Effect of Heat Treatment and Cryogenic Treatment on Chemical Components of Rowan (*Sorbus torminalis* L.) Wood

Ömer Özyürek

This study investigated the changes in the chemical composition of Rowan (*Sorbus torminalis* L.) when subjected to heat treatment (HT) and cryogenic treatment (Cr). Rowan wood was treated with HT at two different temperatures (190 and 212 °C) and times (1 and 2 hours). Subsequently, Cr was applied to some of the HT samples and a control sample (Control), creating a total of 8 experimental variations. The content of lignin, holocellulose, alpha-cellulose, ash, and the solubilities of hot water, 1% NaOH, and acetone, were determined. The control and the samples treated with HT and Cr showed differences in most of the investigated properties. For example, the amount of lignin in the samples increased as the HT temperature and time increased (maximum increase of 94.5%). However, Cr was found to limit this increase. Hemicelluloses were the most degraded components during HT, while the degradation of alpha-cellulose was much more limited. In contrast, Cr slightly increased the amounts of hemicelluloses and alpha-cellulose in the heat-treated samples. While HT (212 °C, 2 h) increased the amount of extractives by 2.3 times, Cr caused a decrease in the Control, but greatly reduced this increase in other samples.

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Contact information: Vocational School of Forestry, Düzce University, 81620, Düzce, Türkiye; omerozyurek@duzce.edu.tr

INTRODUCTION

The chemical composition of a wood species depends on many factors, including habitat and age, geographic location, climate conditions, and silvicultural interventions. The chemical composition of each wood species exhibits unique and specific characteristics. Generally, there are notable differences between deciduous and coniferous trees.

Differences in chemical composition are observed even within the same wood. These differences are seen between different parts of the wood, such as the heartwood and sapwood, and the earlywood and latewood. The heartwood of softwoods contains more extractives and less lignin and cellulose than the sapwood. This difference is generally small in hardwoods. The amount of cellulose is higher in the latewood of trees than in the earlywood, while the amount of lignin is lower. This is because the cell walls, which have a higher cellulose content, are thicker in the latewood, and the middle lamella, which has a higher lignin content, is thinner.

The macromolecular components of the cell wall in hardwood and softwood are cellulose, hemicellulose, and lignin. While cellulose exhibits a similar structure in all wood species, the chemical compositions and proportions of lignin and hemicelluloses differ between hardwood and softwood. In trees growing in mild temperate zones, these components constitute 97 to 99% of the wood, while in tropical trees, this value can drop to an average of 90%. Polysaccharides make up approximately 65 to 75% of the wood. Softwood and hardwood also differ in terms of their extractive compounds. Each wood species' extractive compound composition exhibits specific characteristic compounds. Volatile compounds are present in certain proportions of softwoods, while they are generally absent in hardwoods (Fengel and Wegener 1989). Another difference is in the amount of lignin. In hardwood, the amount of lignin ranges from 16 to 25%, while in softwood it ranges from 23 to 33%. In carbohydrates, the most significant difference is observed between the mannan and xylan fractions of hemicelluloses. In softwood, the mannan content is between 10 to 18%, while in hardwood it is around 3 to 5% (Fengel and Wegener, 1989).

As is known, the use of wood materials is not limited to its natural state and can be expanded through various industrial applications that alter its structure. One of these applications is the modification processes applied to wood material. These processes cause differences in the structure of wood material, including its chemical properties (Yıldız 2002; Korkut and Kocaefe 2009; Aytin *et al.* 2022).

One of the methods applied in wood material modification applications is heat treatment (HT). Heat treatment in wood as an application dates back centuries. Since the 1990s, many European countries have developed numerous HT methods for both scientific and industrial purposes. Among the most important of these methods are ThermoWood® (Finland), PLATO® (Netherlands), OHT (Germany), and Rétification and Bois Perdue (France). However, ThermoWood® is more widespread. It has a higher production volume and is widely commercialized. Today, ThermoWood® is both a method of HT and the name of the modified wood material obtained after HT. Heat treatment is an environmentally friendly production technology, involving dimensional stability and resistance to fungal degradation in wood materials, and resulting in a homogeneous color change. It has been reported that HT causes permanent changes in the chemical composition of the wood's cell wall compound, imparting the wood material with a lower equilibrium, moisture content, and improved dimensional stability, while reducing moisture exchange and equilibrium moisture content. This process increases the wood material's resistance to decay (Aytin *et al.* 2016; Bi *et al.* 2022; Vidholdová *et al.* 2022).

Cryogenic treatment (Cr) is the controlled cooling of metallic materials by holding them at sub-zero temperatures for a specific period. This process aims to achieve the desired mechanical properties by changing the microstructure of the material. In the Cr, liquid nitrogen is introduced into a heat-insulated cryogenic container holding the materials to be processed. Cryogenic treatment can also be defined as "sub-zero processes" and depends on the cooling temperature. It is called "cold processing" between 0 °C and -80 °C, "shallow cryogenic processing" between -80 °C and -160 °C, and "deep cryogenic processing" between -160 °C and -196 °C. While dry ice is used as a coolant in cold processing, liquid nitrogen and liquid helium are used in shallow and deep cryogenic processing (Ates *et al*. 2017).

Cryogenic treatment has the potential to change the acoustic properties of wood. Cr reduces stresses within the wood's internal structure, minimizing residual stress between wood fibers and resulting in purer, more resonant sound. This allows a musical instrument

to achieve the best resonance quality suitable for its design. Cr aims to elevate the wood's sound transmission capacity and tone quality to the level of "aged" instruments. The relationship between the fundamental sound produced by the instrument and the overtones becomes more balanced, resulting in a richer and warmer sound character (Aytin *et al.* 2022; Anonymous 2026).

To best use the wood, it is necessary to understand the components that make up its structure and the methods of isolating these components. Heat treatment and similar modification applications affect the chemical structure of wood material, causing changes in both its elements and its organic and inorganic components. While Cr is primarily preferred for metallic materials, there are few studies that have fully concluded its effects on wood materials. In contrast, HT applications have been used on numerous wood species, and as mentioned above, there are many commercialized examples.

The thermal stability of wood depends on the ratio of chemical components in the wood species. As said by Fengel and Wegener (1989), softwood is thermally more stable than hardwood. This is because of hardwood's higher hemicellulose content compared to softwood. Additionally, the proportion of pentosans, which are more susceptible to thermal decomposition (xylose and arabinose), in hardwood is higher than that of hexosans (glucose, galactose, and mannose). Hardwood has more acetyl groups than softwood. The acetyl groups lead to the formation of acetic acid, causing acid-catalyzed decomposition of polysaccharides (Fengel and Wegener 1989).

According to a study by Gašparík *et al.* (2024), HT causes changes in cellulose crystallinity. This is especially seen in the sapwood of beech, with the most considerable changes among carbohydrates observed in xylose content. However, the increase in lignin content of red heartwood was found to be noticeably lower than that of sapwood.

Výbohová *et al.* (2018) subjected common ash wood to HT at different temperatures (160, 180, and 200 ºC) and times (3, 6, 9, and 12 h). Heat treatment caused the breakdown of hemicelluloses under all treatment conditions, resulting in a decrease in holocellulose content.

Studies that combine both HT and Cr and investigate their combined effects on wood materials are still in their early stages. Some studies in this field have found that certain increases occur in the compressive strength of wood, particularly parallel to the fibers (Aytin 2019; Aytin *et al.* 2016, 2022). However, studies that combine both HT and Cr and investigate their effects on the chemical properties of wood materials have not been found in the literature.

Rowan (*Sorbus torminalis* L.) is the tallest species of its genus. It is distributed throughout Türkiye except for the Southeastern Anatolia Region. This species is a forest tree that can reach 25 m in height and 75 cm in diameter. Due to the high value of its wood, it has suffered a great deal of destruction. It is one of the most important trees in industrial afforestation efforts (Gültekin 2006). This research aims to determine the changes in the chemical composition of rowan wood through the application of HT and Cr under different conditions. For this purpose, rowan wood samples treated with Thermowood® at two different temperatures and times (190 °C and 212 °C, 1 and 2 h) were then treated with Cr. A total of 8 (eight) variations were created together with control samples (Control). Samples were prepared according to the TAPPI T257 sp-14 (2014) standard method for chemical analyses, and the moisture, lignin, holocellulose, alpha-cellulose, and ash content, as well as the solubility in hot water, 1% NaOH, and acetone, were determined.

EXPERIMENTAL

Materials

Within the scope of the study, *Sorbus torminalis* trees, which grow naturally in Türkiye, were evaluated. Trees were taken from the stand according to the TS 4176 (1984) standard and then sawn into 60-mm-thick planks with the sharp cutting method according to TS 2470 (1976). After drying to an average of 12% moisture, the planks were made ready for HT with dimensions of 20 × 100 × 500 (mm × mm × mm) and subjected to HT with the ThermoWood®.

Heat-treated and cryogenically treated rowan wood samples were cut into 2 to 3 cm pieces and left to air dry at room temperature. After grinding the parts in a Retsch SM-100 laboratory mill, they were sieved through a vibrating sieve to the appropriate analytical size (40/60 mesh) according to the T 257 sp-14 (2014) standard method, placed in glass jars, and labeled. The moisture content of the samples was determined according to TAPPI T 412 om-11 (2011), and they were prepared for laboratory analysis. Chemical analyses were carried out on the sawdust fraction using two replicates per treatment.

Heat Treatment

Wood samples from working trees were subjected to HT along with air-dried wood materials to produce ThermoWood® products in the factory of Nova Forest Products Inc. in Gerede/Bolu. Rowan wood was treated with HT at two different temperatures (190 and 212 °C) and times (1 and 2 hours).

Shallow Cryogenic Treatment

Heat-treated rowan wood was then cut to dimensions of 20 × 20 × 300 mm (mm × mm × mm). Cryogenic treatment was applied to a portion of the control samples and some of the HT variations, creating 8 experimental variations (Table 1). Three of the eight experimental groups underwent shallow cryogenic treatment in a specially manufactured deep freezer.

Table 1. Variations Indicating the Type of Treatment and Conditions Applied to the Rowan Wood (*T* = temperature)

Variations	Sample Explanations	Heat Treatment		Cryogenic Treatment	
		T (°C)	Time (h)	*T* (°C)	Time (h)
R_1	Control	-	-	-	-
R_2	Control+Cr	-	-	-80	72
R_3	190 °C 1 hour	190	1	-	-
R_5	190 °C 2 hours	190	2	-	-
R_6	190 °C 2 hours +Cr	190	2	-80	72
R_7	212 °C 1 hour	212	1	-	-
R_8	212 °C 1 hour +Cr	212	1	-80	72
R_9	212 °C 2 hours	212	2	-	-

A Core DF 490 type deep freezer with 611 L capacity and 1 °C temperature sensitivity, without icing, was used in the shallow cryogenic process. An N-SmArt™ control system that stores temperature data numerically and graphically for ten years with 1 h recording intervals was used in the Core DF 490 type deep freezer. The device could

be cooled down to -86 °C. After all the samples were placed in separate compartments, a 12 h pre-waiting was performed for ambient temperatures to reach -80 °C before commencing Cr. At this stage, the samples were kept at -80 °C for 72 h (Aytin *et al.* 2022). Table 1 shows the treatments applied to the rowan wood, the treatment conditions, sample explanations, and variations.

Holocellulose and Alpha-Cellulose Determination

Holocellulose determination of wood samples was performed according to the acidified sodium chloride method developed by Wise and Karl (1962). In this method, sodium chlorite ($NaClO_2$) releases chlorine dioxide (ClO_2), an active oxidizing agent, under the influence of acetic acid. Thus, lignin is oxidized, converted into water-soluble derivatives, and passed into the solution. Carbohydrates, on the other hand, remain unchanged during this process and remain undissolved as holocellulose. Using this method, 5 g of moisture-free wood was weighed and placed in the Erlenmeyer flask, and 1.5 g of $NaClO_2$, dissolved in 160 mL of distilled water, was added. During the 5 h delignification process, 1.5 g of $NaClO_2$ was added to the samples every hour. Before adding $NaClO_2$, 10 drops of ice-cold acetic acid were added every hour to maintain the pH of the medium at around 4. The reaction medium's temperature was determined based on a study by Balaban (2002). The temperature applied was 75 °C. At the end of the delignification period, the samples were placed in an ice bath to rapidly cool and stop the reaction. Samples filtered through the crucible No. 2 were washed with distilled water, dried at 103 ± 2 °C, cooled in a desiccator, and weighed. Holocellulose percentages were calculated in relation to the oven-dried wood.

The determination of alpha-cellulose in wood samples was performed according to the TAPPI T 203 cm-22 (2022) standard. According to this method, a holocellulose sample weighing 2 g (oven dried) was placed in a 100 mL beaker. Ten mL of 17.5% NaOH was added and mixed thoroughly with a glass stirring rod. After 2 min, the mixture was pressed into the wood samples in the beaker using a stirring rod. After 3 min, 5 mL of 17.5% NaOH was added and mixed. After 5 min, another 5 mL of 17.5% NaOH was added and mixed. Finally, the beakers containing the samples were incubated in a water bath at 20 °C for 30 min. Thirty-three mL of distilled water was then added to the mixture, stirred, and left to stand in a water bath at 20 °C for 1 h. Afterwards, it was filtered under a vacuum using crucible No. 2. One hundred mL of 8.3% NaOH was added to the crucible without the suction for 1 min. The suction was then turned on and rinsed again with distilled water. Fifteen mL of acetic acid was poured into the crucible without the suction for 3 min. Then, the suction was turned on and the contents were rinsed again with 250 mL of distilled water. Finally, the crucible was dried at 103 ± 2 °C, cooled in a desiccator, and weighed. The α-cellulose content was calculated in relation to the oven-dried wood. Hemicelluloses were calculated by subtracting alpha-cellulose content from holocellulose content.

Klason Lignin Determination

Lignin determination in wood samples was performed according to the NREL procedure (Sluiter *et al.* 2011). According to this method, a specimen equivalent to 0.3 g of moisture-free wood was carefully placed in glass tubes. Then, 3 mL of 72% sulfuric acid was added to each tube and thoroughly mixed in a vortex to ensure homogeneous mixing. The glass tubes were placed in a water bath set at 30 ± 3 °C, left for 60 ± 5 min, and mixed every 10 min.

Afterwards, the tubes were removed from the water bath and transferred to a 10 mL Erlenmeyer flask. Then, using an automatic burette, 84 ± 0.04 mL of deionized water was added to dilute the acid to a 4% concentration. The tubes were sealed with aluminum foil and taped, and placed in an autoclave. The sealed samples were autoclaved at 121 °C, the temperature the liquids are typically adjusted to, for 1 h. After the autoclave cycle was complete, the hydrolysates were cooled slowly to near room temperature before removing the lids.

The autoclaved hydrolysis solution was filtered through pre-tared crucibles No. 3 and dried in an oven at 103 ± 2 °C until a constant weight was reached. It was cooled in a desiccator and weighed. The lignin percentage was calculated in relation to the oven-dried wood.

Acetone Solubility

The extractive content was determined by the sequential Soxhlet extraction method, using about 2 g of each sample and 150 mL of acetone solvent. Extraction was continued for 5 to 6 h, with a total of at least 24 siphonings from the first siphon. The extract content was determined according to the TAPPI T 204 cm-17 (2017) "Solvent Extracts of Wood and Pulp" standard, relative to oven-dried wood.

Hot Water and One Percent Sodium Hydroxide Solubility

Hot water solubility in the samples was determined according to the TAPPI T 207 cm-22 (2022) standard. According to this method, 2 g of moisture-free wood was weighed, placed in the Erlenmeyer flask, and 100 mL of hot water was added. It was then left in a reflux water bath for 3 h.

At the end of this period, the obtained solution was filtered in a pre-tared filter crucible No. 2, then dried in an oven at 103 ± 2 °C. The percentage of hot water solubility was calculated by weight loss.

The TAPPI T 212 om-22 (2022) standard was used to determine 1% NaOH solubility in the samples. The same procedures as the hot water solubility were carried out with a 1% NaOH solution over a period of 1 hr. The samples were filtered through glass crucible No. 2 and then dried in an oven at 103 ± 2 °C. The percentage of 1% NaOH solubility was calculated by weight loss.

Ash Determination

Inorganic compounds found in wood are determined as ash by burning the material at 575 ± 25 °C. Ash determination was performed according to the National Renewable Energy Laboratory (NREL), Laboratory Analytical Procedure (LAP) (Sluiter *et al.* 2005). According to this method, porcelain crucibles, preheated to 575 ± 25 °C in an ash furnace, were weighed to a constant weight. Then, 1 to 1.5 g of air-dried samples with known moisture contents were weighed into the crucibles. The samples were ashed using a muffle furnace equipped with a ramping program. After the ramping program and once a constant weight was found, the amount of ash in the sample was calculated by ratioing it by the initial weight of the oven-dried wood.

In determining the major components of the cell wall and analyzing the solubility, duplicates were conducted for each experiment.

RESULTS AND DISCUSSION

Figure 1 shows the color changes in rowan wood samples prepared for chemical analysis as a result of HT and after Cr was applied to some of the HT samples.

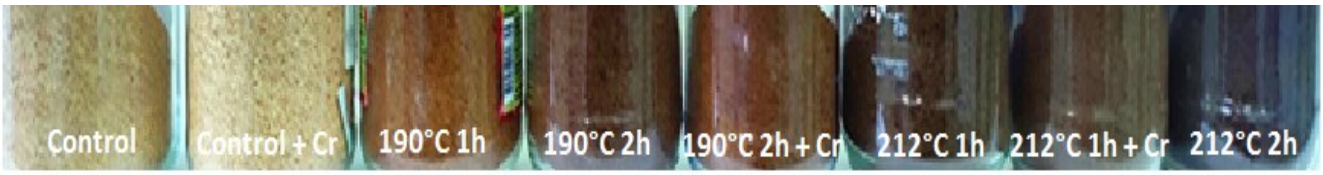

Fig. 1. The color changes in rowan wood samples

The most visible characteristic of HT was the darkening of the wood's color. As the treatment temperature and time increased, the color became noticeably darker compared to the control. However, a slight lightening of the color was observed in Cr applied HT samples. Cellulose and hemicelluloses in untreated wood cannot absorb visible light and do not cause discoloration. However, colored by-products formed during the degradation of hemicelluloses and chromophoric compounds in lignin can contribute to this change (Kocaefe *et al.* 2008). According to Ates *et al.* (2009), the heat-treated wood's darker color is due to an increase in the proportional content of lignin. Many studies of the heat treatment of tree species have observed similar color changes (Ates *et al.* 2009; Kučerová *et al.* 2016; Výbohová *et al.* 2018).

Chemical Composition of Rowan Wood

The results regarding the changes in holocellulose, alpha-cellulose, hemicelluloses, lignin, extractives, and ash content of heat- and cryogenically treated rowan wood samples are given in Table 2.

Table 2. Chemical Composition of Rowan Wood (%)

Variations	Holo-cellulose	α-cellulose	Hemi-celluloses	Lignin	Extractives	Ash
R_1	83.37 (0.09)	45.48 (0.08)	37.89 (0.09)	22.12 (0.13)	2.48 (0.00)	0.778 (0.03)
R_2	82.34 (0.01)	44.63 (0.04)	37.71 (0.03)	20.94 (0.03)	1.82 (0.00)	0.775 (0.00)
R_3	64.22 (0.10)	40.15 (0.24)	24.07 (0.17)	27.80 (0.21)	3.52 (0.17)	0.677 (0.01)
R_5	57.27 (0.14)	40.02 (0.16)	17.25 (0.15)	36.31 (0.31)	6.45 (0.05)	0.647 (0.04)
R_6	60.05 (0.03)	41.15 (0.02)	18.90 (0.03)	33.35 (0.35)	3.95 (0.05)	0.536 (0.06)
R_7	57.64 (0.02)	39.90 (0.01)	17.74 (0.51)	40.49 (0.51)	6.55 (0.25)	0.675 (0.03)
R_8	57.58 (0.16)	39.79 (0.09)	17.79 (0.13)	32.98 (0.67)	4.95 (0.00)	0.654 (0.00)
R_9	58.83 (0.17)	39.11 (1.34)	19.72 (1.25)	43.02 (0.33)	8.20 (0.10)	0.726 (0.05)
* The standard deviations are given in parentheses						

As shown in Table 2, untreated rowan wood (R_1) consists of alpha-cellulose (45.5%), hemicelluloses (37.9%), lignin (22.1%), extractives (2.5%), and ash (0.79%). The holocellulose content (sum of alpha-cellulose and hemicelluloses) on an oven-dried basis of rowan wood was 83.4%. Compared with the literature, the cellulose content was slightly lower, while the lignin and extractive content were higher, and the ash content was similar (Bahmani *et al.* 2020). Factors such as growing environment, growing season, growing location, soil conditions, analysis methods, etc. may have caused slight variances in the values (Amores *et al.* 2013).

Heat treatment of rowan wood causes important changes in its chemical composition, affecting components such as holocellulose, alpha-cellulose, hemicelluloses, lignin, extractives, and ash content. According to Table 2 and Fig. 2, holocellulose, alpha-cellulose, and hemicellulose values decreased in all HT variations compared to the control sample (R_1). The highest holocellulose content was found in R_1 at 83.4%, while the lowest value was detected in R_5 at 57.3%. The amount of decrease was 31.3% and 54.5% for holocellulose and hemicellulose, with the greatest decrease in the R_5 group. The results were in good agreement with the literature (Esteves *et al.* 2022; Gašparík *et al.* 2024). For example, Gašparík *et al.* (2024) stated that a decrease in hemicellulose content occurs during heat treatment. The main reasons are that hemicelluloses are the main component of wood with the lowest thermal stability, and that the acetic acids formed because of deacetylations break down hemicelluloses. Heat treatment reduces holocellulose content due to the degradation of cellulose and hemicellulose, which are more sensitive to heat than lignin (Mohareb *et al.* 2012; Carneiro *et al.* 2023). The structural heterogeneity of hemicelluloses makes it difficult to determine their thermal behavior. Pentosans (xylan) are the most heat-sensitive wood hemicellulose and are susceptible to degradation and dehydration reactions. The lower thermal stability of hemicelluloses compared to cellulose can be attributed, in part, to hemicelluloses' lower degree of polymerization, while cellulose is characterized by a crystalline structure (Yildiz 2002). Because hemicelluloses are highly hydrophilic polymers, a decrease in hemicellulose content reduces the overall proportion of free hydroxyl groups, which increases the water-binding capacity of the wood. This leads to an increase in the dimensional stability and decay resistance of heat-treated wood (Aytin *et al.* 2016; Aytin 2019). The holocellulose, alpha-cellulose, and hemicellulose changes in the rowan wood samples presented in Table 1 are shown in Fig. 2.

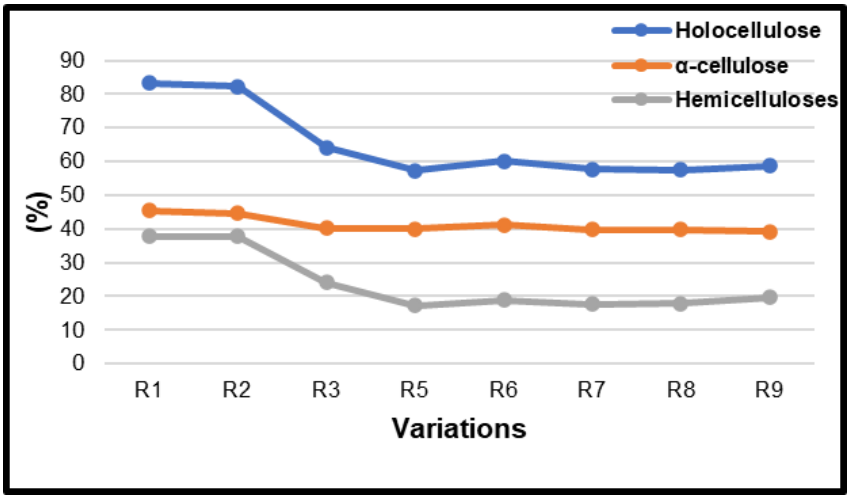

Fig. 2. Changes in holocellulose, alpha-cellulose, and hemicellulose content in rowan wood

The results showed that the amount of alpha-cellulose decreased with increasing HT temperature and time under all treatment conditions except R_6. The greatest decrease, compared to R_1 samples, was 14.0% in the R_9 group. In a study by Huang *et al.* (2010), Chinese white poplar was subjected to HT for 1 to 5 h at temperatures ranging from 170 °C to 230 °C. During HT, increasing temperature and time decreased holocellulose and alpha-cellulose content, while lignin content increased. Temperature had a greater effect than time on this change in the basic component amounts of the wood. On the other hand, in this study conducted to determine the effect of Cr in holocellulose, alpha-cellulose, and hemicellulose values, it was observed that the results were approximately the same in HT samples, although with slight differences.

The study results showed an effect on the change in lignin content in rowan wood treated with both HT and Cr. According to Table 2 and Fig. 3, the lignin content of the experimental groups increased meaningfully with increasing HT temperature and time compared to R_1.

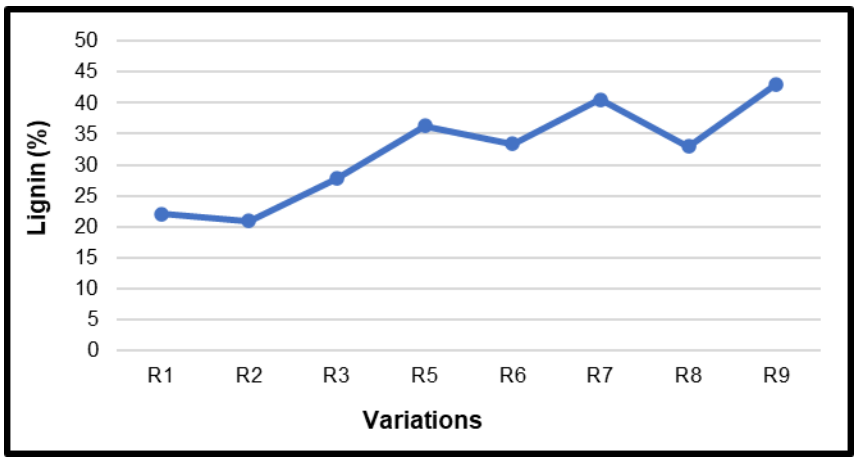

Fig. 3. Changes in lignin content in rowan wood

The highest and lowest increases in lignin content were determined in R_9 and R_3, at 94.5% and 25.7%. The results in this study were in good agreement with the observations of Esteves *et al.* (2022), due to similar temperatures and times of HT. The results revealed that when Cr was applied to the same heat-treated groups, lignin decreased compared to the HT samples. In Table 2, the percentage change in lignin content in Cr variations (R_6 and R_8) was 8.15% and 18.5%, compared to the HT variations (R_5 and R_7). The change in lignin content was similar between R_1 and R_2, with a 5.33% decrease observed in R_2 compared to R_1. The decrease in lignin content within the groups increased with increasing HT temperature and time, and was further enhanced by Cr. Kotilainen (2000) stated that the lignin mass ratio increased with both high temperature and long processing time, while the carbohydrate mass ratio decreased. On the other hand, in Klason lignin determination, some products resulting from the thermal decomposition of carbohydrates can remain in the lignin fraction. Lignin is affected by HT, but its degradation occurs more slowly compared to cellulose and hemicelluloses, leading to a percentage increase in lignin. Moreover, the degradation reactions occurring between lignin and its other products involve various condensation reactions, which also contribute to the percentage increase in lignin (Mohareb *et al.* 2012; Esteves *et al.* 2022; Carneiro *et al.* 2023). An increase in lignin content causes the wood to darken in color (Kučerová *et al.* 2016). There are evident

changes in the chemical composition of the wood because of HT; primarily, acetic acid forming due to the breakdown of amorphous hemicelluloses, and the increase of extractive and lignin content (Dumanlı and Windle 2012; Kučerová *et al.* 2016). The changes in lignin content of the rowan wood samples presented in Table 1 are shown in Fig. 3.

As shown in Table 2, the chemical component that increased the most proportionally with heat treatment was the extractives, rising from 2.48% to 8.20%. According to the results, the amount of extractives increased with increasing temperature and time of HT. However, Cr reduced this increase, and there were differences of approximately 24% to 40% between Cr and HT samples in the same groups. The results were in good agreement with the literature (Dumanlı and Windle 2012; Kučerová *et al.* 2016). The extractive content of wood, including resins, waxes, and other organic compounds, is also affected by HT. The extractive content typically increases in the initial stages of HT due to the evaporation of low molecular weight compounds (Esteves *et al.* 2022; Souza *et al.* 2024). Acetone solubility may increase due to the formation of new extractives and degradation products that are more soluble in organic solvents (Carneiro *et al.* 2023). Kučerová *et al.* (2016) and Výbohová *et al.* (2018) suggest that this increase in the proportion of extractives may be the reason for the darker wood color.

According to Table 2, the ash content in HT and Cr samples decreased compared to the control (R_1). The largest and smallest percentage decreases compared to R_1 were 16.8% and 6.68% in R_5 and R_9. When considering R_1, the ash content in the Cr variations (R_2, R_6, and R_8) was lower than in the R_1, R_5, and R_7 variations at 0.39%, 17.2%, and 3.21%. Cryogenic treatment is known to have a certain effect on the ash content in heat-treated samples. Ash content may increase because of the concentration of inorganic materials following the loss of organic matter during HT (Carneiro *et al.* 2023). Contrary to the literature, an increase in ash content was observed in samples R_7 and R_9, while a decrease occurred in the other samples.

Chemical Solubility of Rowan Wood

The results regarding the solubility of heat- and cryogenically treated rowan wood samples in hot water and 1% NaOH are given in Table 3.

Table 3. Chemical Solubility of Rowan Wood (%)

Variations	1% NaOH	Hot Water
R_1	29.14 (0.15)	3.06 (0.08)
R_2	23.84 (0.22)	3.79 (0.12)
R_3	30.99 (0.05)	7.07 (0.28)
R_5	34.92 (0.08)	9.29 (0.05)
R_6	34.42 (0.58)	7.29 (0.55)
R_7	36.47 (0.08)	7.49 (0.00)
R_8	36.65 (0.35)	8.95 (0.05)
R_9	37.59 (0.67)	7.35 (0.03)
* The standard deviations are given in parentheses		

According to Table 3, HT affected the solubility of both 1% NaOH and hot water in rowan wood, as both increased with increasing HT temperature and time. Compared to R_1, the solubility of 1% NaOH was the highest and lowest in variations R_9 and R_2 at 37.6% and 23.8%. Except for the change between Cr control samples (R_2) and R_1, it was understood that Cr had no significant effect on the solubility of 1% NaOH in the heat-treated groups. According to R_1, the highest and lowest percentage differences in hot water

solubility were 203.6% and 131.0% in the R_5 and R_3 variations. Contrary to the literature, the solubility of hot water and 1% NaOH increased with increasing HT temperature and time (Akyürek *et al.* 2021; Maulana *et al.* 2024).

Future Work

Future work is recommended to determine the causes of significant changes in the apparent amounts of chemicals in response to cryogenic treatments, especially when such treatments are the final treatment step. It is hypothesized here that the cryogenic treatments are not causing significant chemical changes. Rather, it is proposed that the cryogenic conditions are affecting the microstructure of the wood material. Such changes can be expected to affect one or more of the procedures used to determine chemical contents. Accordingly, the changes revealed by differences in compositional test results following cryogenic treatments have potential to be used as evidence related to wood's microstructure and related physical properties.

CONCLUSIONS

1. Heat treatment (HT) and cryogenic treatment (Cr) of rowan wood resulted in notable changes in holocellulose. Hemicelluloses were the most degraded wood cell component during these processes, and the amount of degradation increased with increasing HT temperature and time. The amount of alpha-cellulose also showed a similar change with heat treatment. Cr slightly increased the amount of hemicelluloses and alpha-cellulose in heat-treated samples.
2. Lignin content increased proportionally with increasing HT temperature and time, while only Cr limited this increase, based on the test results. When cryogenics were applied to heat-treated samples, the decrease in lignin content increased with increasing heat treatment temperature and duration, and was further enhanced by Cr.
3. The amount of extractives increases proportionally with increasing temperature and time of HT. However, Cr reduces this measured increase, and there are differences of approximately 24% to 40% between Cr and HT samples in the same groups.
4. Both HT and Cr reduced the determined ash content.
5. The solubility of hot water and 1% NaOH increased with increasing HT temperature and time.
6. While HT of rowan wood darkened the color in wood samples, depending on the increase in temperature and time, Cr slightly lightened the color.

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