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Variation of certain chemical properties within the stemwood of black locust (*Robinia pseudoacacia* L.)

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Abstract From the bottom, middle, and top of three mature 35 to 37-year old black locust tree discs were cut and analysed to determine the variation within the stem of certain chemical properties. Hot-water extractive content was greater in heartwood than in sapwood, while the reverse occurred for the dichloromethane extractive content. Vertical stem analysis of hot-water extractives showed that they increased in heartwood but decreased in sapwood from the bottom to the top of the stems while the reversal occurred for dichloromethane extractive content of sapwood. At the bottom and the middle of the stems, ash content was greater in sapwood than in heartwood, but at the top no difference was found between heartwood and sapwood. Ash content of both heartwood and sapwood was found to increase in the axial direction with respective values of 0.36% (bottom) and 0.76% (top) for heartwood and of 0.65% (bottom) and 0.76% (top) for sapwood. Ash analysis showed that considerable variations were found for the inorganic elements K and P being greater in sapwood than in heartwood. Heartwood was more acid than sapwood except for the top of the stems. Acidity mean values were found to increase from the bottom to the top of the stems in heartwood while they slightly decreased in sapwood. Total buffering capacity of heartwood was greater than that of sapwood and total buffering capacity of sapwood exhibited an inverse relationship to height. Very small acid equivalent values were determined only in sapwood. At the bottom, lignin content in heartwood (25.73%) was greater than in sapwood (18.13%). Lignin content of heartwood decreased from 25.73% at the bottom to 18.33% at the top, while that of sapwood was 18.13% at the bottom, 21.42% at the middle and 19.64% at the top.

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Variation verschiedener chemischer Eigenschaften im Stammholz von Robinie (Robinia pseudoacacia L.)

Zusammenfassung Vom unteren, mittleren und oberen Teil der Stämme von drei ausgewachsenen 35–37 Jahre alten Robinien wurden Stammscheiben herausgeschnitten und analysiert, um die Variation bestimmter chemischer Eigenschaften innerhalb des Stammes zu bestimmen. Insgesamt war der Heißwasser-Extraktstoffgehalt im Kernholz höher als im Splintholz, während für den Di-Chlormethan-Extraktstoffgehalt das Gegenteil der Fall war. Die senkrechte Stammanalyse der Heißwasser-Extraktstoffe ergab, dass der Extraktstoffgehalt im Kernholz vom unteren Stammende zum Zopf hin zunahm, aber im Splintholz abnahm, während der Di-Chlormethan-Extraktstoffgehalt im Splintholz zum Zopf hin zunahm. Die unteren und mittleren Stammteile wiesen im Splintholz einen höheren Aschegehalt auf als im Kernholz. Im oberen Teil unterschied sich der Aschegehalt zwischen Kern- und Splintholz nicht. Der Aschegehalt stieg sowohl im Kern- als auch im Splintholz in Stammlängsrichtung an, im Kernholz von 0,36% (unten) auf 0,76% (oben) und im Splintholz von 0,65% (unten) auf 0,76% (oben). Die Aschenanalyse ergab beträchtliche Schwankungen bei den anorganischen Elementen K und P. Im Splintholz waren diese höher als im Kernholz. Das Kernholz lag mit Ausnahme des oberen Stammbereichs mehr im sauren Bereich als Splintholz. Die durchschnittlichen Säurewerte nahmen im Kernholz in Stammlängsrichtung von unten nach oben zu und im Splintholz leicht ab. Die Gesamtpufferkapazität im Kernholz war größer als im Splintholz, wo sie mit zunehmender Stammhöhe abnahm. Der Ligningehalt war im unteren Stammbereich im Kernholz höher (25,73%) als im Splintholz (18,13%). Im Kernholz verringerte sich der Ligningehalt von 25,73% im unteren Stammbereich auf 18,33% im Zopfbereich, während der Ligningehalt im Splintholz im unteren Teil bei 18,13% lag, in der Mitte bei 21,42% und im Zopfbereich bei 19,64%.

1 Introduction

Black locust (*Robinia pseudoacacia* L.) is a fast-growing, very adaptive species that can grow satisfactorily on a wide variety of

sites, especially old fields of failed agricultural practice (Hanover 1992, Chow et al. 1996, Redei 1999, Arabatzis 2003). Due to its desirable wood quality and ecological characteristics, currently black locust is one of the widely planted broadleaved species in the world (Keresztesi 1988).

Black locust is an important tree species not only for energy production (Stringer and Carpenter 1986) but also for many technological uses such as sawnwood, glue-laminated structures, window frames, doors, parquets, furniture components and agricultural implements (Rendle 1972, Keresztesi 1981, Barrett et al. 1990, Stringer 1992, Molnar 1995). Lately, the increased concerns on environmental hazards of wood preservatives have drawn attention to naturally decay-resistant species, such as black locust.

Several investigations have been carried out to establish basic information on the chemical composition (Table 1) and extractive content and pH (Table 2) of both black locust mature and juvenile wood, which was proved to be quite variable (Koloc 1953, So et al. 1980, Stringer 1981, Lee 1983, Fengel and Wegener 1984, Ahn 1985, Stringer and Olson 1987, Kopitovic et al. 1989, Geyer and Walawender 1994, Molnar 1995, Chow et al. 1996).

In mature wood, between heartwood and sapwood of black locust, chemical differences were proved to exist (Tables 1 and 2). In sapwood, Hart (1968) obtained higher values of ash content, lower values of pH and 1% NaOH solubility and higher values of hot- and cold-water extractive content than in heartwood. Similar differences between sapwood and heartwood were also observed by Kopitovic et al. (1989) for ash content but total extractives in sapwood were found to be lower than in heartwood. The percentage of Klason lignin was found to be higher in heartwood than in sapwood (Kopitovic et al. 1989).

Differences of extractive and ash content between sapwood and heartwood were also reported for juvenile wood ((Stringer 1981, Stringer and Olson 1987), see Tables 1 and 2). A reference on variation of ash and extractive content between base and top of the stems was reported by Stringer and Olson (1987) only for young trees (see Tables 1 and 2). For the profitable utilisation of whole stems of black locust trees an in depth knowledge of the variation of chemical properties between sapwood and heartwood and along the stem axis is needed.

The objective of this study was to examine the within-stem variation of certain chemical properties of mature black locust wood. That would be useful for both better estimating timber quality and increasing the utilisation potentials of the species.

2 Materials and methods

From the bottom, middle and top of three mature 35 to 37-year old black locust trees, discs, 2 cm thick, were cut and were conditioned in the laboratory environment (Table 3). From each disc a radial stripe 2 cm in width was cut and it was divided into sapwood and heartwood parts. Each part was converted into small particles and then they were ground in a Willey mill through a No. 40 mesh sieve (0.425 mm). Same weight of air-dry ground wood material of all trees was thoroughly mixed for each sampling height (bottom, middle, top) and separately for heartwood and sapwood.

Table 1 Chemical composition of black locust wood
Tabelle 1 Chemische Zusammensetzung von Robinienholz

Literature	Organic constituents (%)			Lignin (%)	Ash (%)
	Holo-cellulose	Cellulose	Hemi-celluloses		
Mature wood					
Koloc (1953)		40.0	21.0	30.0	0.32
So et al. (1980)[1]	77.5		19.3	19.4	0.31
Lee (1983)	63.7			29.4	
Fengel and Wegener (1984)	81.7	50.1	23.7	20.6	0.30
Molnar (1995)	72.0–76.0			20.0–25.0	
Hart (1968)					
Sapwood					0.60
Heartwood					0.44
Kopitovic et al. (1989)[2]	66.4–67.9			24.1–24.5	0.22–0.24
Sapwood				21.4–22.2	0.28–0.30
Heartwood				24.4–25.0	0.22–0.23
Juvenile wood					
Ahn (1985)[3]	64.3–65.3			22.0–22.4	0.64–0.65
Geyer and Walawender (1994)[4]		50.9	20.9	17.7	0.37
Chow et al. (1996)[5]	75.3			20.0	
Stringer (1981)[6]					
Sapwood					0.62
Heartwood					0.28
Stringer and Olson (1987)[7]					
Base					0.56
Top					0.87

[1] 42-year old trees, [2] 26-year old trees, [3] 9- to 11-year old trees, [4] 7-year old trees, [5] 10-year old trees, [6] 2- to 10-year old trees, [7] 10- to 12 -year old trees

Table 2 Extractive content and pH of black locust wood
Tabelle 2 Extraktstoffgehalt und pH-Wert von Robinienholz

Literature	Extractives (%)					pH
	Hot water	Cold water	Alcohol-benzene	Total	1% NaOH	
Mature wood						
So et al. (1980)[1]	8.5	5.9	9.8		23.3	
Lee (1983)	5.2		5.1		21.5	
Fengel and Wegener (1984)	4.6		2.8			
Hart (1968)						
Sapwood	9.7	7.6			20.8	5.2
Heartwood	8.6	6.8			22.7	6.1
Kopitovic et al. (1989)[2]				7.7–7.8		
Sapwood				2.5–3.0		
Heartwood				8.6–8.8		
Juvenile wood						
Ahn (1985)[3]	5.5–5.9	3.4–3.8	7.1–7.3		23.1–23.5	
Chow et al. (1996)[4]	5.3		3.2		19.1	
Stringer (1981)[5]						
Sapwood	4.85		7.25			
Heartwood	3.82		7.46			
Stringer and Olson (1987)[6]	3.2					
Sapwood			2.7	6.8		
Heartwood		4.0–4.6	7.9–8.5			
Stringer and Olson (1987)[6]	2.8		3.5	7.3		
Base	2.69		3.7			
Top	3.50		2.7			

[1] 42-year old trees, [2] 26-year old trees, [3] 9- to 11-year old trees, [4] 10-year old trees, [5] 2- to 10-year old trees, [6] 10- to 12 -year old trees

Hot-water extractive content, dichloromethane extractive content and ash content were determined in accordance with ASTM D-1110, D-1108 and D-1102 standard methods, respectively. For the elemental composition of ash, K and Na were determined by flame photometer, P by a chromatometric method and the minerals Mg, Ca, Mn, Fe, Cu, Zn and Pb by atomic absorption.

Acidity (pH) and buffering capacity were measured by a method proposed by Sandermann and Rothkamm (1959). One gram of oven-dry material soaked in 20 ml of distilled water and stored at 20 °C for 24 hours. The pH was recorded by using a Chemtrix electric digital pH-meter after about 2 min when the pH-meter indicated a constant value. The same water solutions (20 ml each) were used for the determination of buffering capacity by titration to a pH of either 3 or 10 with nominal 0.05 N NaOH or H_2SO_4 solutions. Prior to each titration, the pH-meter was calibrated with standardised buffer solution to a range of pH 3–10. The pH was noted after about 3 min of each increment (ml) of base or acid. The acid buffering capacity (acid equivalent) is defined as the quantity (ml) of NaOH required to raise the initial pH of 1 ml of the sample solution to a pH of 10 and was determined as ml NaOH times the normality 0.05 N of NaOH solution. Comparable calculations were used for the determination of alkaline buffering capacity (alkaline equivalent), which utilise the acid solution (H_2SO_4) to lower the starting pH to pH 3. Total buffering capacity is merely the sum of these two values.

The Lignin content was measured on the hot-water extracted wood flour with the use of the diffuse reflectance infrared Fourier transform spectroscopy (DRIFTS) without any chemical pre-treatment of the samples (Pappas et al. 1998). The wood powder was yet ground in a ball mill (Retstch MM 2000) for 10 min and screened with a screen that had 0.090 mm diameter holes. The sieved material was placed in the freeze for 1 hour, liophilised (freeze-dried) in a Christ Alpha 1-2 apparatus for 24 hours and then kept in a desiccator containing P_2O_5. Infrared spectra were recorded, after mix of the samples with KBr powder in 1 : 100 ratios, with a Nicolet Magna 750 FT-IR spectrophotometer. A Spectra Tech microcup DRIFTS accessory was used, and the background spectra were collected by using pure dried KBr in powder form. Spectra were acquired and manipulated with the use of Omnic (ver. 3.1) FT-IR software at 4 cm^{-1} resolution and 64 scans per sample. Afterwards, the peak area

Table 3 Tree characteristics and sampling positions within the stem
Tabelle 3 Baumeigenschaften und Entnahmestelle der Stammscheiben

Tree no.	Age (years)	Height (m)	Sampling position (m)			Heartwood proportion[1] (%)		
			Bottom	Middle	Top	Bottom	Middle	Top
1	35	18	0.25	9	13.50	65.3	66.6	58.8
2	36	18	0.25	9	13.50	74.4	70.8	67.2
3	37	20	0.25	10	15.75	75.5	67.9	44.4

[1] % of cross-section area

at 1505 cm^{-1} (1534–1485 cm^{-1} region) was measured by using the corresponding function contained in the Omnic (ver. 3.1) FT-IR software. A linear relationship (coefficient of determination $R^2 = 0.98$) between the peak area E_{1505} at wave length 1505 cm^{-1} and lignin content was determined as follows:

$$E_{1505} = (0.36 \pm 0.15) + (0.17 \pm 0.01) \times \text{lignin}(\%) \quad (1)$$

3 Results and discussion

The results of the study are shown in Tables 4–5 and in Figs. 1–2.

3.1 Extractive content (hot-water, dichloromethane)

At all sampling heights (bottom, middle, top), hot-water extractive content was greater in heartwood than in sapwood, while the reverse occurred for the dichloromethane extractive content (Table 4). However, Hart (1968) has reported higher values of hot-water extractive content in sapwood than in heartwood. For heartwood, hot-water extractive content was the same at the bottom and middle (8.7%) but increased at the top (9.8%). A similar increase with height for hot-water extractive content was observed in juvenile black locust wood (Stringer and Olson 1987). The dichloromethane extractive content of heartwood remained more or less stable in the same direction and averaged 1.0% at the bottom and middle and 0.9% at the top. For sapwood, hot-water extractive content slightly decreased from 5.5% at the bottom to 5.0% at the top. The dichloromethane extractive content of sapwood increased with height from 1.1% at the bottom to 1.9% at the top.

The hot-water extractive content values calculated in this study (4.7%–9.8%) for the different stem parts were found to be similar with those (4.6%–9.7%) reported by other authors (Hart 1968, So et al. 1980, Lee 1983, Fengel and Wegener 1984, Kopitovic et al. 1989).

3.2 Ash content

Ash content (0.36%–0.79%, see Table 4) was found to be higher than most reported values varying from 0.22% to 0.32% (Koloc

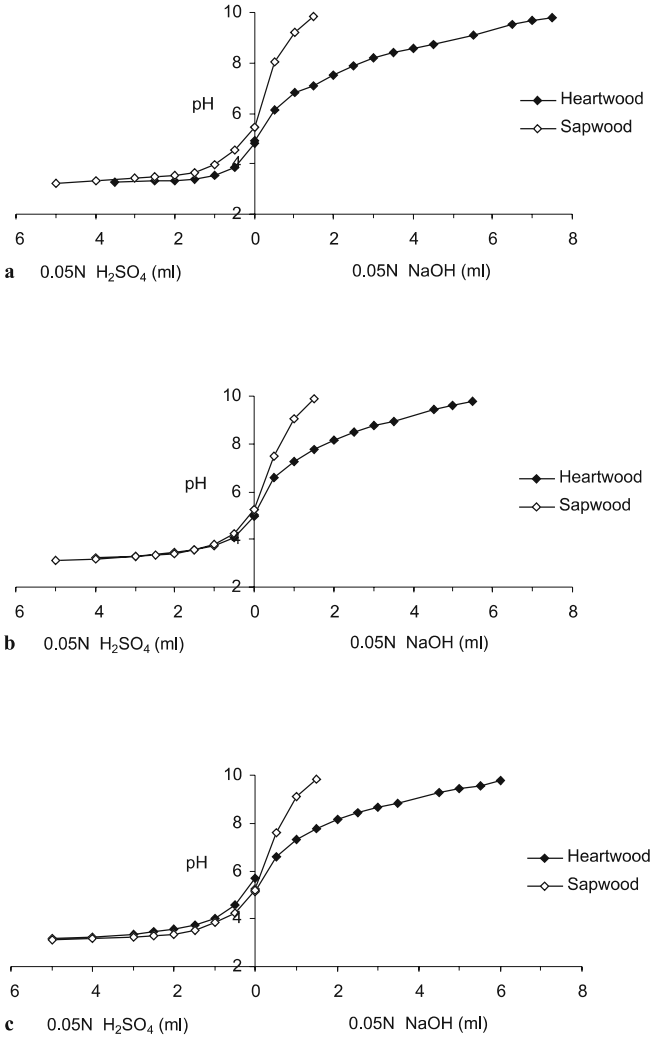

Fig. 1 Variation of acidity (pH) after each increment (ml) of 0.05 N NaOH or H_2SO_4 solutions at the bottom (**a**), middle (**b**) and top (**c**) of the stemwood of black locust

Abb. 1 Veränderung des pH-Wertes nach schrittweiser Zugabe von 0,05 N NaOH bzw. H_2SO_4 Lösungen im unteren (**a**), mittleren (**b**) und oberen (**c**) Stammbereich der Robinie

Table 4 Variation of chemical properties within the stemwood of black locust

Tabelle 4 Variation der chemischen Eigenschaften im Stammholz der Robinie

Property	Heartwood			Sapwood		
	Bottom	Middle	Top	Bottom	Middle	Top
Extractives[1] (%)						
Hot-water	8.7	8.7	9.8	5.5	4.7	5.0
Dichloromethane	1.0	1.0	0.9	1.1	1.2	1.9
Ash[1], (%)	0.36	0.50	0.76	0.65	0.79	0.76
Acidity (pH)[1]	4.85	5.01	5.42	5.44	5.27	5.23
Buffering capacity[1] (ml/ml)						
Acid equivalent	0.0188	0.0138	0.0150	0.0038	0.0038	0.0038
Alkaline equivalent	0.0088	0.0100	0.0100	0.0125	0.0100	0.0075
Total	0.0276	0.0238	0.0250	0.0163	0.0138	0.0113
Lingnin[2]	25.73	20.03	18.33	18.13	21.42	19.64

[1] mean values of two replicate measurements
[2] mean values of three replicate measurements

Table 5 Analysis of the ash of black locust wood
Tabelle 5 Aschegehalt-Analyse des Robinienholzes

Sampling position	Inorganic elements[1] (ppm)									
	Ca	K	Mg	Na	P	Mn	Fe	Cu	Zn	Pb
Bottom										
Heartwood	1,615	95	180	118	9	1	30	5	6	0.134
Sapwood	1,478	1,250	305	168	222	2	32	5	7	0.340
Middle										
Heartwood	1,830	383	163	187	21	2	23	5	5	0.268
Sapwood	2,027	1,067	187	190	206	1	21	5	5	0.550
Top										
Heartwood	2,013	650	158	113	35	2	21	4	4	0.088
Sapwood	1,845	617	183	182	198	2	27	5	6	0.257

[1] mean values of two replicate measurements

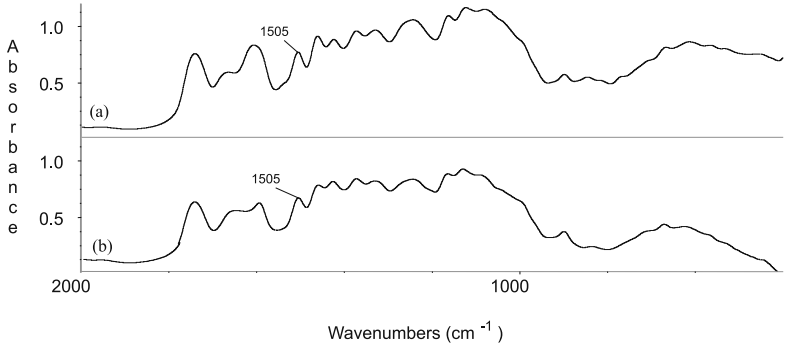

Fig. 2 FT-IR spectra obtained from the heartwood (**a**) and sapwood (**b**) samples at the bottom of black locust stem
Abb. 2 FT-IR Spektren von Kern- und Splintholzproben aus dem unteren Stammteil der Robinie

1953, So et al. 1980, Fengel and Wegener 1984, Kopitovic et al. 1989), while Hart (1968) has reported intermediate values (0.44%–0.60%).

According to Table 4, at the bottom and middle ash content was greater in sapwood than in heartwood and the same was concluded by Hart (1968) and Kopitovic et al. (1989). At the top no difference was found between heartwood and sapwood ash content (0.76%). Ash content increased with height (0.36% and 0.76% for heartwood and 0.65% and 0.76% for sapwood, at the bottom and the top, respectively). The same trend was also observed in juvenile black locust wood by Stringer and Olson (1987).

Qualitative analysis of the ash content showed that the differences in accumulation of the nutrients K and P were great between sapwood (higher values) and heartwood (lower values) except for K at the top. The content of the inorganic elements Mg, Na and Pb was consistently greater in sapwood than in heartwood for all stem heights. Inconsistent differences between heartwood and sapwood were also determined for Ca.

3.3 Acidity (pH) and buffering capacity

At the bottom and middle, heartwood was more acid than sapwood while the reversal occurred at the top (Table 4). Hart (1968) has found sapwood more acid than heartwood. Acidity of heartwood exhibited a positive vertical relationship (4.85 at the bottom, 5.01 at the middle and 5.42 at the top) while acidity of sapwood slightly decreased in the same direction.

At all sampling heights, total buffering capacity of heartwood was greater than of sapwood. According to Table 4, this was attributed mostly to the differences that exist in acid equivalent between heartwood and sapwood. The total buffering capacity of both sapwood and heartwood decreased from the bottom to the top. The least acid equivalent values were determined only in sapwood at all stem heights.

Change of acidity after each increment (ml) of 0.05 N acid or base solutions was similar at all sampling heights for both heartwood and sapwood (Fig. 1). In all cases the changes of acidity in the region from pH 3 to 10 was slow except the change for sapwood from the initial pH to pH 10 which was rapid.

The differences of the investigated chemical properties among different parts of black locust stemwood may cause difficulties in processing (e.g. procedures of gluing and pulping) of black locust wood and should be taken into account particularly in the manufacture of products where the adjustment of acidity in various levels is necessary. In manufacturing fiberboard and particleboard the acidity and buffering capacity of the furnish may affect the quality of polymerization and the curing rates of adhesives. Urea-formaldehyde resins, the adhesives typically used in bonding particleboard for interior uses and for wet-formed hardboard, are sensitive to pH, and cured under acidic conditions, thus the proper pH must be attained for the furnish, perhaps by the use of a catalyst (Chen and Paulitsch 1974, Johns and Niazi 1980, Maloney 1977, Slay et al. 1980). A number of investigations indicate the possibility of improving phenolic bonded

hardboard properties, by controlling the pH of wood, to produce dry-formed hardboard (Myers 1978, Nelson 1973). Furthermore, acidity in combination with the moisture content of the raw material can be a problem in grinding wood fibres. During digestion and refining, as the acid content increases, chemical acid hydrolysis of lignin and cellulose increase and, when combined with the already high temperature and pressure in the digester-refiner, the action on the fibres can be problematic (Maloney 1977). Acidity is also fundamental to the efficient use of wood in coating processes where the coating is pH sensitive, and low pH values might cause accelerated corrosion of metals and affect machinery and instruments in the manufacturing process. Discoloration of wood might also be influenced or even caused by pH values in many cases (Sandermann and Rothkamm 1959).

3.4 Lignin content

In Fig. 2, typical FT-IR spectra of heartwood and sapwood samples (triplicate) at the bottom are shown. The peak at 1505 cm^{-1} has been assigned to lignin (Pappas et al. 1998).

Lignin content values ranged between 18.13%–25.73% for all stem heights and for both sapwood and heartwood (Table 4). Most of the reported values (17.7%–25.0%) in the literature (So et al. 1980, Fengel and Wegener 1984, Ahn 1985, Kopitovic et al. 1989, Geyer and Walawender 1994, Molnar 1995, Chow et al. 1996) were found to be similar. Higher values of 30.0% and 29.4% were reported by Koloc (1953) and Lee (1983), respectively (see Table 1).

At the bottom, lignin content in heartwood (25.73%) was greater than in sapwood (18.13%) as Kopitovic et al. (1989) also stated. However, at the middle and top, lignin content of heartwood is slightly lower than of sapwood. Lignin content of heartwood decreased gradually from 25.73% at the bottom to 20.03% at the middle and to 18.33% at the top. Lignin content of sapwood exhibited a slight increase from 18.13% at the bottom to 19.64% at the top and it was found to possess its higher value at the middle (21.42%) (see Table 4).

4 Conclusions

The values of all chemical properties investigated in wood along the stems of black locust trees (bottom, middle, top) differ between sapwood and heartwood. Sapwood-heartwood differences are summarised as follows:

- Hot-water extractive content was higher in heartwood than sapwood at all stem heights. It ranged between 8.7%–9.8% for heartwood and between 4.7%–5.5% for sapwood. Dichloromethane extractives were found to be higher in sapwood (1.1%–1.9%) than heartwood (0.9%–1.0%).
- Ash content was higher in sapwood (0.65%–0.79%) than in heartwood (0.36%–0.76%). The content of the inorganic elements of K, Mg, Na, P and Pb was higher in sapwood than in heartwood.
- Heartwood was found more acid ($pH = 4.85 - 5.42$) than sapwood ($pH = 5.23 - 5.44$).
- Total buffering capacity was greater in heartwood (0.024–0.028ml/ml) than in sapwood (0.011–0.016 ml/ml).
- Lignin values were found also higher in heartwood (18.33–25.73%) than in sapwood (18.13%–21.42%).

Vertical stem analysis of samples obtained at bottom, middle, and top showed an increase with height for hot-water extractive content of heartwood, dichloromethane extractive content of sapwood, both heartwood and sapwood ash content, pH of heartwood and lignin content of sapwood, and a decrease with height for hot-water extractive content of sapwood, dichloromethane extractive content of heartwood, acidity of sapwood, total buffering capacity of both sapwood and heartwood and lignin content of heartwood.

The properties investigated in the present study are related with pulping characteristics, gluing behaviour and energy production, and, thus, they can be utilised for proper production of particleboards, fiberboards and glue-laminated timber products.

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