1982, Paciorek 1993, Ciabach 2001). It forms low viscosity solutions. The glass-transition temperature is 40°C, the softening point is 70-75°C, and at 145-150°C it begins to flow (Szczepińska 2015). Paraloid B72 is one of the most light-resistant resins, under the influence of which it does not yellow and is used both as a protective coating agent and for structural strengthening of porous materials (Szczepińska 2015). Paraloid B72 is resistant to microorganisms, water and aqueous solutions of acids, alkalis and salts (Ciabach 1998). It is used in historic preservation because it has very high resistance to light and water, it is colorless and what is very important in historic conservation - it is theoretically reversible (Domasłowski 1979). It is used to strengthen the structure of damaged wood with reduced mechanical properties (Ciabach 2001).

An alternative that allows the chemicals to penetrate deeper the sample is the *in situ* polymerization. Due to the small size of the monomers and low viscosity, better penetration is achieved than with polymer solutions. It can be used to obtain the same polymer or copolymer (e.g. Paraloid B72) in wood through impregnating wood with monomers and subjecting the monomers to polymerization in the cell lumen or cell wall (Rowel 2005). One of the most common methods of inciting chain polymerization is the use of an initiator, which under the influence of various factors (usually temperature) breaks down into free radicals. Commonly used initiators are organic and inorganic peroxides or hydroperoxide (Porejko et al. 1965). The most commonly used monomers for wood modification are acrylic monomers, styrene or acrylonitrile. The modification of wood with monomers results in a composite with greater hardness, and enhanced dimensional stability. In addition, it is more resistant to abrasion, decay, weathering and its fire resistance is increased, while the mechanical properties of the modified material are improved (Ibach and Ellis 2005).

The presented study compares modification of aged wood by two methods. *In situ* polymerization with ethyl methacrylate and methyl acrylate with modification with the Paraloid B72 solution. The weight percentage gain (WPG), color change and water absorption were determined for modified wood. Unmodified aged wood and unmodified unaged wood were the control samples. Also modified, but unaged wood samples were prepared to compare results.

MATERIAL AND METHODS

The research material consisted of 60 samples of spruce wood (*Picea* sp.) with dimensions of 250 x 59 x 7 mm, average density of 445 kg/m^3, and the average moisture content of 7% (dry-weight method). All samples before the investigation were weighed, measured in three dimensions and the color was determined.

Firstly, the test material (60 samples) was divided into two groups, one of which (containing groups A, B, C) was aged accordingly, and the other (containing groups D, E, F) remained

unaged. Then each group (aged and unaged) was divided in 3 groups of 10 samples each – one for *in situ* polymerization modification, the second for modification with Paraloid B72 and the third for control (unmodified). The division of samples is shown in Table 1.

Table 1. Description of samples and their purpose

60 samples					
aged 30 samples			unaged 30 samples		
in situ polymerization 10 samples	Paraloid B72 10 samples	control non- modified 10 samples	*in situ* polymerization 10 samples	Paraloid B72 10 samples	control non- modified 10 samples
A	B	C	D	E	F

Color determination

The color was examined with a SP60 spectrophotometer X-Rite Europe GmbH (Regensdorf, Switzerland) using a D65 illuminant. The sensor head was 8 mm in diameter. The mathematical color space model contained in CIE DS 014-4.3/E:2007 was used to determine the color change of the samples. The CIELab color space is mathematically described by three values, each located on one of the axes: L - lightness, a - color from green to red, b - color from blue to yellow. △C and △E were investigated by determining the color change between successive stages. △C is a change of color intensity and △E means total color change.

The change of color intensity of the samples was figured using the equation:

$$\triangle C = \sqrt{a_2^2 + b_2^2} - \sqrt{a_1^2 + b_1^2}$$

Based on the three values, a point in the 3-dimensional coordinate system was obtained. When having two points – one before color change and second after color change - the result of mathematical equations determines if a average observer is able to notice the color difference:

*$0 < \Delta E < 1$ – observer does not notice the difference,
*$1 < \Delta E < 2$ - only an experienced observer notices the difference,
*$2 < \Delta E < 3.5$ - also inexperienced observer notices the difference,
*$3.5 < \Delta E < 5$ – a observer notices a clear color difference,
*$5 < \Delta E$ - an observer gets the impression of two different colors.

The total color change of the samples was figured using the equation:

$$\Delta E = \sqrt{(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2}$$

The sample's color was determined at the beginning of the research – samples unaged and unmodified, after part 1 of the aging process, after part 2 of aging process and after modification. In each case, three color measurements were taken on all samples. The first measurement was at the top, the second at the center, and the third at the bottom of the front surface of the sample.

Aging process

The aging method was developed on the basis of a number of other aging methods (Jankowska 2010). The process began by subjecting the samples to boiling in water (100 °C), after which they were put in a freezer (-20 °C) and then subjected to drying (105 °C). The cycle of boiling, freezing and drying was repeated 3 times with different times of each process. Then samples were subjected to soaking by leaving in room temperature water under load. In the next step, the samples were again subjected to boiling, freezing and drying successively. The whole aging process was planned of alternating cycles of mentioned treatments (boiling, freezing, drying and soaking) to accelerate the destructive aging process of wood in outdoor conditions. The exact aging scheme with the duration of the treatments is shown in Table 2. Before the start of the second part of the aging process, the samples weight and color were determined. After these steps, the samples were transferred to a chamber where they were exposed to UV light. After the second part the color of the samples was again determined. The whole process of aging took a 629.5-hour (26 days and 5.5 hours).

Table 2. The sequence of the aging process carried out on spruce wood

Part	Ageing stage	Temperature [℃]	Time [h]	Total cycle time [h]
1	boiling	99	3,5	24
	freezing	-20	4,5	
	drying	105	16	
	boiling	99	3	24
	freezing	-20	5	
	drying	105	16	
	boiling	99	3	48
	freezing	-20	21	
	drying	105	24	
	soaking	room temperature	144	144
	boiling	99	3	24
	freezing	-20	5	
	drying	105	16	
	boiling	99	3	72
	freezing	-20	69	
	boiling	99	3	24

	freezing	-20	5	
	drying	105	16	
	boiling	99	3	24
	freezing	-20	5	
	drying	105	16	
	boiling	99	8	56
	freezing	-20	24	
	drying	105	24	
	soaking	room temperature	72	136
	freezing	-20	24	
	drying	105	40	
2	UV	-	53,5	53,5

Weight loss

Weight loss was calculated by dividing the difference in weight before and after aging by the weight of the sample before aging and converting the result to percentages.

In situ polymerization

Samples from group C (10 aged samples) and from group D (10 unaged samples) were modified by *in situ* polymerization method. It started by introducing monomers into the wood. Ethyl methacrylate (Sigma-Aldrich), methyl acrylate and an initiator (ethylmethylketone peroxide) (Sigma-Aldrich) were used to prepare a solution in the ratio of: $70:30:0.5_{v/v}$. The prepared solution was poured into a beaker, in which the samples had been placed earlier. In the next step, beakers were placed in a vacuum vessel and the pressure was lowered for a period of 3 hours, and then the atmospheric pressure was restored. Samples were kept in for another 3 hours for better penetration into the sample. After the impregnation, the samples were carefully removed from the beakers and, after removing the superfluous liquid, they were vacuum-packed into foil and transferred to the dryer for 48 hours. After this time they were unpacked and left for another 48 hours in a dryer.

Paraloid B72

Samples from group B (10 aged samples) and from group E (10 unaged samples) were modified with a solution of Paraloid B72. The modification was started by dissolving 30 grams of Paraloid B72 (Blik) in 3 liters of toluene (Chempur, pure). The solution was stirred for 50 minutes at 45 °C. The beaker with the samples in the dissolved Paraloid B72 solution was

transferred to a vacuum vessel for 3 hours and, as in the previous modification (*in situ* polymerization), the samples were left in the solution for another 3 hours. After this time, samples were left at room temperature to evaporate the toluene.

WPG

The WPG was calculated by dividing the difference in dry sample weight before and after modification by the dry sample weight before modification and multiplying the result by 100%.

Water absorption

At the end all samples (A-F) were dried and placed in laboratory cuvettes, which were filled with distilled water to completely cover the samples. Samples were loaded with glass elements to prevent the samples from floating to the surface of the water. To avoid rapid evaporation of water, the cuvettes were covered with foil. The weight of the samples was measured successively at the beginning of the test and after: 2 hours, 11 days and 28 days to determine the moisture content of wood. After 28 days of soaking, the moisture content of samples was determined.

RESULTS AND DISCUSSION

In Table 3 the weight loss results for aged samples are shown.

Table 3. Weight loss of spruce wood after the aging process with standard deviation

Group of samples	Weight loss [%]	Average weight loss [%]
A	-9,2 ± 0,4	-9,3 ± 0,3
B	-9,6 ± 0,2	
C	-9,0 ± 0,3	

All aged wood samples showed a similar weight loss of 9-10 %. The action of cold water alone leads to the extraction of some water-soluble substances, which are, among others, tannins and sugars, which already account for 4%. After prolonged exposure to cold water, part of hemicelluloses are leached out. As the temperature increases, hydrolytic decomposition also proceeds, and the amount of leached substances can reach up to 15%. Hot water also removes extractives, selected polysaccharide fractions and decomposition products caused by the variable factors of the aging process (Prosiński 1984).

Table 4 shows color change of samples after aging and modification. The differences in L, a and b parameters are included as well as calculated parameters $\triangle C$ and $\triangle E$.

Table 4. Color parameters of native spruce wood and color changes of spruce wood caused by aging and modification with standard deviation

Group of samples		A	B	C	D	E	F
Before the test	L	85,34 ± 1,34	86,61 ± 1,48	85,82 ± 1,71	86,32 ± 1,45	85,92 ± 1,26	86,19 ± 1,31
	a	3,08 ± 0,79	2,55 ± 0,42	2,75 ± 0,65	2,63 ± 0,47	2,65 ± 0,52	2,86 ± 0,58
	b	18,67 ± 1,18	17,49 ± 1,32	18,47 ± 1,37	18,00 ± 1,76	18,64 ± 0,91	18,02 ±1,05
After aging process	△L	-17,23 ± 1,80	-17,31 ± 1,12	-16,70 ± 2,39	-	-	-
	△a	7,15 ± 0,59	7,33 ± 0,63	6,95 ± 0,89	-	-	-
	△b	11,99 ± 1,56	13,44 ± 1,88	11,89 ± 1,38	-	-	-
	△C	13,39 ± 1,62	14,79 ± 1,94	13,20 ± 1,51	-	-	-
	△E	22,23 ± 1,77	23,14 ± 1,84	21,71 ± 2,38	-	-	-
After UV light	△L	-1,21 ± 0,80	-1,39 ± 0,51	-1,10 ± 1,39	-	-	-
	△a	-1,71 ± 0,51	-1,42 ± 0,39	-1,34 ± 0,46	-	-	-
	△b	-1,73 ± 0,82	-1,49 ± 0,40	-1,14 ± 1,04	-	-	-
	△C	-2,16 ± 0,93	-1,84 ± 0,47	-1,49 ± 1,14	-	-	-
	△E	2,91 ± 0,61	2,58 ± 0,21	2,66 ± 0,45	-	-	-
After modification process	△L	-3,49 ± 1,75	-4,50 ± 3,01	-	-4,34 ± 2,21	-2,69 ± 0,88	-
	△a	3,48 ± 1,44	4,24 ± 0,52	-	1,65 ± 0,79	0,97 ± 0,41	-
	△b	5,50 ± 1,57	7,19 ± 0,41	-	7,00 ± 1,55	4,71 ± 0,75	-
	△C	6,31 ± 1,77	8,13 ± 0,49	-	7,17 ± 1,63	4,80 ± 0,75	-
	△E	7,62 ± 1,91	9,72 ± 2,09	-	8,47 ± 2,56	5,58 ± 0,85	-

The samples after part 1 of aging processes obtained a darker color, as shown the negative △L. There is also a strong change in the a and b parameters, showing that the sample began to be more red and yellow than the native spruce wood. The changes after UV light (part 2 of the aging process) are not that significant. The color of the samples (compared to samples after part 1 of the aging process) is darker, more green and blue. The color intensity decreased after exposure to UV light. . It is shown in research that high temperature causes changes in wood color for darker (Bekhta and Niemz 2003, Patzelt et al. 2003, Brischke et al. 2007). The total color change of the aged samples is >5, which means that the observer gets the impression of two different colors.

The color after modification of the aged spruce wood took on a more red and yellow hue, and became darker compared to its color before modification, but after aging. The color after the modification of unaged wood behaved in a similar way. Considering only aged samples, it is seen that spruce wood modified with Paraloid B72 shows a greater change in color intensity and in total color change compared to *in situ* polymerization modified wood. In unaged wood, on the other hand, a greater change in color intensity was observed in *in situ* modified samples. Nevertheless, this differences between modifications types are minor and comparable. This shows that, considering the color, the use of ethyl methacrylate and methyl acrylate to modify historic wood by the in situ polymerization method is not problematic. The perception of the color change is very similar to that of paraloid B72, which is already used in the conservation of historic wood.

The value of $\triangle E$ after modifications is higher than 5 so the observer gets the impression of two different colors.

The WPG of modified spruce wood is shown in Table 5.

Table 5. WPG of modified spruce wood with standard deviation

Group of sample	A	B	C	D	E	F
WPG [%]	52,1 ± 25,6	11,9 ± 4,2	-	11,8 ± 3,7	7,4 ± 0,2	-

The WPG of aged samples is higher than that of unaged samples - this is due to the fact that during the aging process the extractives were leached out, thus creating space for the paraloid or methacrylates. Also, a dissolution of resins, waxes and tannins can be caused by toluene used for modification with Paraloid B72 (Prosiński 1984). The highest WPG was observed for group A - 52,1%. Samples from groups B and D increased in weight by 11,9% and 11,8%. Group E showed a WPG at the lowest level - 7,4%. Other researchers obtained a WPG from 1 to 10 % depending on wood, methods and additives (Tuduce Trăistaru et al. 2011, Timar et al. 2014).

For both aged and unaged samples, the WPG is higher for *in situ* polymerization-modified wood, because unlike the Paraloid B72 solution with toluene - the solution of ethyl methacrylate, methyl acrylate and initiator penetration better wood because they are a small particles (monomers) and paraloid B72 is a big in size polymer. In the case of wood modified with Paraloid B72, there was, mentioned before, dissolution of wood components such as resins, waxes, fats and tannins with toluene, which contributed to a slight weight gain after modification (Prosiński 1984). The obtained results of WPG showed that modification with monomers gives higher values, which means a higher amount of polymer stays in wood. Especially in the case of aged wood.

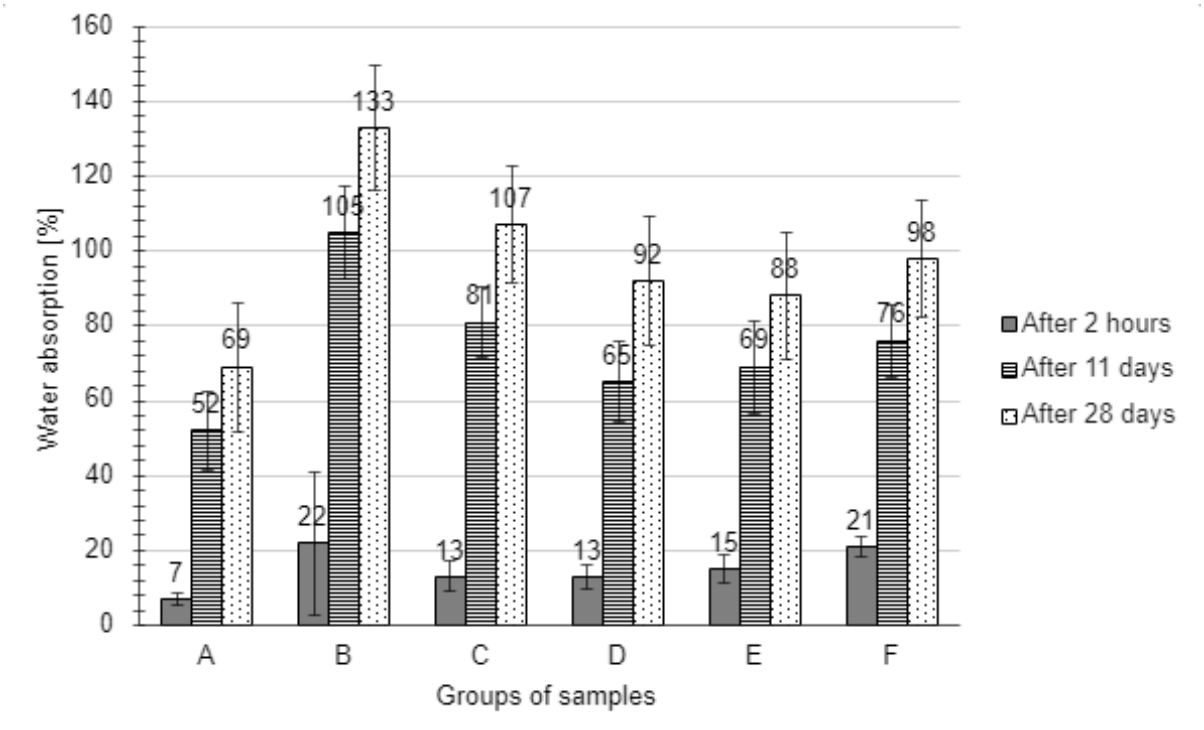

Figure 1. Water absorption of native, aged and modified spruce wood with standard deviation

Spruce wood after aging shows slightly higher water absorption than unaged wood. It is particularly evident after 28 days of contact with water. This correlation is not apparent after 2 h of testing. Wood modified by both applied in this work method obtained a lower water absorption level than unmodified wood, very similar for both methods. After a shorter soaking time (2 h and 11 days), slightly lower values of water absorption are observed for spruce wood modified with monomers, but after a longer time (28 days) - a slightly lower values of water absorption are obtained for wood modified with Paraloid B72. In this case, the reduction in water absorption was 2, 4 and 4 percentage points, respectively. At this point, it is worth noting that unaged wood modified with both paraloid B72 and acrylic monomers achieved similar WPG results.

Among all spruce wood samples - both aged and unaged, modified and unmodified – aged spruce wood modified with paraloid B72 (group B) had the highest water absorption, while aged spruce wood samples modified by *in situ* polymerization (group A) had the lowest. Modification with monomers reduced soakability by 35% after 28 days of soaking relative to unmodified samples. Aged wood modified with acrylic monomers had much greater WPG compared to aged wood modified with paraloid B72 – almost 4,5 times greater. It was also almost 4,5 times greater than WPG of unaged wood modified with the same method. Therefore, the water absorption was much lower for aged wood modified with acrylic monomers than for unaged wood modified with acrylic monomers. The modified wood with the highest WPG showed the lowest water absorption. Modifying aged wood by introducing monomers in wood and *in situ* polymerization is a more efficient way to preserve wood from water impact than modification with paraloid B72.

Figure 2 shows all the samples after the aging and modification process and the control samples. All sets of 10 samples in the figure are labeled with letters A-F corresponding to the names of the sample groups described in Table 1.

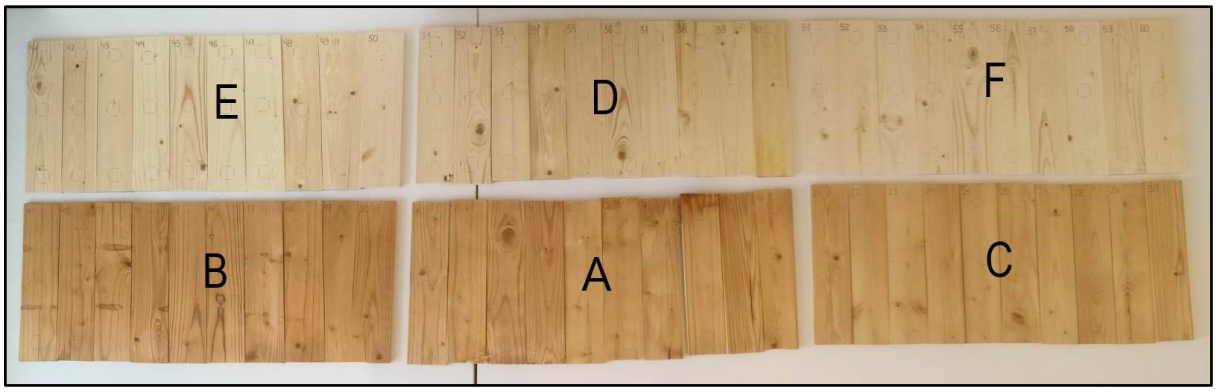

Figure 2. Aged and unaged spruce samples after modification process

Visually comparing the colors of the aged and unaged samples shown in Figure 2, one gets the impression of two completely different colors, which is consistent with the results of the total color difference (E) study. Among the samples of group A, one can see sample number 8, which has an excess of ethyl methacrylate and methyl acrylate on its upper face. Due to the inadequate amount of substance introduced, this sample was not taken into account when testing the color after modification, WPG and water absorption.

CONCLUSIONS

- After an aging process involving cooking, freezing and drying, the spruce wood darkened and became more yellow and red in color compared to native spruce wood.
- After an aging process that included UV light exposure, the spruce wood darkened and took on more blue and green colors compared to wood after the aging process involving cooking, freezing and drying.
- After both modification methods, aged and unaged spruce wood darkened, became more yellow and red in comparison to wood before modification.
- A change in color intensity and total color change of the aged spruce wood after modification with Paraloid B72 and after was minor and comparable. It was slightly lower for aged spruce wood modified by *in situ* polymerization method.
- The WPG of aged wood after modification is higher than that of unaged wood.
- Wood modified by *in situ* polymerization method, both aged and unaged, in comparison to wood modified with paraloid B72 showed higher WPG.
- Aged spruce wood modified by *in situ* polymerization modification method showed the highest WPG.
- Aged wood modified with acrylic monomers showed the lowest water absorption.

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Streszczenie: *Wpływ modyfikacji starzonego drewna świerkowego metodą polimeryzacji in situ metakrylanem etylu i akrylanem metylu oraz paraloidem B72 na barwę oraz nasiąkliwość.* Drewno ma charakter hydrofilowy, co stwarza problemy takie jak niestabilność wymiarowa i podatność na degradację biologiczną. Stosując modyfikację chemiczną, drewno może być uszlachetnione tworząc nowy produkt o ulepszonych właściwościach. Takie połączenie drewna i polimeru jest uważane za kompozyt drewno-polimer (WPC). W prezentowanej pracy porównano modyfikację starzonego drewna dwiema metodami: polimeryzację *in situ* metakrylanem etylu i akrylanem metylu z oraz modyfikację roztworem Paraloidu B72. Wyznacznikami przeprowadzonych modyfikacji są: procentowy przyrost masy (WPG), barwa i absorpcja wody. Próbki kontrolne stanowiło drewno niemodyfikowane i starzone. W celu porównania wyników przygotowano również próbki drewna niestarzonego i modyfikowanego. Największą zmianę intensywności barwy i całkowitej różnicy barw po procesie starzenia odnotowano dla próbek modyfikowanych Paraloidem B72, a po działaniu światła UV - modyfikowanych polimeryzacją *in situ*. Próbki modyfikowane polimeryzacją *in situ* charakteryzowały się najniższą chłonnością wody wśród próbek starzonych, a próbki modyfikowane Paraloidem B72 najniższą chłonnością wody wśród próbek niestarzonych. Najwyższą WPG uzyskano z próbek starzonych, modyfikowanych polimeryzacją *in situ*.

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