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X-ray Computed Tomography Based Estimation of Charring Around Knots in Sawn Timber

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ABSTRACT: Charring in knot regions of wood remains understudied, leaving a knowledge gap in charring behavior characterization. This research aims to study the charring behavior in and around knots in Scots Pine and Norway Spruce using computed tomography (CT) scanning. To achieve this, we quantified the impact of extractives and fiber orientation on charring rates and depths. Python codes were developed to handle image processing and charring depths calculations. This paper discusses the experimental setup, data processing techniques, and the implications of extractives content in the charring behavior of wood. The conducted experiments revealed that there is probably a correlation between higher extractive content in knot wood correlates with lower charring rates and reduced hardness pre- and post-burning.

KEYWORDS: charring, knots, computed tomography, extractives, hardness.

1 – INTRODUCTION

Knots, which result from the inclusion of branches in a tree's growth, introduce localized fiber deviation and variations in extractives content that can significantly affect the wood's overall properties. These knots create spatially heterogeneous regions within the wood, leading to variations in charring patterns during combustion [1]. Such heterogeneity may not only influence the material's mechanical and thermal properties but also its susceptibility to degradation under high temperatures. The understanding and measurement of these variations in charring are critical for applications where wood is exposed to fire or high-heat conditions, such as in construction, fire safety engineering, and wood preservation.

Traditional methods for assessing charring, such as visual inspection and destructive sampling, are limited in their ability to provide detailed, spatially resolved information about the 3D distribution of charred material. These methods often fail to capture the complexity of internal variations, especially in regions with knots, which can have a significant impact on the charring behavior. Moreover, they tend to focus on superficial damage and may overlook deeper structural changes that could affect the wood's performance in fire scenarios.

To address these challenges, this study employs computed tomography (CT) scanning, a non-destructive imaging technique that produces high-resolution 3D images of the wood's internal structure, including regions near and within knots. CT scanning offers a powerful means of investigating the heterogeneous nature of charring, enabling the visualization and quantification of charred areas without compromising the integrity of the specimens [2]. By using CT images, this study aims to provide new insights into how extractives in knot and heartwood affect the charring process and how these variations influence the wood's thermal degradation.

In this study, the impact of extractive on the charring rate was evaluated in samples impregnated with these substances, comparing them with untreated samples subjected to the same burning conditions. Hardness testing was performed to assess any changes in the

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mechanical properties of the wood before and after the burning process. Additionally, an image processing and analysis method was developed to process CT scan data, allowing for the detailed quantification of charring in specific regions of the wood, with a particular focus on areas surrounding the knots. This integrated approach not only enhances our understanding of the charring process but also provides a more accurate, non-destructive method for studying the effects of extractive treatments on wood's fire behavior.

2 – MATERIALS AND METHODS

2.1 IMPREGNATION TEST METHOD

Wood samples of Scots Pine and Norway Spruce (50g each) were first treated to isolate extractives - including sugars, phenols and other organic compounds - from the knots and heartwood using a Soxhlet extractor Fig.1. The extraction was carried out with a solvent mixture of 80% acetone and 20% water until the extractives were fully separated. The resulting extractives were then concentrated using a rotary evaporator to remove the solvent, leaving behind the extractive compounds. These extractives were then used to impregnate the wood samples. Impregnation was performed under controlled conditions in an autoclave – 15 bar for 1 hour - Fig.2, ensuring complete absorption of the extractives into the wood.

Figure 1: Soxhlet extraction setup

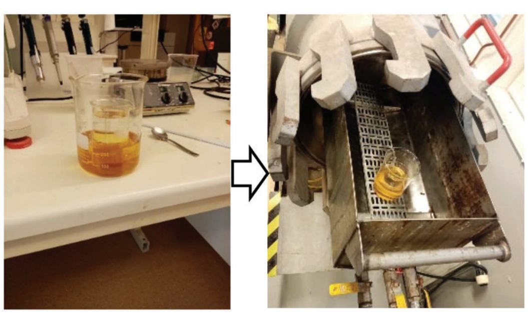

Figure 2: Steps of the impregnation and burning process of wood samples

After impregnation, both treated and untreated wood samples were subjected to a burning process. The samples were placed on a steel cuboid in a furnace preheated to 580°C, Fig.3. They remained on the metal surface for 10 minutes, allowing charring to occur. To halt the charring process, the samples were immediately immersed in water. Post-burning, all samples were conditioned in a chamber to achieve a stable moisture content of 10%.

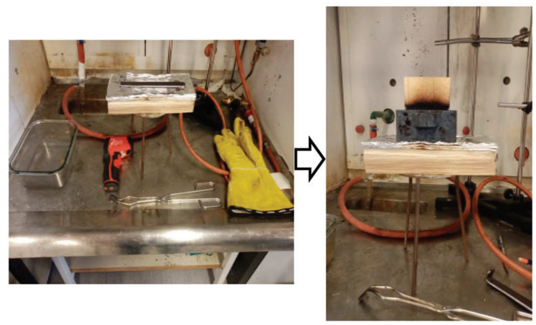

Figure 3: The burning process setup

Before and after the burning procedure, all wood samples, both impregnated and untreated, were scanned to assess changes in their morphological structure providing a detailed comparison of the effects of impregnation and burning. High-resolution imaging was performed using an industrial CT scanner, which, with voxel dimensions of 0.3 mm × 0.3 mm × 0.3 mm, offered significantly finer detail than the medical scanner's 0.5 mm × 0.5 mm × 0.5 mm voxels, Fig.4.

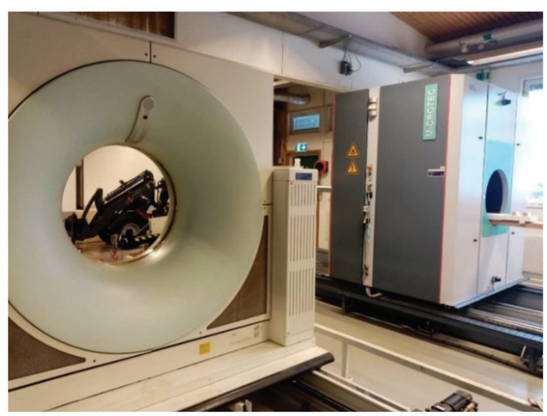

Figure 4: Simens medical scanner on the left and Microtec industrial scanner on the right

2.2 HARDNESS TEST METHOD

Table 1: Characteristics of the wood samples used in the burning test and hardness test

Groupe's name	Knots' type	Knots' direction	Species
BNS	Live knots	Perpendicular to the surface	Norway Spruce
BSP			Scots Pine
BNS'		Parallel to the surface	Norway Spruce
BSP'			Scots Pine

Four groups of 3 wood samples - Scots Pine and Norway Spruce, categorized by knot and fiber orientation - were tested, Tab.1 & Fig.5.

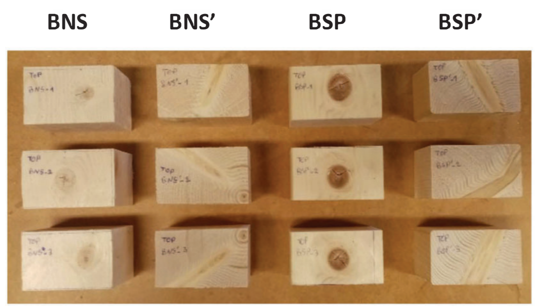

Figure 5: the hardness test wood samples before burning

This test consists in three major parts. First, the hardness of the samples was measured before burning. Second, the samples were burned using the same method previously described. And finally, the hardness of the charred layer was measured.

All samples were scanned pre- and post-burning to provide a detailed comparison of hardness changes and to analyze any variations in wood structure, particularly around knots, before and after the charring process.

To assess the changes in wood hardness due to charring, the samples underwent a depth-controlled loading test following the Janka method, with specific adjustments to minimize surface destruction Fig.6. The test consisted in pressing a steel sphere into the wood to a depth of 0.32 mm at a rate of 5 mm/min, applying maximal force for 0.1 seconds, and then unloading at 5 mm/min [3]. This method was applied to samples both before and after burning to evaluate the impact of charring on the material's hardness.

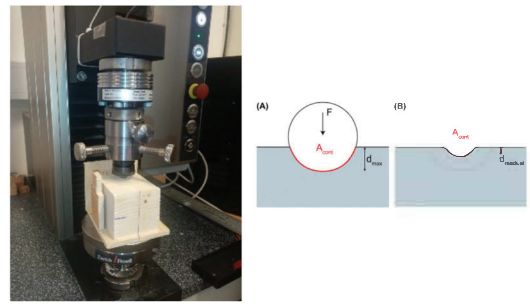

Figure 6: hardness test setup

3 – ANALYSIS

3.1 CT DATA ANALYSIS

The scanned files were opened and processed in 3D Slicer. To measure the depth of the charred layer, voxels within the char density range were thresholded, isolating the charred layer into a separate file, Fig.7.

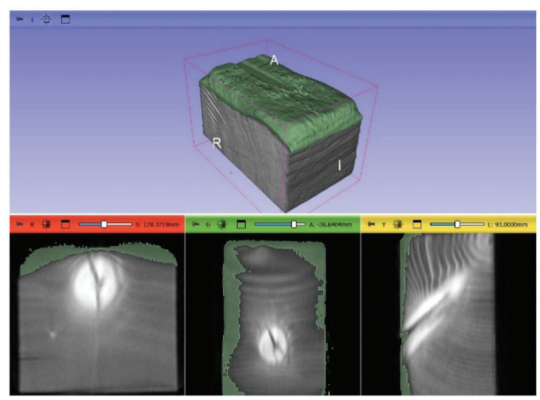

Figure 7: the automatic isolation of the char layer on 3D Slicer

The regions in and around the knot were manually segmented within the same software and saved in a different file, Fig.8.

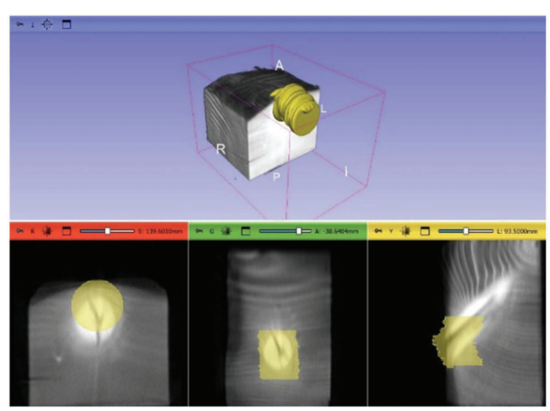

Figure 8: the manual isolation of the knot on 3D Slicer

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A Python script was written to convert both files into binary format. The script then performed two operations: subtraction of the knot wood region from the charred layer to isolate the charred regular wood, and intersection of the two files to obtain the charred knot wood , Fig.9.

These operations were applied to each slice across the stack. Finally, the charring depth was calculated by averaging the values across all slices of the resulting file.

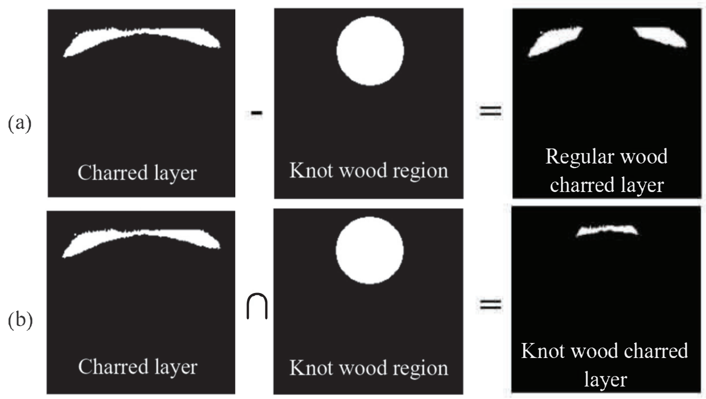

Figure 9: (a) regular wood charred layer resulting from the subtraction operation; (b) knot wood charred layer resulting from the intersection operation

3.2 HARDNESS TEST ANALYSIS

On each sample, the hardness values of 18 points were tested, and force VS standard travel/displacement curves were drawn. These curves, with pre-burning and post-burning tests, were used to draw stress VS displacement curves Fig.10, which were used instead of stress VS strain curves because calculating strain was impractical due to localized superficial compression. The pseudo coefficient Z was designed and calculated, and the ratio of Z1 - loading phase (MN) slope - to Z2 - unloading phase (PQ) slope – Eq. (1) was mapped to see if there is a correlation with the presence of knots and to compare hardness property before and after the formation of the char layer.

$$\frac{E1}{E2} = \frac{\frac{\Delta\sigma 1}{\Delta\varepsilon 1}}{\frac{\Delta\sigma 2}{\Delta\varepsilon 2}} = \frac{\frac{\Delta\sigma 1}{\Delta\frac{l1}{L}}}{\frac{\Delta\sigma 2}{\Delta\frac{l2}{L}}} = \frac{\frac{\Delta\sigma 1}{\Delta l1}}{\frac{\Delta\sigma 2}{\Delta l2}} = \frac{Z1}{Z2} = \frac{loading\ slope}{unloading\ slope} \quad (1)$$

With:

- E the punctual modulus of elasticity
- σ the stress
- Ɛ the strain
- Δl the displacement (standard travel)
- L the thickness of the sample

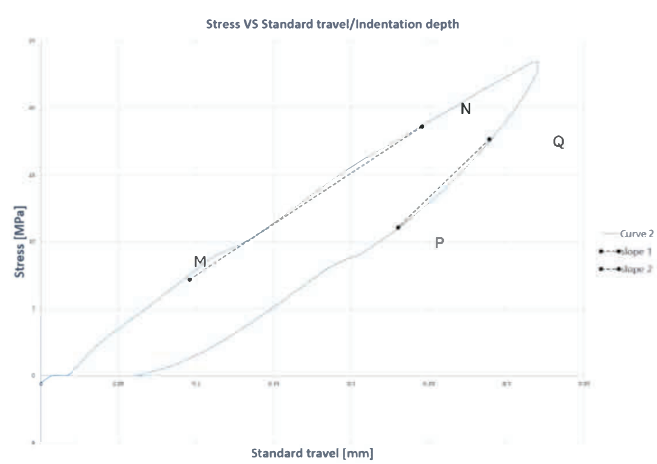

Figure 10: Stress VS Standard travel/displacement curve of a typical testing point

4 – RESULTS

4.1 RESULTS OF THE EXTRACTION PROCESS:

Table 2: the impregnation test percentages of extractives in knot wood and heartwood of Scots Pine and Norway Spruce

		Percentage to dry mass	Ratio
Scots Pine	Knot wood	33.09%	18.89%
	Heartwood	6.25%	
Norway Spruce	Knot wood	7.29%	18.07%
	Heartwood	1.32%	

Higher extractives in knot wood: Both Scots pine and Norway spruce have a significantly higher percentage of extractives in knot wood compared to heartwood:

- Scots Pine: Knot wood (33.09%) vs. Heartwood (6.25%)
- Norway Spruce: Knot wood (7.29%) vs. Heartwood (1.32%)

The ratio of extractives in heartwood compared to knot wood is quite similar for both species, around 18%.

4.2 RESULTS OF THE BURNING TEST (IMPREGNATION METHOD):

Table 3: Averages of charring rate/depths of the untreated and the impregnated samples

	Char depth mm	Charring rate mm/min
Untreated samples	2.36	0.236
Impregnated samples	2.09	0.209

The mean value of the charring rate in the impregnated samples is 0.209 mm/min, and in the normal samples, it is 0.236 mm/min. The impregnated samples showed a decrease of 11.44% in the charring rate.

4.3 RESULTS OF THE BURNING TEST :

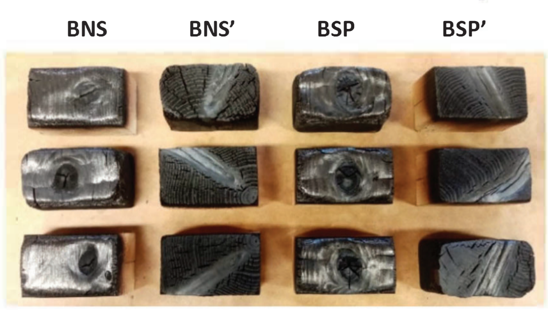

Figure 11: the hardness test wood samples after burning

Macro-comparison between sets:

BNS: post-burning, these samples showed some cracking along the grain lines.

BNS': after burning, vertical cracks and deeper fissures can be seen, but the overall structure remains relatively intact.

BSP: post-burning, there are obvious cracks, suggesting a different burn pattern due to the fiber orientation.

BSP': these samples exhibit deep vertical cracks after burning, indicating the influence of the vertical fiber orientation on the burning behavior.

Table 4: Averages of charring rate/depths of each of the 4 sets of samples

	Char depth in mm		Charring rate in mm/min	
	regular wood	knot wood	regular wood	knot wood
BSP	4.39	1.71	0.439	0.171
BSP'	3.17	1.53	0.317	0.153
BNS	4.57	1.70	0.457	0.170
BNS'	3.82	1.34	0.382	0.134

The burning of the 4 sets of samples highlighted varying charring rates influenced by wood structure and knot orientation. Samples with perpendicular knots and horizontal fibers (BNS and BSP) exhibited higher charring rates compared to those with parallel knots and vertical fibers (BNS' and BSP'). Overall, knots demonstrated greater resistance to charring than regular wood, Tab.4.

4.4 RESULTS OF THE HARDNESS TEST:

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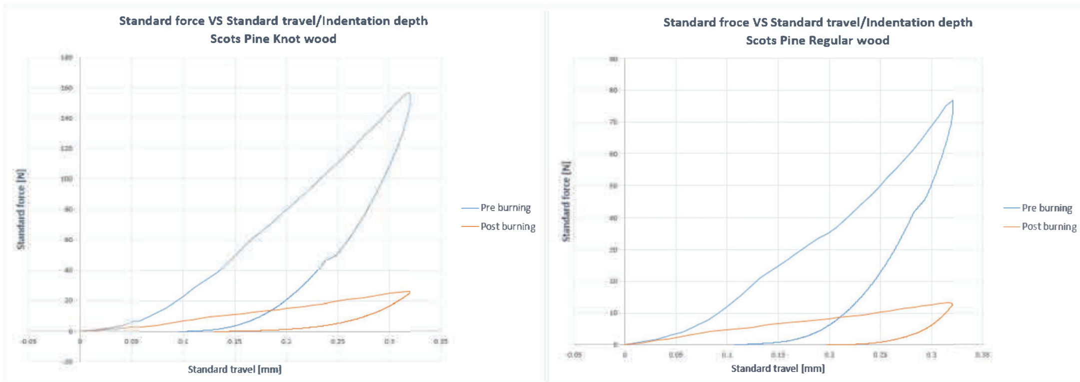

Figure 12: Graphs of Standard force VS Standard travel curves pre-burning and post-burning in knot wood – to the left - and in regular wood – to the right - on one testing point. BSP' sample.

Interpretation

- **Pre-Burning:**

Knot wood: Shows high resistance to indentation, with a steady increase in force with depth. Exhibits elastic recovery with minimal permanent deformation.

Regular wood: Also shows increasing force with depth but requires less force than knot wood. Exhibits elastic recovery with slightly more permanent deformation compared to knot wood.

- **Post-Burning:**

Knot wood: Becomes much softer, showing very low force values and reduced resistance to indentation. Displays less elastic recovery and more permanent deformation.

Regular wood: Similarly becomes softer, with significantly lower resistance to indentation. Shows reduced elastic recovery and increased permanent deformation.

The method previously explained was used to draw maps of the ratio $\frac{Z1}{Z2} = \frac{Loading\ slope}{Unloading\ slope}$ of the stress vs. indentation depth graph on each point on the samples' surfaces before and after burning , Fig.13.

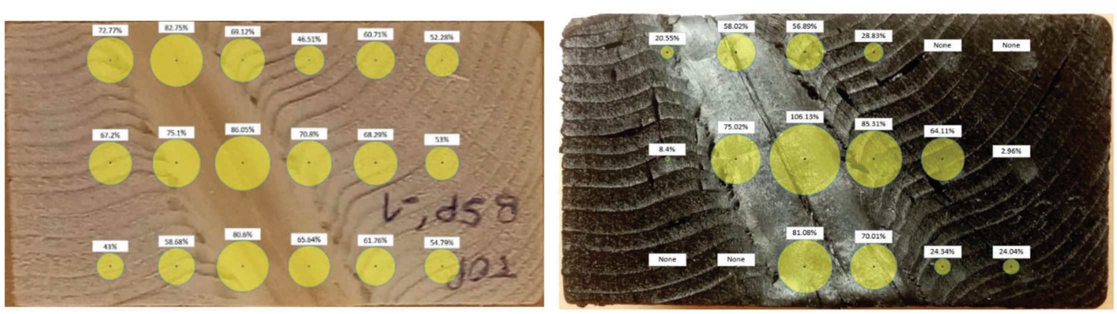

Figure 13: the map of the ratio Z1/Z2 on a BSP' sample pre- and post-burning

High ratios: Indicate minimal difference between loading and unloading slopes, suggesting greater elastic recovery. Observed mostly in and around knot wood.

Low ratios: Indicate a significant difference between loading and unloading slopes, suggesting more permanent deformation and a plastic response. Observed primarily in regular wood, especially at the sample edges.

The same tendency was observed on the burnt samples but with lower hardness values.

The points to which the annotation "None" was assigned showed a negative slope on the Stress VS Standard travel graph at the loading phase which is not normal and may be caused by the imperfection of the test apparatus designed.

Another possible reason to this is that when the load is applied on wood char, the internal structure slightly collapses until it becomes dense enough to exhibit a more uniform and predictable behavior.

5 – DISCUSSION

The series of experiments conducted on Scots Pine and Norway Spruce wood samples, including extractives content and charring rate analysis and hardness tests, possibly illustrate a potential interplay between wood structure, chemical composition, and mechanical properties.

The extractives content analysis provided a chemical perspective on these observations. By the extraction procedure, it was found that knotwood contains significantly more extractives compared to regular wood.

The experiment of impregnation has shown that the charring rate in the normal samples is greater than that of the impregnated samples.

This observation is the absolute opposite to the initial hypothesis supposed during the impregnation procedure design.

Initially, it was expected that the impregnation process would result in a higher charring rate for the impregnated samples compared to the untreated samples. This expectation was based on the assumption that the higher concentration of organic compounds, such as sugars and phenols, within the impregnated samples would accelerate the charring process.

While the exact mechanism remains unclear, this outcome might be explained by two hypotheses:

- The formation of an insulative layer: The presence of more extractives could fuel the formation of a thicker char layer, which acts as an insulating barrier protecting the wood underneath from further pyrolysis and combustion.

- Increased density due to impregnation: The impregnation process increased the density of the wood. Higher density could affect the outcome of the burning experiment and slow down the charring rate.

The burning procedure showed how different wood structures and configurations react to high temperatures. Samples with perpendicular knots and horizontal fibers, such as Norway spruce (BNS) and Scots pine (BSP) exhibited high charring rates. In the other hand, configurations with parallel knots and vertical fibers (BNS' and BSP') lower charring rates. The lower charring rates for knotwood in both Scots Pine and Norway Spruce compared to regular wood may indicate a potential impact of extractives and/or density on fire resistance.

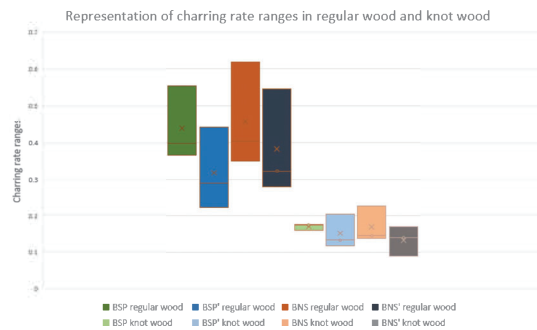

Figure 14: Boxplot of the charring rate ranges in regular wood and knot wood categorized by sets of samples

The charring rates in regular wood are more variable than those in knot wood suggesting that the charring rate of regular wood is less predictable, which can confirm the stability of knotwood under high temperature.

Hardness tests conducted on the 4 groups before and after burning offered a mechanical approach. Before burning, knotwood exhibited less inelastic permanent deformation compared to regular wood. This may be an important property for applications where the wood is subject to shallow indentation. However, after burning tests showed a significant decrease in hardness for knotwood and regular wood, with a more permanent deformation in regular wood. These results can, possibly, be explained by the high charring depth in regular wood compared to knot wood and the surrounding areas. When burned, regular wood regions are softer because of the thickness of the char layer and the huge loss of matter leading to a very fragile charred surface with weak internal bonds. As the stress goes higher, these areas collapse in the inside, leading to higher plastic deformation. As for the charred wood in the knots and in the surrounding areas, when undergoing stress, these areas are likely to return closer to the initial position demonstrating a less inelastic permanent deformation.

This research used different perspectives to approach the charring behavior starting from direct charring quantification and the chemical analysis to hardness testing pre- and post-charring. However, it highlighted many sides that can be improved to enhance the effectiveness of future works:

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Extraction procedure improvements: Usage of Dichloromethane to extract lipophilic molecules (resins, fatty acids…).

Impregnation procedure improvements: Isolating the effect of density from that of the extractives by adding a 3rd group of samples impregnated with a neutral substance.

Burning procedure improvements: Usage of an iso standard burning method, ideally, by employing a cone calorimeter.

7 – CONCLUSION

This study explored the charring behavior of wood, particularly around knots, using X-ray CT scanning to quantify the charred layer with high accuracy. It was found that knotwood chars at a slower rate than regular wood, likely due to its higher extractive content, which may create a protective barrier against charring. However, it remains uncertain whether this effect is primarily due to the extractives themselves or the increased density of knotwood. Additionally, the orientation of knots and fiber direction significantly influenced the charring rate, with perpendicular knots and horizontal fibers charring faster. These results may be influenced by the non-standard burning method used. Before and after burning, knotwood exhibited less permanent deformation compared to regular wood, while both types experienced a reduction in hardness post-burning. Overall, the findings suggest a potential interplay between wood structure, chemical composition, and mechanical properties, with the initial higher extractive content in knotwood correlating with reduced plastic deformation and improved fire resistance. Further research is needed to clarify these interdependencies.

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