Thermal treatment in saturated vapour pressure for spruce

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ABSTRACT

Tests of thermal treatment in conditions of saturated vapour pressure, with different combination of time/temperature (from 140° to 180° C), were performed on spruce sawn timber from 30 to 200 mm thick. The objective was to study the influence of the combination of temperature and time (of exposition to a given temperature) on the colour and on the residual physical and mechanical properties of the treated wood.

INTRODUCTION

The thermally modified wood has been on the European market for 10-15 years, above all for exterior uses products because of its improved natural durability when treated at temperatures not lower than 212°C. Others favourable modifications concern the reduction of the equilibrium moisture content and the increase of the dimensional stability. Finally, the thermal treatment modifies the colour of wood which turns to dark brown-reddish depending on the temperature and time of exposition. On the other hand it is known that the thermal treatment reduces the mechanical properties of wood.

In Italy the colour modification makes the thermal process a very interesting process in the market of flooring and furniture even if, the high price of energy and of treatment plant has discouraged the enterprises with the result that up to now only a few industrial plants are working in Italy.

The thermal treatment is usually performed in special kilns where, in order to avoid fire risks and to reduce thermal oxidative degradation phenomena, the oxygen is substituted with vapour or (in some cases) with nitrogen. During a first phase of the process the wood is dried at 100-130 °C and the MC decreases to nearly 0%. Then, in the second phase the heat treatment takes place for some hours and finally the third phase is to lower the temperature and the MC is increased to 4-7%.

A third alternative is the process performed at high temperature and high pressure in saturated vapour conditions. This thermal treatment requires an autoclave plant corrosion and pressure resistant. This process does not dry the wood and for this reason it offers some advantages, above all for the treatment of large diameter timber which is not possible to treat with the super-heated thermal treatment because the drying phase produces high internal stresses and consequent degradations of the wood quality. Moreover, an industrial plant for saturated vapour treatment is probably less expansive than a superheated vapour plant. On the other hand the energy requirement is probably higher and is some country the use of plants having to do with pressure is restricted or requires special licences.

The paper reports results of tests of thermal treatment in saturated vapour conditions performed at CNR-IVALSA in a small laboratory plant on spruce sawn timber of different thickness. Different combination of temperature in the range from 140° to 180°C and time of exposition was used and the main physical and mechanical properties of the treated wood measured.
The objective was to study the influence of the variables on the residual properties of the material and to verify if it was possible to combine temperature and time of exposition in order to obtain a given target.

METHODS

The thermal treatment

The laboratory plant used for the tests (Fig. 1) is composed by a small stain steel autoclave (about 120 x 40 cm) with a double wall. The internal wall of the cylinder is 6 mm thick. The gap between the two walls is filled with diatermic oil heated with an electric resistance. The oil heats the internal walls of the cylinder and the water taking place on the bottom of the cylinder.
The wood pieces take place inside the cylinder with metal sticks among the layers of the stack.
The cap of the cylinder has a sealing-screw device which allows to keep the pressure within the cylinder in safe condition.

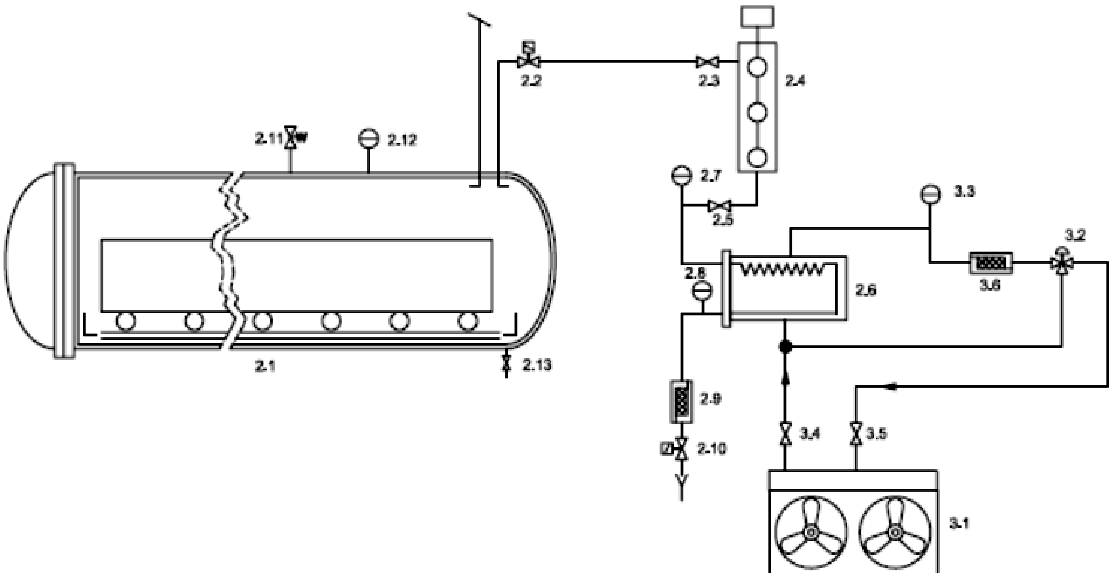


Figure 1: schematic of the plant

The treatment during the test has three phases: a warm-up phase needed to reach the temperature within the cylinder; a treatment phase at constant conditions for a given time; a cooling off phase, performed expelling the vapour and taking down the temperature to 100°C. At the beginning, before the warm-up phase, the air is extracted from the autoclave with a vacuum pump. The duration of the treatment (exposition time) is calculated since the pressure reaches the set value.
The saturated vapour conditions are reached keeping the water in the bottom of the cylinder at a given boiling temperature. In close conditions the boiling temperature and the saturated vapour pressure has an exponential relationships as showed in Fig. 2.

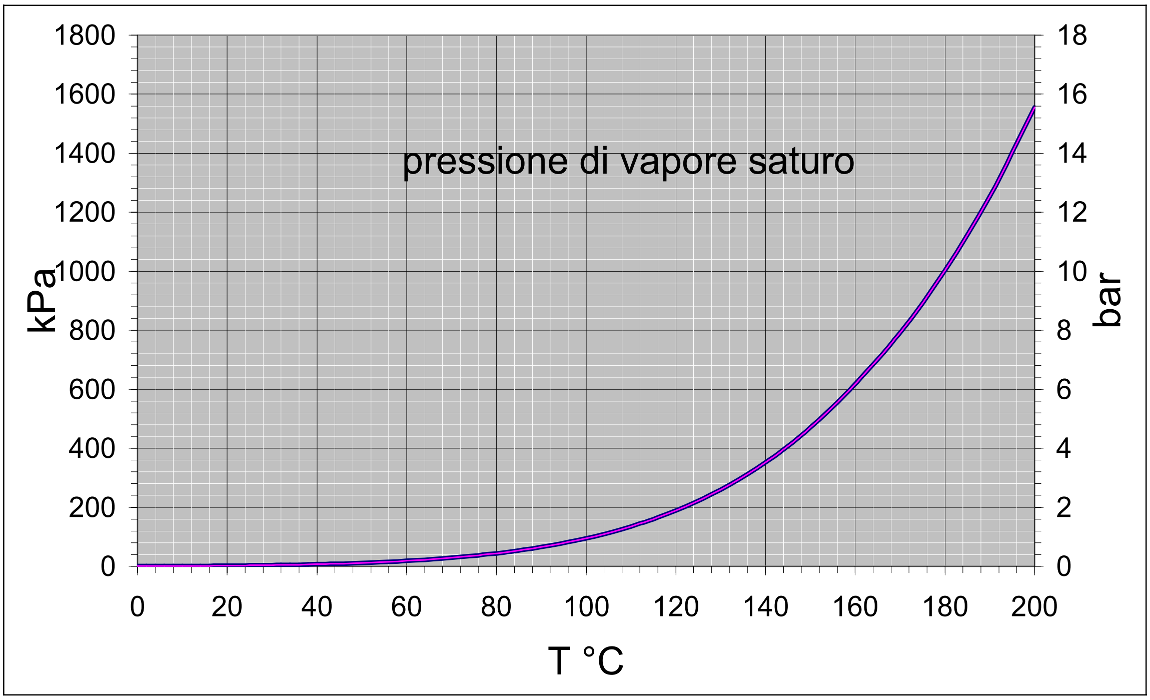


Figure 2: saturated vapour pressure vs. boiling temperature

The experimental procedure

Study of the colour

A first series of tests were focused on the study of the colour. The objective was to study the influence of the process variables on the colour at the surface and at the inner core. The colour of the modified wood was compared with the colour on new and aged spruce sawn timber.

The colour was measured with a spectrophotometer for visible light (700-900 ηm) with a spot probe with a 3 cm diameter at the surface and at the inner core after sawing of the treated piece.

About 120 specimens where treated in 20 tests. The tests were carried out treating the wooden pieces with different thickness and initial MC (*MCi*) at different combination of pressure-temperatures *(p)* and time of exposition *(t)* at a given pressure-temperatures.

- *s*: thickness (30, 200 mm)
- *t:* exposition time (test 1,2,4,6,12 hour long)
- *p*: pressure/temperature (2,4,6 bar)/(120,150,170 °C);
- *MCi*: initial MC grouped in 4 classes:I: <18%; II: 18-22%; III: 23-28%; IV: >28%.

Properties of the treated wood

Spruce sawn boards 200 X 10 x 3 cm (*MCi 15%*) were cut in two pieces. One piece for each board was treated at p/t=4/4. the other pieces were kept untreated as reference cases. Physical and mechanical properties was measured on small sound wood oriented samples cut from treated and on reference specimens. Mechanical properties measurement was performed using a laboratory testing machine. All the tests physical and mechanical were performed according to EN standard rules.

Density, shrinkages and swellings was measured on 120 specimens 2 x 2x 4 cm (60 specimens treated, 60 specimens untreated) equilibrated at 20° C and 100%, 76%, 65%, 24% RH and oven-dry.

Bending strength was calculated on 60 specimens 2 x 2 x 34 cm (30 specimens treated, 30 specimens untreated) from results of 3 point loading bending tests using the following equation:

$$\sigma_{\max bending} = \frac{3P_{\max} l}{2bh^2};$$

Apparent young modulus E was calculated from the stress strain curve using the following equation : $E_{m,app} = \frac{1}{4}\left(\frac{F_2 - F_1}{W_2 - W_1}\right)\frac{l^3}{bh^3}$;

Compressive strength in the grain direction was calculated on 60 specimens 2 x 2 x 4 cm (30 specimens treated, 30 specimens untreated) using the laboratory testing machine using the following equation $\sigma_{\max comp.} = \frac{P_{\max}}{ab}$;

Because of the different sorption curve of treated and untreated specimens the mechanical properties measured (both E and σ) was corrected to the normal MC value (12%) with the following:

$$E_{12} = E_w \frac{E_w}{1+\alpha(W-12)}; \quad \sigma_{12} = \sigma_w[1+\alpha(W-12)].$$

RESULTS

Colour

Differences between new and aged wood

The characteristic values of colour coordinate measured in new and in aged wood (target colour) are reported in the Table 1.

Table 1: Characteristic colour coordinate measured in new and old Spruce wood

	not treated wood (new)			target wood (old) surface (e)			target wood (old) inner core		
coordinate	L*	a*	b*	L*	a*	b*	L*	a*	b*
average	**85,34**	**3,81**	**20,06**	**49,1**	**5,6**	**15,2**	**77,0**	**8,0**	**25,7**
st. dev.	1,44	0,71	1,03	4,8	1,2	1,4	2,4	1,1	1,4
CoV %	1,69	18,63	5,15	9,7	20,6	9,2	3,1	13,6	5,6

The data reported in the table 1 means that the natural ageing:

- turns the colour of wood to reddish colour;
- darkens the wood, above all on the surface;
- turns the colour to grey;
- the ΔE between aged and new wood ranges from 11 in the inner core to 37 on the surface.

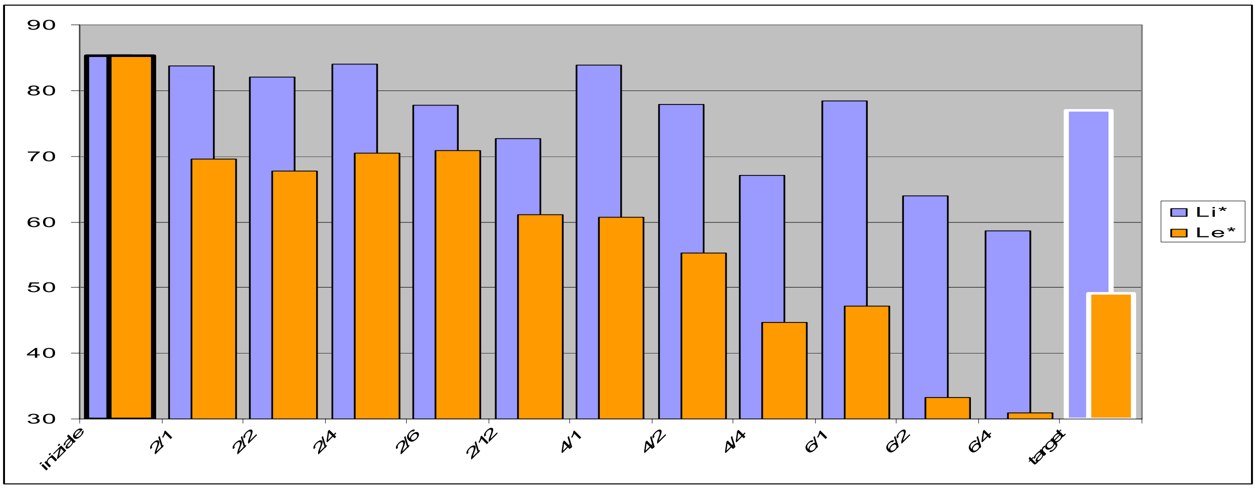
Global influence of variables on the colour

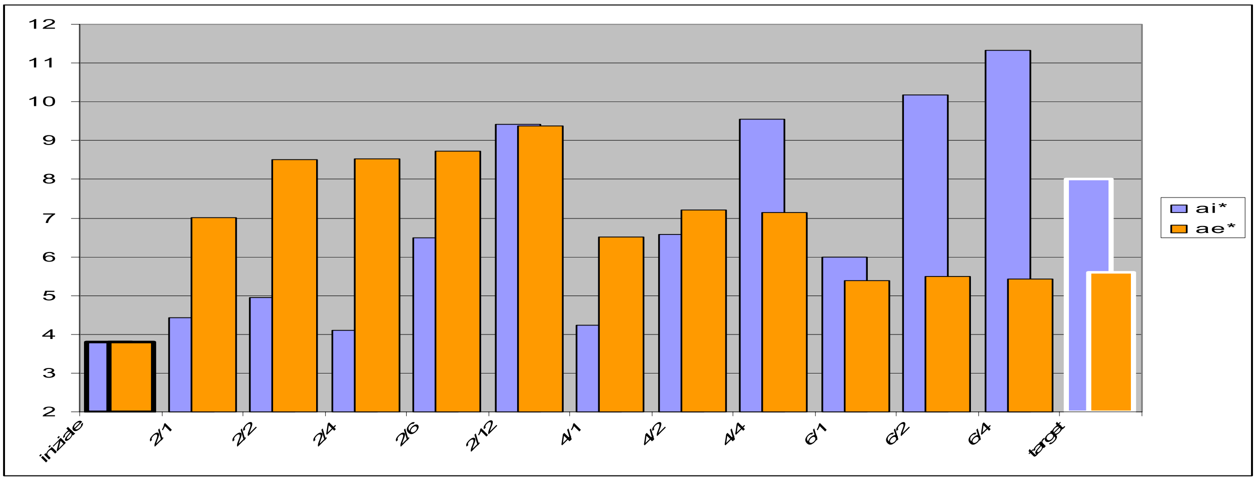
In the Table 2 the global influence of the process variables pressure (bar) and temperature (°C) in terms of ΔE between treated and inner core ΔEe and surface ΔEi of aged wood are reported.
The variable initial MC did not influence the colour modification and it is neglected in the results description.

Table 2: average surface and inner core ΔE between aged and treated specimens with different p/t combinations

average ΔEe			average ΔEi		
p/t		*ΔEe*	*p/t*		*ΔEi*
4	*4*	4,9	*2*	*6*	2,6
6	*1*	5,0	*4*	*2*	3,5
4	*2*	8,6	*2*	*12*	4,5
4	*1*	13,1	*6*	*1*	5,1
6	*2*	13,2	*2*	*2*	6,9
2	*12*	15,8	*4*	*1*	8,7
6	*4*	18,0	*6*	*2*	8,9
2	*1*	22,9	*2*	*1*	9,0
2	*2*	23,0	*2*	*4*	9,2
2	*4*	23,9	*4*	*4*	10,5
2	*6*	24,6	*6*	*4*	19,3

The influence of different treatments, identifies by different p/t combinations are reported in the Fig. 3 a-c.





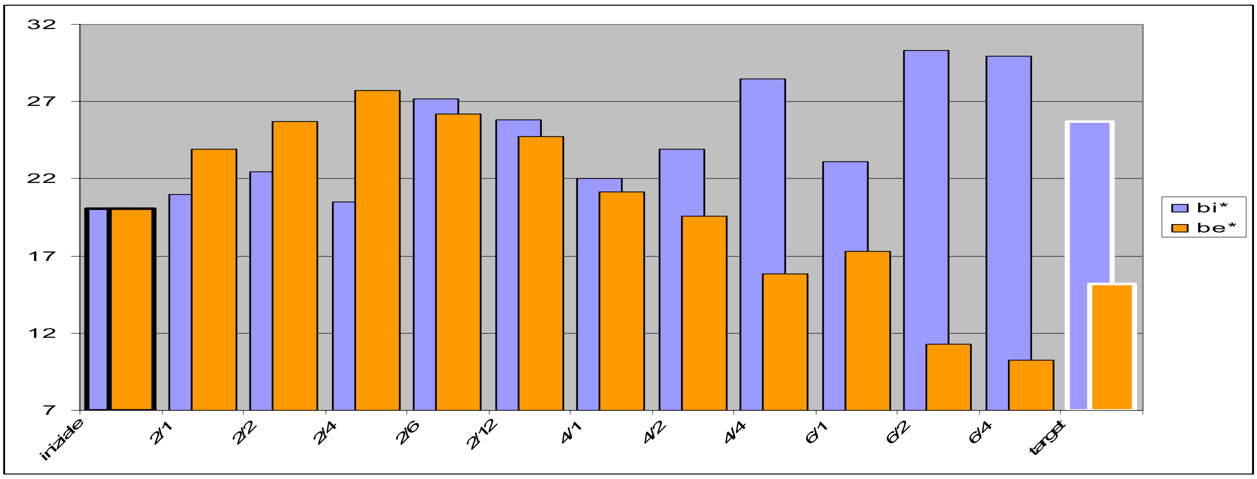


Figure 3 a-c: average colour coordinates of the surface (blue) and inner core (orange) of the new wood (left) aged (right) and treated wood under different p/t combinations

The results indicates that the treatment at the p/t conditions lower or equal to 4/2 keeps the colur lighter than the target colour while p/t conditions equal or higher to 6/1 makes the colour darker than the target colour. After 3 hours of exposition time at a treatment conditions of 6 bar (160 °C) the surface of the wood starts to char.
The conclusion of the tests suggests that the best p/t combination to reproduce target colour of aged wood was 4/4.

Physical properties
The density of specimens conditioned at 20 °C RH 65% is 449 kg m^{-3} for untreated specimens and 419 kg $m^{-3}$for treated specimens. The difference (of about 6,8%) has a statistical relevance.
The treatment modifies both the sorption curves (Fig. 4) of wood and the shrinkages coefficients (Table 3).
The absorption of water is reduced in treated specimens in the MC range 0- 12%. On the other hand it can be observed that in conditions of saturated vapour pressure UR 100% the EMC of treated wood, corresponding to the FSP is much more high than in untreated wood. This phenomena is not explained yet.
However, no matter how the EMC is, the dimensional stability is always increased as well as the ratio and difference between radial and tangential shrinkages.

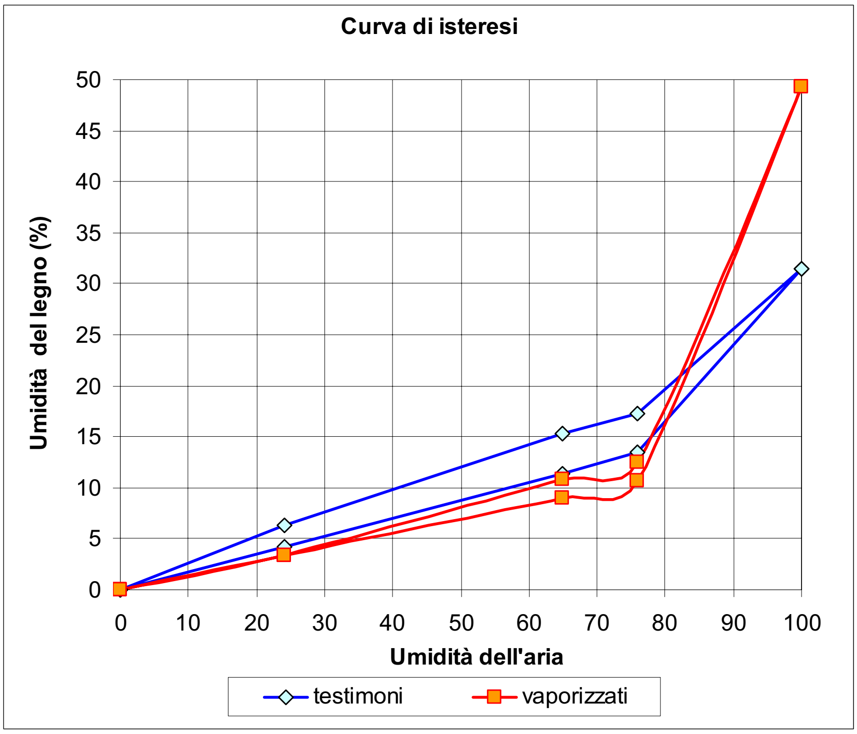


Figure 4: sorption curves (absorption and desorption) of untreated (blue) and treated (orange) wood

Table 3: shrinkages in radial (r) and tangential (t) direction of treated and untreated specimens equlibrated at different UR%

		untreated					treated					differences %			
ur %	T °C)	MC (%)	βr (%)	βt (%)	βt/βr	βt-βr	MC (%)	βr (%)	βt (%)	βt/βr	βt-βr	βr	βt	βt/βr	βt-βr
100	***20***	31,4	0,00	0,00	-	-	49,2	0,00	0,00	-	-				
76	***20***	17,2	1,62	4,08	2,55	2,47	12,5	1,56	3,11	2,10	1,54	3,3	23,9	17,5	37,5
65	***20***	**15,3**	**1,99**	**4,78**	**2,42**	**2,79**	**10,8**	**1,99**	**3,73**	**2,01**	**1,74**	**0,2**	**21,9**	**16,9**	**37,4**
24	***24***	6,4	3,81	7,92	2,09	4,11	3,4	3,37	6,11	1,88	2,73	11,4	22,8	10,1	33,5
0	***103***	0,0	5,92	9,81	1,67	3,88	0,0	5,36	7,42	1,42	2,06	9,5	24,3	14,7	47,0

Mechanical properties

The differences of measured mechanical properties between treated and untreated specimens are reported in Table 4. In Table 5 the corrected value, taking into account the difference of EMC between treated and untreated specimens are reported.

It can be observed that the mechanical properties are slightly reduced. This reduction is very probably related with the density decay.

Table 4: average mechanical properties of untreated and treated specimens

	untreated	*treated*	*Difference (%)*
$E_{apparent}$ (GPa)	10,4	9,2	11,5
$б_{max}$ bending (MPa)	81	66	19,1
$б_{max}$ compression (Mpa)	43	39	8,8

Table 5: average mechanical properties of untreated and treated specimens with values corrected to MC = 12%

	untreated	*treated*	*Difference (%)*
$E_{apparent}$ (GPa)	11,1	9,2	17,0
$б_{max}$ bending (MPa)	86	66	23,3
$б_{max}$ compression (Mpa)	47	39	17,4

CONCLUSION

The paper explored the thermal modification process of spruce sawn timber with the use of high temperature in saturated vapour conditions.
The measured variations of the wood characteristics treated at 4 bar- 150 °C for 4 hours are summarized below:

- Elastic modulus ($E_{bending}$) - 10%
- Compressive strength ($\sigma_{max\ comp.}$) - 9%
- Bending strength ($\sigma_{max\ bending}$) - 19%
- Swelling in the RH range 24-76% ($\alpha_{R,T}$) - 30%
- shrinkage in the RH range 24-76% ($\beta_{R,T}$) - 23%
- anisotropy ratio (RH 65%) (β_T / β_R) - 17%
- angular shrinkage (RH 65%) ($\beta_T - \beta_R$) - 37%
- density ($\rho_{12\%}$) - 7%

The results show that this treatment can be used to modify the colour of wood and to increase the dimensional stability. The process is suitable to operate at a temperature in the range 140 -160 °C. At temperatures lower than 120 °C the colour change requires a lot of time, it depends on the initial MC and is however often negligible. At temperature higher than 180°C the pressure rises at 10 bar and it probably increases the cost of the plant. Moreover, at that temperature we observed charring phenomena at the surface. It excludes the use of this process to improve the durability, because it needs temperatures higher than 210°C.
The process does not dry the wood. We observed that after a treatment at 150°C for 4 hours the surface of wood goes at a MC of about 15% while the MC at the core does not change. This makes the treatment advisable to treat large thickness timber which cannot be treated with superheated vapour.
The density and mechanical properties are reduced by the thermal treatment. The measured reduction is however not high and it probably can be neglected in solid wood beams or large section elements where the limiting factor is given by knots or other defects. This makes the process suitable for the treatment of large section solid wooden elements, for joinery, carpentry, flooring etc.

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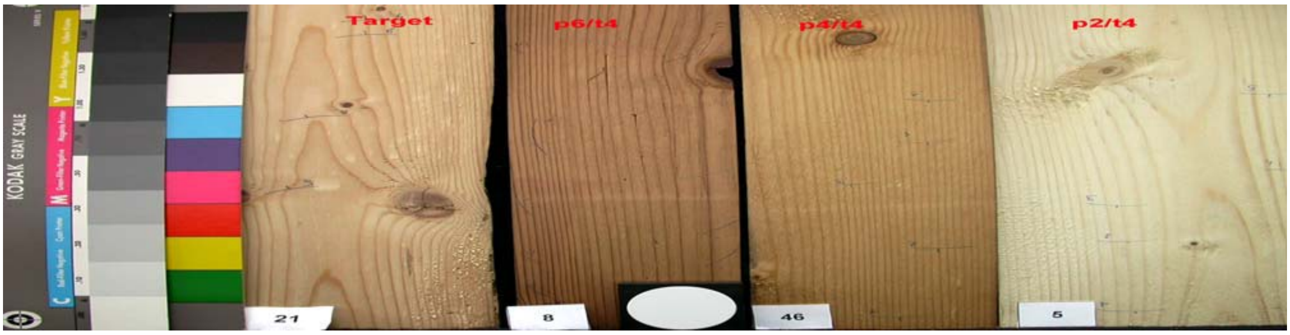
ANNEX

a

b



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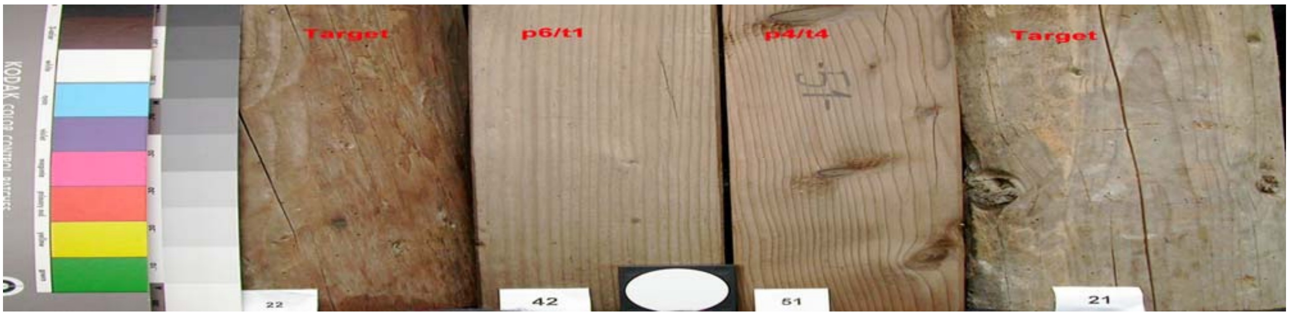

d

Figure 5 a-d: comparative pictures of colour of wood treated with different p/t combinations and aged wood (on the left, named "target"). First two pictures: inner core. Last two pictures: surface

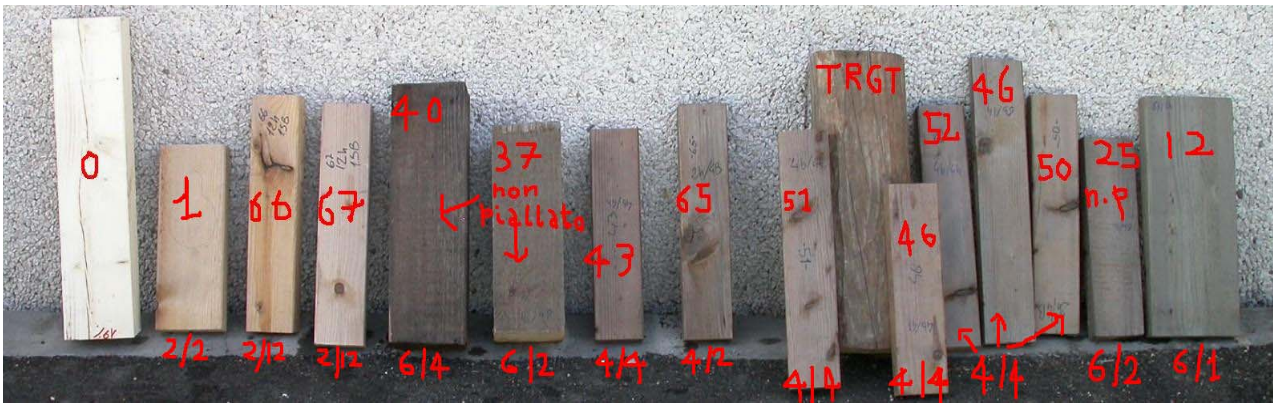

Figure 6: exposition of pieces treated with different p/t combiantion.