Development of the "bog oak" technology: modification of wood in environmentally friendly way

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Abstract

The study was based on sustainable wood eco-treatment in order to improve its properties and resistance without harming the environment. The aim of this study was to determinate the influence of different thermal treatments – additives Fe_2O_3 or $FeCl_3$ with and without commercial tannins - on Pinus Sylvestris L., Picea Abies (L.) H. Karst., Pseudotsuga menziesii, Juglans Regia L. and Acer platanoides L. wood mass loss and wood density. The total phenolic compounds in the wood of different species after thermal processing were also analysed.

This study has shown that wood reaction to different treatments after thermal processing were much dependent on tree species, therefore, different treatments gave quite contradictory responses. More similar response was found for two conifers – Pinus sylvestris and Picea abies – in case of mass loss and wood density. The treatments with additives Fe_2O_3 or $FeCl_3$ with and without commercial tannins decreased the content of total phenolic compounds compared with the control treatment in the wood of Pinus sylvestris, Picea Abies and Acer platanoides. While the content of total phenolic compounds in Juglans Regia wood increased when pure $FeCl_3$ and $FeCl_3$ together with tannins were added, and in Pseudotsuga menziesii wood when $FeCl_3$ together with tannins were added.
Keywords: iron oxides, iron salt, commercial tannins, phenolic compounds, mass loss, wood density.

Introduction

Despite good characteristics, wood also has disadvantages, such as resistance to the atmospheric effect, anisotropy of properties and instability of dimensions, which limit wood use. To eliminate these defects, wood is modified.

The idea of this work is to create technology of specific wood modification to selected different tree species to get similar properties to "bog oak wood". It has been observed that the trees in the bog environment have undergone a great chemical change over the years. Bog wood is a wood preserved in a bog due to anaerobic conditions. The extremely low oxygen conditions of the bog protect the wood from decay, while the underlying peat provides acidic conditions where iron salts and other minerals react with the tannins in the wood, gradually giving it a distinct dark brown to almost black colour. Under the influence of the minerals and iron from the water, the decomposition of wood is considerably slow.

When wood is exposed outdoors, the photooxidation or photochemical degradation process begins: the color changes followed by the loss of fibers and surfaces erosion are often observed (Ayadi et al. 2003; Williams 2005; Garcia et al 2014). The wood properties change due to internal chemical reactions, and wood strength is lost as it biologically degrade due to oxidation, hydrolysis, and dehydration reactions (Rowell 1983, 2006).

Thermal treatment induces the changes in wood chemical composition, reduces the water absorption, and improves dimensional stability and biological durability of wood (Wang et al. 2015).

The basic idea of more sustainable wood eco-treatment is to improve its overall properties and performance without harming the environment. Overall, this study was based on the search for environmentally friendly wood modification technologies and methods to improve the physico-chemical and aesthetic properties of wood.
The simulation of a specific bog environment for wood eco-modification was hypothesised as a relatively short way to develop a sustainable product. Grosse et al. (2019), aiming to improve the wood properties, treated the wood with lactic acid-based treatments with addition of chestnut tannins. Their results showed increased biological resistance and product persistence of modified wood.
The aim of the study is to develop environmentally friendly wood modification technology based on the reaction of bioactive compounds with modification reagent, ensuring rational use of lignocellulosic raw material and providing higher added value to the products.
Main objectives of this study were to determinate the influence of different thermal treatments with additives Fe_2O_3 or $FeCl_3$ with and without commercial tannins on the following issues: the total phenolic compounds (TPC) in the solvents after the thermal processing of different tree species; the mass and density of Pinus sylvestris L., Picea abies (L.) H. Karst., Pseudotsuga menziesii, Juglans Regia L. and Acer platanoides L. wood; the Fe concentration in different wood layers after modification.
Unique and environmentally friendly technology will improve the properties of wood, thus expanding the potential of wood use in international market. Different parameters of the modification process and different concentrations of the added chemical compounds for each tree species have been tested for the development of a special organic modification reagent for searching of appropriate methodological solutions. The total concentration of phenolic compounds in different modification solutions was determined. After each test, the most important modified physical properties (mass, density) were evaluated to determine the effectiveness of the wood modification process. Investigations of the initial properties of modified wood of studied tree species are carried out according to standardized research methods widely used in the material science. The wood properties after modification are assessed and compared with the control wood samples, also the relations of these properties are analysed. After testing various wood modification variants and adjusting the parameters of the modification process, the selected wood samples were subjected to further investigation by determining their chemical composition. The obtained results showed that the wood properties of the investigated tree species change in different ways under different modification solutions and under different modification conditions.

Methodology

Five tree species *Pinus sylvestris* L., *Picea Abies* (L) H. Karst., *Pseudotsuga menziesii*, *Juglans Regia* L. and *Acer platanoides* L. were obtained from a sawmill and their samples were used for the experiment. The sets of 20 wood samples of 20×20×30 mm dimensions from each tree species were prepared for the hydrothermal processing. The prepared wood samples were exposed to the different solvents prepared of distilled water, as a base, and iron oxide (Fe_2O_3), iron salt ($FeCl_3$) and commercial tannins for 75 hours at temperature 100 °C in a laboratory equipment. There were five treatments (15 g $FeCl_3$; 15 g $FeCl_3$ and 50 g commercial tannins; 15 g Fe_2O_3; 15 g Fe_2O_3 and 50 g commercial tannins), including the control with no additives.
To assess the concentrations of extractible phenolics, the solvents with Fe_2O_3 or $FeCl_3$ and with or without tannins after the processing of each tree species were sampled into individual laboratory test-tubes and transported to the laboratory. The chemical study was focused to compare the concentrations of extractible phenolics from selected tree species. Total phenolic compounds in the solvents after processing were semi-quantitatively evaluated with a Folin-Ciocalteu reagent (Singleton and Rossi, 1965). According to this procedure commercial reagent was diluted with distilled water at a ratio 1:10. The reaction mixture was prepared by mixing of 150 µL of tested solvent after thermal treatment, 750 µL of Folin-Ciocalteu reagent solution and 600 µL of 7.5% sodium carbonate solution. The absorption was read after 60 min at λ = 765 nm. The samples were prepared in triplicate for each analysis and the mean value of absorbance was obtained. The same procedure was repeated for the standard solution of gallic acid, and the calibration line was construed ($y = 10.8386x + 0.036$, $R^2 = 0.993$). The TPC was expressed in mg gallic acid equivalents (GAE) / 1 kg of tested wood species after processing.

To assess mass and volume changes of wood samples, all wood samples were oven-dried to constant weight before and after the treatment processing, and then the mass and volume of the samples were measured.
To assess Fe concentration in wood, the treated wood samples were removed from the solvents and cut following such scheme: each sample was cut into three sub-samples/layers, representing outer, intermediate, and inner wood layer with the thickness of 2 mm per each layer. Total Fe concentration was analysed by inductively coupled plasma optical emission spectroscopy according the LST EN ISO 16968:2015.
A statistical analysis was conducted using the Statistica 11.0 program with the analysis of variance (ANOVA) applying Tukey's Honest Significant Differences (THSD) Test. The one-way comparison tests were performed at a 0.05 significance level.

Results

Chemical changes of solutions, used for hydrothermal wood treatments

The solvents, obtained after the thermal treatment using the different additives (Fe_2O_3; Fe_2O_3 and commercial tannins; $FeCl_3$; $FeCl_3$ and commercial tannins) and the control without additives, were chemically analysed for the solvent pH and the content of total phenolic compound. The data showed that pH significantly decreased in all solvents with $FeCl_3$ compare with the control treatment of each species. The addition of Fe_2O_3 did not change the mean solution pH significantly. The measured exchanges of total phenolic compounds (TPC) in the wood of different tree species presented in Figure 1.:

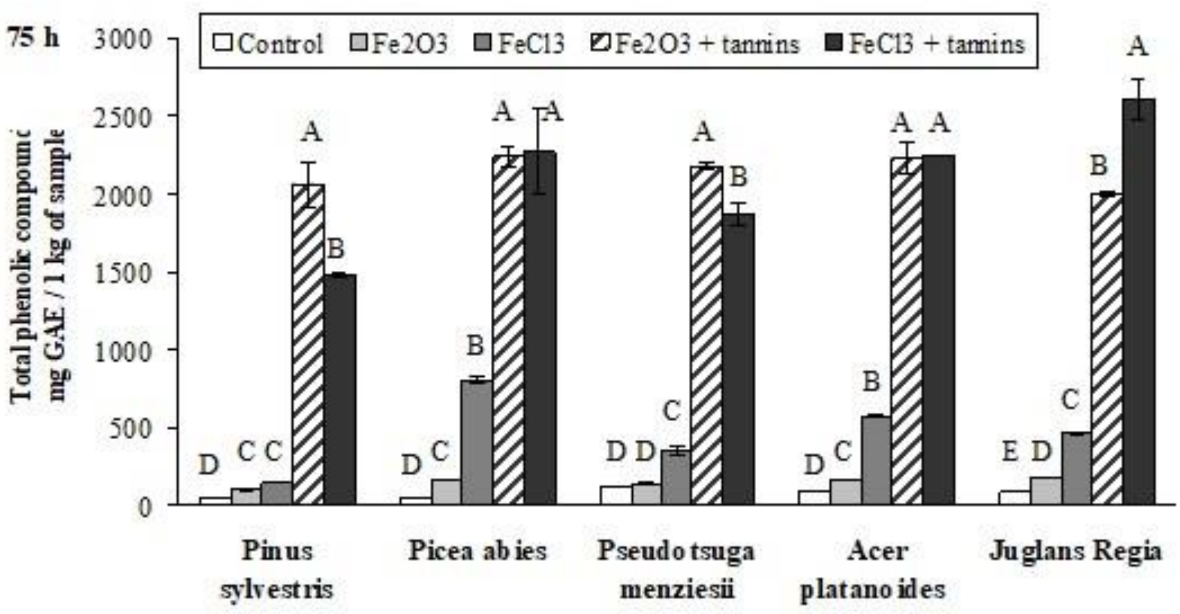

Figure 1. Content of total phenolic compounds (mg GAE - gallic acid equivalent/1 kg of sample after processing) in the solvents after different treatments of different tree species. Bars show standard deviation of mean; different capital letters given above the bar show statistically significant differences between the treatments for each tree species at $p < 0.05$ ($n = 3$)

The increased content of TPC in the solvents was obtained after the thermal treatment of all tree species wood, when Fe_2O_3 and $FeCl_3$ with and without commercial tannins were added compared to the control treatment. The TPC content increased from 15-25 times (for fir, maple, and walnut) to 30-40 times (for pine and spruce) in the solvents after the treatments with additives Fe_2O_3 and $FeCl_3$ together with commercial tannins for all species. Wood treatment with pure additive $FeCl_3$ without tannins showed different changes in TPC contents: the difference from the control comprised up to 3 times for all species, except spruce, which showed the difference of 15 times. The treatment with pure additive Fe_2O_3 did not change the TPC content in the solvent after the thermal treatment compare with the control treatment.

Changes of wood mass, volume, and density after treatment

The thermal processing when the control treatment without additives was used, caused the wood mass loss by 4.2-5.3 percent for pine, spruce and maple, and by 9.2-10.4 percent for fir and walnut wood (Table 1). When wood samples were treated with Fe_2O_3, the mass of wood samples decreased slightly by 2-4% for all species, except Picea Abies, which mass did not change (data not shown). When wood samples were treated with the pure additive Fe_2O_3, the mass of wood samples decreased by 3.5-5.8 percent for all species, except fir wood, which lost the 15 percent of its mass. The treatment with the additives Fe_2O_3 and tannins responded in relatively slight mass loss for spruce, fir, maple, and walnut. The highest loss of mass was fixed in wood after the treatment with the pure additive $FeCl_3$: the different from the untreated comprised from 13.7-15.6 percent for pine, spruce, fir and walnut to 20.7 percent for maple.

The thermal processing and different treatments caused different response of wood volume (Table 1). After the control treatment, wood volume decreased by 1.8-2.6 percent for fir and maple, 7.7-10.1 percent for pine, spruce, and walnut. Pure Fe_2O_3 induced wood volume reduction from 2.6-3.8 percent for fir, maple, and walnut to 7.6-8.4 percent for pine and spruce. In this case, the addition of tannins caused higher wood volume reduction only for maple wood. The thermal processing with the pure additive $FeCl_3$ reduced wood volume by 9.2-11.4 percent for all species, except fir, which volume decreased only by 3.8 percent. When tannins were added together with $FeCl_3$, the wood volume decrease was slightly reduced.

The wood density response was different both between different treatments and between tree species (Table 1). The control treatment caused the increase of pine and spruce wood density, the decrease of fir wood density, and no impact on maple and walnut wood density. Similar response in wood density change was obtained after the treatment with the additive Fe_2O_3, showing the increased wood density of pine and spruce. The thermal treatment with the additive $FeCl_3$ decreased wood density of all tree species. The highest changes of 10.5-12.1 percent from untreated wood were obtained for fir and maple wood.

Table 1. Changes of wood mass, volume, and density after treatment, '+' means increase, '-' means decrease. Different capital letters given next to the mean value show statistically significant differences between the treatments for each tree species at $p < 0.05$ ($n = 20$)

Treatment*	Changes (%) of parameters after treatment								
	Mass			Volume			Density		
	Mean	Std Dev		Mean	Std Dev		Mean	Std Dev	
Pinus sylvestris									
Control	-5.3	0.3	C	-9.2	2.6	B	+4.4	3.1	C
Fe_2O_3	-4.1	0.7	D	-7.6	1.3	BC	+3.9	1.7	C
$FeCl_3$	-15.6	0.8	A	-11.1	1.3	A	-5.0	1.9	A
Fe_2O_3 + tannins	+3.8	2.7	E	-5.9	3.1	C	+10.4	5.2	D
$FeCl_3$ + tannins	-8.9	1.1	B	-7.9	1.3	B	-1.1	1.8	B
Picea abies									
Control	-4.8	0.4	C	-7.8	2.3	B	+3.3	2.7	B
Fe_2O_3	-5.8	0.5	BC	-8.4	2.7	AB	+3.0	3.2	B
$FeCl_3$	-13.7	2.5	A	-10.5	2.1	A	-3.6	3.0	A
Fe_2O_3 + tannins	-0.6	0.1	D	-3.7	0.8	B	-5.0	1.1	C
$FeCl_3$ + tannins	-6.4	0.7	B	-7.8	1.1	B	+1.5	1.3	B
Pseudotsuga menziesii									
Control	-10.4	1.2	B	-1.8	0.7	B	-8.7	1.3	B
Fe_2O_3	-15.5	1.5	A	-3.8	0.9	A	-12.1	2.0	A
$FeCl_3$	-15.5	1.5	A	-3.8	0.9	A	-12.1	2.0	A
Fe_2O_3 + tannins	-4.5	1.1	C	-0.7	0.9	C	-3.9	1.2	C
$FeCl_3$ + tannins	-10.8	1.5	B	-1.9	0.8	B	-9.1	1.9	B
Acer platanoides									
Control	-4.2	0.5	D	-2.6	0.7	D	-1.7	0.8	C
Fe_2O_3	-3.5	1.1	D	-2.6	0.6	D	-1.0	1.4	C
$FeCl_3$	-20.7	1.4	A	-11.4	1.7	A	-10.5	1.8	A
Fe_2O_3 + tannins	-6.3	0.6	C	-7.0	1.2	C	+0.8	1.6	D
$FeCl_3$ + tannins	-14.5	1.4	B	-9.2	2.2	B	-5.8	2.8	B
Juglans Regia									
Control	-9.2	1.5	B	-10.1	1.4	A	+1.1	2.3	C
Fe_2O_3	-4.3	0.7	C	-3.3	1.5	C	-1.0	2.0	BC
$FeCl_3$	-13.9	1.9	A	-9.2	1.4	AB	-5.2	3.1	A
Fe_2O_3 + tannins	-3.4	0.8	C	-3.7	1.5	C	+0.4	2.1	B
$FeCl_3$ + tannins	-9.8	2.0	B	-8.1	1.3	B	-1.9	3.3	B

* Note: control - H_2O; $FeCl_3 \times 6H_2O$ – 270.3 g mol^{-1}; Fe_2O_3 – 90%; commercial tannins

Fe concentration in treated wood

Table 2. Mean Fe concentration in sub-layers (outer, intermediate, and inner wood layers with the thickness of 2 mm per each layer) after the thermal treatment. Different capital letters given next to the mean value show statistically significant differences between the treatments for each tree species at $p < 0.05$ ($n = 20$)

Treatments	Sub-layers, starting from outer layer in every 2 mm		
	Outer layer	Intermediate layer	Inner layer
Pinus sylvestris			
Control[a]	111.40±3.70 D	111.40±3.70 D	111.40±3.70 D
Fe_2O_3	8548.48±1925.16 A	473.60±32.44 C	372.59±37.68 B
$FeCl_3$	5358.97±426.94 B	653.31±141.13 B	322.49±12.79 C
Fe_2O_3 + tannins	3903.08±1481.83 C	1080.95±87.68 A	876.34±52.31 A
$FeCl_3$ + tannins	3224.85±393.19 C	585.27±91.34 B	383.67±22.98 B
Picea abies			
Control	45.00±0.48 D	45.00±0.48 D	45.00±0.48 D
Fe_2O_3	12453.94±2096.81 A	354.39±78.38 D	324.36±26.11 C
$FeCl_3$	8589.60±1161.92 B	1703.88±371.60 B	763.20±143.67 B
Fe_2O_3 + tannins	5608.70±700.68 C	937.37±126.64 C	770.43±128.01 B
$FeCl_3$ + tannins	7510.81±1209.56 B	2798.82±823.61 A	1094.26±108.84 A
Pseudotsuga menziesii			
Control	103.85±2.48 D	103.85±2.48 D	103.85±2.48 E
Fe_2O_3	14189.04±2520.21 A	751.68±155.74 B	565.20±63.27 B
$FeCl_3$	4457.39±715.83 B	685.24±60.76 B	405.52±19.01 C
Fe_2O_3 + tannins	4059.72±410.19 B	1365.48±64.82 A	1157.76±7.03 A
$FeCl_3$ + tannins	2888.95±218.92 C	500.15±36.90 C	325.58±28.54 D
Acer platanoides			
Control	37.61±3.01 E	37.61±3.01 D	37.61±3.01 D
Fe_2O_3	15966.13±1495.33 A	9327.27±1129.27 A	7851.80±651.68 A
$FeCl_3$	4794.83±1364.64 B	988.27±312.80 B	608.28±73.77 BC
Fe_2O_3 + tannins	3265.34±451.30 C	943.79±173.76 BC	707.02±126.17 B
$FeCl_3$ + tannins	1851.71±367.27 D	492.28±117.50 CD	407.38±44.58 C
Juglans Regia			
Control	196.00±8.74 D	196.00±8.74 D	196.00±8.74 E
Fe_2O_3	5808.97±3140.73 A	516.66±100.55 C	243.80±18.63 D
$FeCl_3$	3113.23±365.63 BC	791.47±65.74 B	614.88±10.11 B
Fe_2O_3 + tannins	4260.61±643.11 B	1576.83±47.91 A	1179.18±66.29 A
$FeCl_3$ + tannins	2185.00±189.69 C	828.16±127.60 B	566.69±6.42 C

[a] The data for the control treatment was obtained from one wood sample, not from its separate layers, because no Fe was added to the control treatment; the obtained minor Fe concentration in the control indicates the background concentration.

The Fe concentrations in wood after different treatments showed variability between treatments and tree species (Table 2). The highest Fe concentrations were obtained in outer wood layers of all tree species compared with the deeper layers. For pine, the Fe concentration in the outer wood layer was from 3.6-8.2 times (for the treatments with $FeCl_3$, Fe_2O_3 and tannins, and $FeCl_3$ and tannins) to 18.1 times (for Fe_2O_3) higher than in the intermediate wood layer. For spruce, this difference was from 2.7-6.0 times for all treatments, except for the treatment with Fe_2O_3, which showed 35.1 times difference between outer and intermediate layers. The lowest differences between Fe concentrations in all three layers were obtained for maple wood.

The comparison between different treatments within each of species showed that the highest Fe concentration in the outer wood layer was found in the treatment with Fe_2O_3 followed by $FeCl_3$, Fe_2O_3 and tannins, and $FeCl_3$ and tannins (in descending order) for pine, spruce, fir, and maple wood (Table 2). However, Fe concentration in walnut wood after the treatment with Fe_2O_3 and tannins was higher than in $FeCl_3$ with and without tannins.

Different situation was found for the inner wood layer (Table 2). In this layer, the highest Fe concentration was found after the treatment with Fe_2O_3 and tannins for pine, fir, and walnut. However, the additives $FeCl_3$ together with tannins and Fe_2O_3 responded in the highest Fe concentration for spruce and maple wood, respectively. The lowest Fe concentrations were obtained in spruce and walnut inner wood layer after the treatment with Fe_2O_3; and in fir and maple inner layer after the treatment with $FeCl_3$ and tannins.

Discussion and Conclusions

The simulation of artificial conditions, containing various chemical additives close to bog environment, was hypothesized as a relatively short way to develop a more sustainable wood product. As the objective of this study was test different wood modification treatments, searching for environmentally friendly technology, the impact of the phenolic substances - tannins, naturally found in wood, especially oak wood, were also tested. It is well known that iron ions are used in the qualitative analyses of various phenolic substances because such ions react easily with them to produce coloring substances (Hon and Shiraishi, 2010). One of the most efficient indicators of the treatment efficiency is mass loss of wood due to its thermal degradation (Tenorio and Moya 2013, Candelier et al. 2016).

During thermal processing solvent soluble compounds can cause an accumulation of the discolored compounds on the surface due to migration from the inner wood when the solvent evaporates. This reaction usually caused by a chemical interaction between iron ions and wood compounds such as hydrolysable tannins, which form non-extractable compounds and increases the stain and durability of wood. Usually water, iron and phenolic substances enrich a color of wood. When iron dissolves in water, iron ions are produced, since wood is an acidic substance and together with iron ions undergoes partial hydrolysis in an exothermic reaction and result in a strongly acidic solution which may accelerate ionization by oxygen, in this case iron ions react with hydroxyl groups in phenolic compounds in wood and form dark color and durability of wood (Hon and Shiraishi 2001). Acidic conditions may act as a depolymerization catalyst that further increases decomposition causing loss of mass, density, and mechanical properties. For instance, Wang et al. (2015) reported that low molecular phenolic compounds in the wood of conifers, contain flavonoids, stilbenes and lignans, which molecules may prove antifungal, antibacterial and antioxidant properties.

Different studies showed contradictory data obtained from different wood treatment experiments: the data varied due to wood species, initial moisture contents, and treatment intensities. For example, Tenorio and Moya (2013) studied mass loss along with heat treatment and found its dependence on wood species, specifics of drying process and treatment intensity. Chaouch et al. (2010) found that hardwoods (Fagus sylvatica L., Populus nigra L., Fraxinus excelsior L.) were more sensitive to thermal degradation than softwoods (Pinus sylvestris L., Abies pectinate Lam.), as demonstrated by the higher mass losses recorded for the same treatment duration. Only small mass losses were observed due to vaporization of volatile extractives and of bound water absorbed on the wood fibers until 160°C and thermo-degradations begin at higher temperatures (Chaouch et al., 2010). Earlier findings also concluded that wood species of lower densities generally are more resistant to thermal treatment than those of higher density and this was tried to explain by intrinsic thermal properties of each wood species.

This study has shown that mass loss of all studied tree species wood depended on individual wood properties and different environmental resistance. The mass loss after thermal processing was obtained in all treatments, including the control treatment, for all species, except Pinus sylvestris and Picea Abies wood in the treatment with additive Fe_2O_3 together with tannins.

Among all species, Pinus sylvestris and Picea Abies wood density after thermal processing also responded differently than other species, i.e., the wood density of these conifers increased after the thermal processing. However, wood density of other species - Pseudotsuga menziesii, Juglans Regia and Acer platanoides evidently increased in the control and $FeCl_3$ with tannins treatments or slightly decreased in Fe_2O_3 with tannins treatment.

Generally, different treatments of pure Fe_2O_3 and $FeCl_3$ or Fe_2O_3 and $FeCl_3$ applied together with commercial tannins decreased the content of total phenolic compounds compared with the control treatment in the wood of Pinus sylvestris, Picea Abies and Acer platanoides. More contradictory values were obtained for the wood of Pseudotsuga menziesii and Juglans Regia.
As a result of the modification process, as could be expected, the concentration of iron ions decreases proportionally with the distance from the surface of modified sample. In order to obtain a higher concentration of iron ions in the deeper layers of wood, it is necessary to increase the temperature and/or modification time. The theoretical implications of these findings still remain unclear and further experimentation is provided.

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