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CHARACTERIZATION OF Acrocarpus fraxinifolius WOOD SUBMITTED TO HEAT TREATMENT

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ABSTRACT

Aiming to provide greater visibility for the wood species *Acrocarpus fraxinifolius*, the present study sought to analyze the influence of heat treatment on an industrial scale applied to wood species, also popularly known as Indian cedar. The heat treatment was carried out in an autoclave, with temperature and pressure control, and with saturated steam injection, for temperatures 155 °C, 165 °C, 175 °C, and 185 °C. Physical, chemical, and mechanical tests were carried out for the analyzed wood. The content of holocellulose and total lignin decreased, while the content of extractives showed a substantial increase. The density increased after the heat treatment, however the treated wood showed cracks, and these cracks influenced the significant loss of the values of the mechanical properties of compression, tension, and flexion. The shear showed strength gain for the temperature of 155 °C, and 185 °C presented strength losses. The heat treatment in question contributes to increase the visibility, use and market value of wood.

Keywords: Acrocarpus fraxinifolius, chemical analyses, Indian cedar, mechanical properties, thermal modification, thermal treatment.

INTRODUCTION

With the exploitation of native species prohibited by law in Brazil, one of the alternatives capable of meeting industrial demand is the management of planted forests with fast-growing species. Currently, 93 % of the forests planted in Brazil correspond to different species of the pine and eucalyptus (IBÁ 2019). However, to increase the diversity of wood, planting of other species has been introduced in the country.

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One species that started to be indicated and has been gaining space in reforestation plantations in the North of Paraná, Southeast and Midwest regions of the country is *Acrocarpus fraxinifolius*. It is a species native to Asia, India, Burma and Bangladesh, which was introduced in Brazil in the 1990s, which shows good performance and superior growth when compared to plantations in other regions of the world (Carvalho 1998, Higa and Prado 1998, Prado *et al.* 2003). This species is known in Asia for mundani and lath tree, and in South America for pink cedar. In Brazil, the specie became popularly known as Indian cedar (Lorenzi *et al.* 2003, Firmino *et al.* 2015).

Indian cedar produces light and resistant wood, with a density of 438 kg/m³, short fibers (1,2 mm), the productivity of 30 m³/ha year to 45 m³/ha year, reaching 20 meters to 40 meters in height. Its wood is widely used in civil construction and for the manufacture of furniture and coffins (Prado *et al.* 2003, Lorenzi *et al.* 2003). However, in Brazil, the specie is still practically unknown, and national studies using the species are focused on silviculture (Nisgoski *et al.* 2012, Venturin *et al.* 2014) and physical and chemical characterization (Prado *et al.* 2003). Few studies have been carried out to expand the range of uses of this species, such as the potential use in OSB panels (Iwakiri *et al.* 2014), agglomerated wood panels (Trianoski *et al.* 2013), wood cement (Oliveira *et al.* 2020) and other construction applications.

Thus, the present work aimed to characterize the chemical and mechanical properties of Indian cedar wood, submitted to heat treatment on an industrial scale, carried out in an autoclave, with an application of heat and pressure, in comparison to untreated wood, seeking to bring more visibility, for the species, in addition to knowledge and application alternatives for the timber industries, as well as its market value. One way to increase the use and economic value of Indian cedar wood is to undergo it to heat treatment, which consists of a procedure used in wood species, which, in their majority, have lighter colors and lower market values.

To perform the heat treatment, the wood is exposed to high temperatures (180 °C to 280 °C), usually in an inert atmosphere, with air deficiency or in the presence of water vapor (Homan and Jorissen 2004). Under these conditions, there are changes in the chemical components of wood, cellulose, hemicellulose, lignin and extracts (Sundqvist 2004).

Chemical modifications benefit the wood, by increasing its dimensional stability, hygroscopicity, as well as increasing biological durability and color change throughout the thickness of the piece, the latter two being the most coveted benefits after heat treatment (Moura *et al.* 2012, Conte *et al.* 2014).

The darker color acquired after the heat treatment resembles the tones of tropical woods, replacing the use of native woods for certain purposes of greater value such as doors, windows, floors, musical instruments, internal and external furniture, boats, among others, making heat treatment an excellent method for adding value (Gunduz *et al.* 2009, Moura and Brito 2011).

Several studies sought to quantify the intensity of color modification in different species of wood subjected to heat treatment, such as for *Pinus radiata*, *Eucalyptus pellita*, *Tectona grandis*, *Luehea divaricata*, *Acacia auriculiformis*, among others. It is observed in these studies that, regardless of the use of different heat-treated species by different techniques, uniform browning occurs throughout the thickness of the wood, however with different colorimetric behaviors (Pincelli et al. 2012, Schneid et al. 2014, Zanuncio et al. 2015, Shukla 2019, Lengowski et al. 2021).

When exposed to sunlight, wood undergoes photooxidation or chemical degradation due to the absorption of solar radiation and ultraviolet (UV) rays, making it, depending on its chemical composition, more yellowish, reddish, darkened, pale or greyish, thus compromising its aesthetic appearance (Chang *et al.* 1982, Ayadi *et al.* 2003). Studies have shown that heat treatments can provide greater color stability to wood when exposed to UV radiation (Ayadi *et al.* 2003, Garcia *et al.* 2014), however, the same treatment may not be efficient to prevent discoloration of different woods (Gouveia 2008).

As for the modification of wood color, heat treatment is also considered a preservation method with low environmental impact due to the non-use of chemical products. After heat treatment, wood becomes more resistant to fungal decomposition when exposed to the dry rot fungus *Serpula lacrymans*, white rot fungus *Trametes versicolor*, and the brown rot fungi *Gloeophyllum trabeum*, *Coniophora puteana* and *Postia placenta* (Sivrikaya *et al.* 2015, Yalcin and Sahin 2015, Salman *et al.* 2017, Shukla 2019, Kamperidou and Barboutis 2021).

Increased resistance to termite attack was observed in some, but not all, studied species found in the literature (Salman *et al.* 2017, Sivrikaya *et al.* 2015), as well as the weathering of biotic and abiotic factors in an external environment (Kamperidou and Barboutis 2021). In order to overcome these disadvantages, the combination of heat treatment and additional chemical treatment is indicated (Salman *et al.* 2017).

On the other hand, with the changes in the chemical components of the cell wall of the wood, there is also a loss of mass and, consequently, a change in the value of mechanical properties, making it impossible to use heat-treated wood for some structural purposes (Sundqvist 2004, Moura *et al.* 2012).

The intensity of the changes is the result of a set of variables related to the method used, like as time, temperature, heating cycle and the surrounding atmospheres, and the raw material, like as species, density, initial moisture content, and extractives content (Sun *et al.* 2013), being extremely important to characterize different wood species heat-treated by different methods.

MATERIALS AND METHODS

Wood

The species used in the present study was the Indian cedar (*Acrocarpus fraxinifolius* Wight ex Arn.), With nine years of age, from a plantation in the municipality of Ribeirao Branco, in the interior of the state of Sao Paulo, southeastern Brazil. The pieces were obtained with dimensions of 6 cm x 16 cm x 3000 cm, and previously dried at room temperature, until they reached the moisture content of 12 % \pm 2 %, for subsequent performance of the heat treatment.

Heat treatment

The heat treatment was carried out on an industrial scale, in an autoclave, with temperature and pressure control, and saturated steam injection. Initially, the empty equipment was heated until it reached a temperature of 100 °C. Once this temperature was reached, the passage of steam was prevented, and the wooden pieces were inserted into it.

The thermal treatment was carried out for the following temperatures: 155 °C, 165 °C, 175 °C and 185 °C. The maximum pressure used was 735,5 kPa and the heating rate was 1,66 °C/min. The desired temperature for the heat treatment was maintained for two hours. Finally, the equipment and the wood cooled simultaneously to room temperature.

Characterization

The production of the specimens, the mechanical tests, and the density analysis were performed according to the Brazilian standard ANNEX B of ABNT NBR 7190 (1997). For the mechanical tests, the universal testing machine EMIC with a capacity of 300 kN, and the software TESC Emic (Instron, Brazil) were used for data acquisition.

Mechanical tests of compressive strength and stiffness (f_{c0} and E_{c0}), tensile strength and stiffness (f_{t0} and E_{t0}), strength and stiffness on static bending (f_{M0} and E_{M0}), and shear strength (f_{v0}) parallel to the fibers were performed.

The apparent density (ρap , 12 %), conventional specific mass, is defined by the ratio between the mass of the specimen and its volume with the moisture content at 12 %, according to Equation 1, where m12 % is the mass of the wood at 12 % humidity (g) and V12 % the volume of the wood at 12 % humidity (m³).

$$\rho ap, 12\% = \frac{m12\%}{V12\%}$$
 (1)

The samples for chemical analysis were obtained according to TAPPI T257-CM-85 (1985). The wood passed through the grinding process until it passed through particles in a 40 mesh (0,420 mm) sieve using a chopper and knife mill, both from the MARCONI brand.

The extractives content was carried out using the TAPPI T264-CM-97 (1997). The removal of the extracts was carried out in a Soxhlet extractor coupled to a flat-bottomed extraction flask, heated by a heating blanket, carried out in three stages: a) ethanol/toluene extraction for 6 hours; b) 95 % pure ethanol for 5 hours, c) boiling deionized water for 30 minutes.

After the three steps of removing the extractives, the samples were washed with deionized water, filtered, and dried in an oven at 103 °C \pm 2 °C for 24 hours. The extractives content was calculated by Equation 2, where *mi* is the initial mass of the absolutely dry sample (g) and *mf* is the final mass of the absolutely dry sample (g).

% Extractives =
$$\frac{mi - mf}{mi} x \ 100$$
 (2)

The determination of the lignin content was carried out by the Klason method, modified by Gomide and Demuner (1986), called the mini-sample method. The method consisted of treating the sample, free of extracts, with 72 % sulfuric acid in a water bath at 30 °C \pm 2 °C for 30 minutes and later, the sample diluted in 84 mL of deionized water is heated in an autoclave at 118 °C \pm 2 °C, for an hour. The filtered mixture in a number 2 porosity crucible results in two different samples, a solid sample being retained in the crucible and subsequently oven-dried at 105 °C \pm 3 °C, for analysis of insoluble lignin, and a filtered liquid sample, for analysis of soluble lignin.

The insoluble lignin content was calculated by Equation 3, where Pi is the initial weight of the absolutely dry sample (g) and Pf is the weight of the dry residue (g).

% Insoluble lignin =
$$\frac{Pf}{Pi} x \ 100$$
 (3)

For the determination of soluble lignin, the liquid sample was analyzed by a spectrometer in the ultraviolet region (UV-VIS) at absorbances of 215 nm and 280 nm, and calculated using Equation 4, where *A215* is the absorbance value at 215 nm, *A280* is the absorbance value at 280 nm and *ms* is the mass of the absolutely dry sample (g).

% Soluble lignin =
$$\frac{4,53 \times A215 - A280}{300 \times ms} \times 100$$
 (4)

Holocellulose was calculated by difference by Equation 5, where Ext is total extractive (%), *Lins* is insoluble lignin (%) and *Lsol* is soluble lignin (%).

$$%Holocelulose = 100 - (Ext + Lins + Lsol)$$
(5)

For each heat treatment temperature, as well as for the untreated wood, twelve repetitions were performed for mechanical tests and density analysis and six for chemical properties analysis.

Statistical analysis

Variation in the density, mechanical and chemical properties of submitted to heat treatment and untreated wood were compared and analyzed by one-way analysis of variance (ANOVA) at the 5 % level of significance using with Minitab statistical software (Minitab Inc., USA).

RESULTS AND DISCUSSION

The color of the wood is a very important property for the final consumer, with aesthetics, in some cases, the determining factor for the selection of a species of wood (Esteves and Pereira 2009). The heat treatment process used in the present study was able to change and standardize the color of the wood in all its thickness.

Figure 1 shows the effects of the four heat treatment temperatures used (Figure 1b, Figure 1c, Figure 1d and Figure 1e) in the samples of Indian Cedar, in comparison to untreated wood (Figure 1a).



Figure 1: Color variation of the samples: (a) Untreated and heat-treated at (b) 155°C, (c) 165 °C, (d) 175°C, and (e) 185 °C.

The heat-treated samples showed a considerable color change, from light coloration, with yellowish coloration (untreated wood) to a brownish coloration (heat-treated wood) that gradually darkened with the increase of the treatment temperature, being the darkest sample obtained with 185 °C.

Similar changes in the color of the wood were also evident in the studies developed by Cademartori *et al.* (2013) for *Eucalyptus grandis* wood heat-treated in a climate chamber at 180 °C, 200 °C, 220 °C, and 240 °C for 4 hours and 8 hours, and by Griebeler *et al.* (2018) for the same species heat-treated in an autoclave with steam at 140 °C, 160 °C, and 180 °C.

According to results found in the literature, the darkening of thermally treated wood is caused by the changes suffered by the chemical components of the wood, more specifically, by the degradation of holocellulose and extracts, water elimination, formation of carbonaceous coal, and the formation of oxidation products (Sundqvist 2004, Hill 2006, Esteves *et al.* 2008, Moura and Brito 2011 and Zanuncio *et al.* 2015).

The results of the chemical analysis of untreated and heat-treated wood are shown in Table 1.

	Untreated	155 °C	165 °C	175 °C	185 °C
Holocellulose (%)	66,2 (0,4) ^A	50,4 (4,0) ^B	48,3 (3,5) ^{BC}	46,7 (2,8) ^{BC}	44,5 (3,2) ^C
Total lignin (%)	31,7 (1,6) ^A	29,4 (6,6) ^{AB}	28,3 (3,5) ^{AB}	26,1 (6,4) ^{BC}	24,7 (3,7) ^C
Insoluble lignin (%)	28,7 (2,0) ^A	28,7 (6,7) ^A	27,7 (3,5) ^{AB}	25,7 (6,4) ^{AB}	24,3 (3,7) ^B
Soluble lignin (%)	2,98 (2,9) ^A	0,76 (2,0) ^B	0,57 (9,3) ^C	0,43 (7,4) ^{CD}	0,37 (12,7) ^D
Extractives (%)	2,1 (11,7) ^E	20,2 (2,0) ^D	23,4 (3,2) ^C	27,2 (2,5) ^B	30,9 (1,6) ^A

Table 1: Changes in chemical properties of heat-treated Indian cedar wood at different temperatures.

Averages followed by the same letter mean that they do not differ statistically at 5 % probability by Tukey's test. Values in parentheses refer to the coefficient of variation.

According to the results presented in Table 1, the holocellulose content reduced significantly for heat-treated wood in relation to untreated wood. The reduction in the holocellulose content increased from 23,9 % for wood treated at 155 °C, up to 32,8 % for treatment at 185 °C. The reduction of holocellulose was also observed in species of *Eucalyptus saligna* (Cademartori *et al.* 2015), *Eucalyptus grandis* (Cademartori *et al.* 2015, Batista *et al.* 2018) and *Tectona grandis* (Lopes *et al.* 2022).

According to Santos et al. (2001), holocellulose is the combination of cellulose and other polysaccharides,

called hemicellulose. Hemicelluloses are differentiated from cellulose in that they have different sugar units in five or six carbon atoms.

Therefore, the reduction presented by holocellulose, in the present study, results from the degradation of the fraction of hemicellulose, since, according to Fengel and Wegener (2003) and Sundqvist (2004), the heat treatment temperatures used are not high enough to degrade cellulose, which presents a high order in its crystalline structure and microfibrils, and acts as protection against acid attack during hydrolysis. While hemicelluloses have an amorphous structure and low molecular weight, they are therefore more susceptible to thermal degradation.

As for the total lignin content, there was no significant difference between the means of the untreated wood and wood heat-treated at 155 °C to 165 °C. Sundqvist (2004) and Soratto (2012) report that lignin is the structural component of wood that is more resistant to the action of heat, mainly due to the size and complexity of its structural arrangement, which is able to mitigate the effects produced by high temperatures. For the highest temperatures, 175 °C and 185 °C, the total lignin content showed a significant reduction of 17,5 % and 22,1 %, respectively.

The extractives content gradually increased with the increase in the treatment temperature, from 2,1 %, of untreated wood, to up to 30,9 %. Increased content of heat-treated wood extractives has been reported in the literature by Esteves *et al.* (2011), Batista *et al.* (2018), Esteves *et al.* (2022), Lengowski *et al.* (2021) and Lopes *et al.* (2022). It was also observed in the heat treatment of wood particles (Crespo *et al.* 2014), as well as in heat treated wood by different methods, as in the heat treatment with silicone oil studied by Okon and Udoakpan (2019).

According to Esteves *et al.* (2008) and Esteves *et al.* (2022), the original extracts of the wood are almost or totally degraded during the heat treatment, with the increase in the content of extracts observed related to the changes caused in the lignin content and mainly in the degradation of hemicellulose, which results in the formation of new chemical compounds, which are extracted during extractives analysis.

Table 2 shows the values obtained for the apparent density, and the mechanical properties of stiffness and compressive strength (E_{c0} and f_{c0}), stiffness and tensile strength (E_{t0} and f_{t0}), stiffness and strength on static bending (E_{M0} and f_{M0}), and shear strength (f_{v0}) parallel to the fibers, for untreated and heat-treated wood.

	Untreated	155 °C	165 °C	175 °C	185 °C
$\rho_{ap} \left(g/cm^3 \right)$	0,425 (14,0) ^C	0,643 (1,4) ^A	0,554 (6,5) ^B	0,546 (12,2) ^B	0,535 (6,1) ^B
$E_{c0}(GPa)$	9,3 (19,0) ^A	6,4 (8,7) ^B	4,9 (13,1) ^{BC}	4,3 (10,3) ^C	4,0 (17,8) ^C
f_{c0} (MPa)	35,3 (18,0) ^A	19,3 (14,5) ^B	18,3 (5,8) ^B	17,3 (3,8) ^B	16,4 (9,2) ^B
Et0 (GPa)	11,3 (24,1) ^A	6,3 (12,0) ^B	5,9 (13,2) ^B	5,4 (9,4) ^B	5,3 (16,0) ^B
f _{t0} (MPa)	66,9 (21,9) ^A	25,8 (21,1) ^B	19,6 (21,6) ^{BC}	17,6 (16,1) ^{BC}	13,3 (23,2) ^C
E _{M0} (GPa)	8,6 (16,9) ^A	4,9 (14,8) ^B	3,7 (23,1) ^{BC}	3,4 (24,1) ^{BC}	2,8 (27,8) ^C
f _{M0} (MPa)	56,4 (16,9) ^A	31,6 (23,2) ^B	21,7 (12,3) ^{BC}	17,7 (26,4) ^C	14,9 (20,5) ^C
f _{v0} (MPa)	7,9 (13,0) ^B	12,9 (5,9) ^A	7,2 (7,9) ^B	4,9 (13,0) ^C	3,5 (17,8) ^D

 Table 2: Changes in mechanical properties of heat-treated Indian cedar wood at different temperatures.

Averages followed by the same letter mean that they do not differ statistically at 5 % probability by Tukey's test. Values in parentheses refer to the coefficient of variation.

A significant increase was observed in the apparent density of heat-treated Indian cedar wood in relation to untreated wood. The increase in wood density after heat treatment was also observed for *Eucalyptus grandis* heat treated species in an oven at 140 °C, 160 °C and 180 °C by Brito *et al.* (2006) and at 200 °C and 230 °C by Batista *et al.* (2011).

With the degradation of the chemical components of the cell wall of the wood, it was expected that the density would decrease, as already observed in the literature for the species of *Eucalyptus grandis* (Calonego *et al.* 2014, Batista *et al.* 2018), *Eucalyptus camaldulensis* (Unsal and Ayrilmis 2005) and *Carpinus betulus L.* (Gunduz *et al.* 2009). However, Brito *et al.* (2006) suggested that the increase in heat treatment temperature was not enough to promote mass loss in the same proportion as the reduction in wood volume.

It is also observed that the highest density occurred for the heat treatment at $155 \,^{\circ}$ C and later decreased for the other temperatures, which did not present significant differences, we can consider that, probably, the heat treatment caused the volumetric contraction of the wood and the increase in the temperature used increased the quantity and/or size of the internal cracks of the pieces, which previously did not exist in the untreated wood, as can be seen in the regions indicated by the arrows in Figure 2 for the transversal cut of the wood, and in Figure 3 for the longitudinal cut. These cracks may have contributed to the density reduction and to the loss of mechanical resistance of the woods submitted to heat treatment.



Figure 2: Cross-section: (a) Untreated wood and (b) Heat-treated wood.



Figure 3: Longitudinal section: (a) Untreated wood and (b) Heat-treated wood.

It should be noted that the monitoring of wood fissures was not the subject of the present study, as well as the evaluation of mass loss and volumetric contraction of heat-treated wood. Therefore, the explanations offered are only indicative and, therefore, could only be proven by carrying out more specific studies on the themes. We emphasize that such analyzes contribute to the scientific qualification of the intensity of the heat treatment performed.

In general, the mechanical properties were negatively influenced after heat treatment. The mechanical properties of compressive strength and stiffness (f_{c0}, E_{c0}) , tensile strength (f_{t0}, E_{t0}) , and static bending (f_{M0}, E_{M0}) parallel to the fibers, showed significant reductions, since the property of shear strength parallel to the fibers (f_{v0}, E_{m0}) , presented strength gain for the first heat treatment temperature, followed by reductions for the other studied temperatures.

Reduction in the compressive strength property was also observed by Gunduz *et al.* (2009) for the wood of *Carpinus betulus*, where the greatest loss of strength recorded was 34 % for the treatment at 210 °C for 12 hours. The same was also observed by Korkut *et al.* (2008) for *Pinus sylvestris* wood, the greatest reduction occurred for treatment at 180 °C for 10 hours, 25,4 %. Gunduz *et al.* (2008) reported a maximum reduction of 27,2 % for the species of *Pinus nigra* treated, and Unsal and Ayrilmis (2005) reduction of 19,0 % for the species *Eucalyptus camaldulensis*, both for treatment at 180 °C for 10 hours.

Elaieb *et al.* (2015), observed similar reduction values of strength and stiffness to static bending parallel to the fibers for the species of *Pinus halepensis*, *Pinus radiata*, *Pinus pinaster* and *Pinus pinea*, heat-treated under a vacuum atmosphere at 230 °C, reductions of up to 50 % stiffness and up to 70 % strength.

When studying the species of *Pinus taeda* heat-treated in an electric oven with an inert atmosphere of nitrogen gas at 180 °C, Silva *et al.* (2013) observed a 25 % gain in shear strength for the treatment time of 30 minutes, and for wood treated for 120 minutes, no significant difference was found with untreated wood.

On the other hand, Moura *et al.* (2012) observed a reduction of 23,7 % for *Pinus caribea* wood heat-treated at 200 °C, for the other treatment temperatures studied (140 °C, 160 °C, and 180 °C) there were no significant changes in this property.

The reductions in mechanical properties presented by heat-treated wood are strictly related to the thermal degradation of the chemical constituents of the cell wall of the wood, especially hemicelluloses (Sundqvist 2004, Moura *et al.* 2012). In addition, as mentioned above, the cracks in the treated wood also influenced the reduction of these properties.

Unfortunately, there are no other studies on *Acrocarpus fraxinifolius* wood heat-treated in an autoclave, which makes it difficult to effectively compare the results, however, comparisons with different species and heat treatment processes are important to understand the results obtained.

CONCLUSIONS

The Indian cedar wood (*Acrocarpus fraxinifolius*) showed uniform browning after the heat treatment in an autoclave with water vapor and under pressure, allowing the use of the species for aesthetic and decorative purposes.

After the heat treatment, the wood showed small cracks that may have contributed to the loss of mechanical strength. The reductions in the mechanical properties of compressive strength, tensile strength, and static bending parallel to the fibers, make it difficult to use for purposes that demand high mechanical strength.

The observed reductions for mechanical properties were up to 53,5 % for strength and 57,4 % for stiffness compression parallel to the fibers; 80,2 % for strength and 52,9 % for stiffness parallel to the fibers; 73,5 % for strength and 67,0 % for stiffness static bending parallel to the fibers.

The strength to shear parallel to the fibers showed a gain of 64,3 % for heat treatment at 155 °C, for other temperatures there was a reduction of up to 55,9 %.

As for the chemical properties, the holocellulose content decreased significantly with the heat treatment, a reduction of up to 32,8 %. The total lignin content did not show significant changes for the heat-treated woods at 155 °C, 165 °C, and 175 °C, the maximum reduction was 22,1 %. There was a significant increase in the content of extractives, from 9,5 times to 14,5 times more than untreated wood.

It is recommended to carry out tests of color stability and biological resistance, for a better understanding of the influence and intensity of the heat treatment.

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