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INFLUENCE OF SOLVENT ON THE AMOUNT OF EXTRACTIVE CONTENT IN SAPWOOD, HEARTWOOD AND BARK OF ROBINIA PSEUDOACACIA

Pavel SABLÍK
Ing. et Ing. – Mendel University in Brno, Faculty of Forestry and Wood Technology, Department of Wood Science
Address: Zemědělská 3, 613 00 Brno, Czech Republic
E-mail: sablik.pavel@email.cz

Peter RADEMACHER
Dr. – Mendel University in Brno, Faculty of Forestry and Wood Technology, Department of Wood Science
Address: Zemědělská 3, 613 00 Brno, Czech Republic
E-mail: peter.rademacher@mendelu.cz

Abstract:
Total amount of extractive content of heartwood, sapwood and bark of Robinia pseudoacacia L. were determined. For extraction there were used fexIKA vario control extractor and several types of solvents (Cold Water, Hot water, Acetone, Benzene, Cyclohexane and Ethanol) with different results. Best results respectively mean highest amount of extracts, were identified with using ethanol, on the other side, lowest amounts were obtained with using cyclohexane as solvent.

Key words: *Robinia pseudoacacia L.; Extractives; fexIKA extractor; soluble substances.*

INTRODUCTION

Wood has an excellent mechanical performance, but wider utilization of this renewable resource as an engineering material is limited due to unfavourable properties such as low dimensional stability upon moisture changes and a low durability. However, there are some wood species, known for their ability to produce wood of higher quality by inserting mainly phenolic substances in the already formed cell walls. This process is called heartwood formation (Mahmut *et al*. 2012). At local market and in local environment in Czech Republic, there are several wood species which form heartwood. The tree species with almost best durability properties is wood of *Robinia pseudoacacia* L.

Traditional purpose of Robinia wood was to use it as fuelwood and for material use as tools and roundwood timber. Current utilization of this material is mostly oriented towards high value use (Molnar 1995).

Existence of biocides in these wood species and possibility of utilization of these substances are new fields of material or chemical use (Wu *et al*. 2012). Bioactive substances of nature origin are promising components to increase durability of wood species with lower natural durability (Ermeydan MA *et al*. 2012, Sa *et al*. 2009).

Extractives of most wood species range approximately from 2 to 10% of dry matter, in some cases (mostly in tropical species) this content can increase up to 20% or more of dry matter (Yang *et al*. 2003). In literature there are various results of amount of extractives possible to gained out of Robinia wood, short overview is shown in Table 1.

A lot of investigations have been carried out on these wood species dealing with extractive content (Schultz and Nicholas 2000, Hillis 1987, Syafii *et al*. 1987, Bamber and Fukazawa 1985, Scheffer and Cowling 1966). For investigation of interesting substances, it is necessary to extract native or residual material and analyse the chemical composition of extracts. Result of extraction could be affected by several causes: from equipment and methodology used for extraction, through origin of material and its quality (manufactory), to type of used solvent.

The objective of this paper is to compare results from different solvents used for extraction of *Robinia pseudoacacia* L. sapwood, heartwood and bark.

MATERIAL AND METHODS

Material and Sample preparation

For this investigation, wood from 70 years old trunk of *Robinia pseudoacacia* L. was used. Tree grown on North slope approximately 25km from Brno, Czech Republic. All groups of specimens (sapwood, heartwood, bark) were made from chosen parts across whole trunk, with aim to gain average values. From trunk, five 0.6 meters long logs at heights of 0m (base of tree), 1.3m, 4m, 6m and 9m were cut and centre boards were prepared. For the first overview about amount of extractive content, samples from different highest and diameter position were mixed, only separation bark, heartwood and sapwood. Bottom and top parts of each log were cut and conditioned at room temperature for one month. These parts were used for specimen preparation. After conditioning, material was ground with a cutting mill to pass a 0.5mm screen.

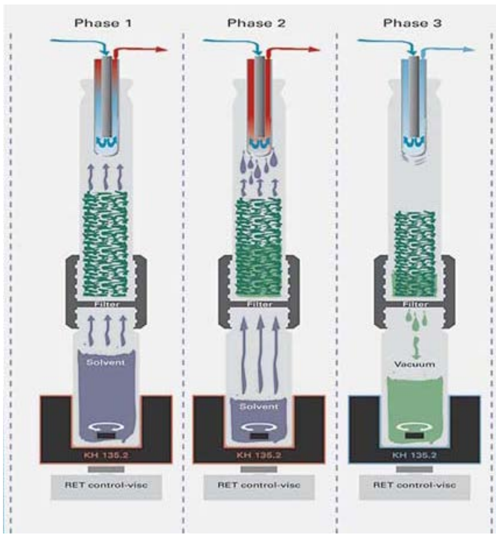

Fig. 1
Extraction phases (foto labicom.cz).

Extraction cycle of the fexIKA extraction method can be divided into three phases. Detailed description of the functional principle is given in the material and methods section.

Wood extraction and evaporation

Prepared specimens were extracted using fexIKA vario control soxhlet apparatus. This equipment accelerates extraction process, compared with classic Soxhlet extraction system.

Phase 1

Solvent is heated up to the boiling point and evaporates. The vapour of solvent penetrates through the filter and the extracted material. At the beginning it condenses within the extraction material.

Phase 2

The solvent vapour condenses at the cooling element at top of the extraction tube and drops on the sample. The condensed solvent in the extraction tube is heated and the sample is extracted intensively in the solvent. This technique ensures effective extraction kinetics for the whole extraction period.

The condensed solvent is collected in the extraction tube, while the solvent in the basic vessel is permanently evaporated.

Phase 3

Heating is switched off after all amount of solvent in the basic vessel had evaporated and condensed into the extraction tube. By switching the solenoid valve cooling of the heating block is started. The induced cooling and the condensing of the solvent create a vacuum in the basic vessel. The resulting differential pressure in relation to the atmospheric pressure conveys the solved extracts through the filter into the basic vessel.

Prepared wood powder was extracted by several different solvents (Cold Water, Hot water, Acetone, Benzene, Cyclohexane and Ethanol).

For evaporating of solvent from extract roto-evaporator was used. Heating temperature for evaporating was set based on solvent type, rotation of flask was set on 15RPM and vacuum on 700mBar. After evaporation, specimens were inserted to the box drier with temperature set on 80°C for 30 minutes and then conditioned in desiccator for 30 minutes. After this process, extracted residue was weight and percentage of extract amount of extract in original specimen was calculated.

Table 1

Example of different values of total amount of extracts obtained from Robinia pseudoacacia L. by using hot water as solvent, values from: Singh T and Singh AP (2012), Costas (2008), Kartal et al. (2006)

Spieces	Location	Solvent	Extract yield [%]
Robinia pseudoacacia L.	Bark	Hot water	13,5
	Bark	Hot water	12,3
	Heartwood	Hot water	10,1
	Bark	Hot water	9,8
	Heartwood	Hot water	9,5
	Bark	Hot water	9,3
	Heartwood	Hot water	8,7
	Heartwood	Hot water	7,5
	Heartwood	Hot water	6,9
	Heartwood	Hot water	5,2
	Sapwood	Hot water	4,9
	Sapwood	Hot water	4,2
	Sapwood	Hot water	3,3

RESULTS AND DISCUSSION

As was mentioned before, to identify and confirm role of extractives in natural durability of wood and their future utilization to improve natural durability, it is necessary to develop the optimal process. This needs to find out how to extract reactive solutions out of the wood without damaging or inactivating (for example by using high temperatures).

As seen in Table 2 and Table 3, total amount of extractives is strongly dependent on used solvent and type of used material (sapwood, heartwood, bark). The values differed between 1% (sapwood, cyclohexane) and 8% (heartwood, ethanol).

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Results also show increasing values of total extractive content for heartwood with using different solvents: 1,0% with Cold water, 1,4% Cyclohexane, 4,4% Acetone, 5,0% Benzene, 7,3% Hot Water, 8,1% Ethanol. Similar results are achieved for sapwood and bark of *Robinia pseudoacacia* L.

Table 2

Total amount of extractives in different types of material with using different types of solvents. Data shows amount of extract (in %) gain in 1st, 2nd, 3th and 4th cycle – separately. (HW – heartwood, SW – sapwood)

Solvent	Volume of Solvent [ml]	Speciment Wood Spieces		1st Cycle Boiling Temp. Set [°C]	Boiling Time [min (hours)]	Final Cooling Temp. [°C]	Cooling Time [min]	Extractive Content [%]	2nd Cycle Extractive Content [%]	3rd Cycle Extractive Content [%]	4th Cycle Extractive Content [%]
Acetone	100	*Robinia pseudoacacia L.*	bark	75	32	40	1	5.25	0.59	0.40	0.23
Acetone	100	*Robinia pseudoacacia L.*	HW	75	32	40	1	3.08	0.20	0.15	0.10
Benzen	100	*Robinia pseudoacacia L.*	bark	92	25	40	1	2.25	2.15	0.85	0.61
Benzen	100	*Robinia pseudoacacia L.*	HW	92	25	40	1	2.54	2.34	0.10	0.10
Cyklohexan	100	*Robinia pseudoacacia L.*	bark	95	15	40	1	see Table 3			
Cyklohexan	100	*Robinia pseudoacacia L.*	SW	95	15	40	1	see Table 3			
Cyklohexan	100	*Robinia pseudoacacia L.*	HW	95	15	40	1	0.60	0.70	-	-
Ethanol	100	*Robinia pseudoacacia L.*	HW	95	25	40	1	5.32	5.39	2.50	0.08
Ethanol	100	*Robinia pseudoacacia L.*	SW	95	25	40	1	2.21	1.79	0.30	-
Hot Water	100	*Robinia pseudoacacia L.*	HW	130	35	40	1	3.79	1.50	0.36	0.14
Hot Water	100	*Robinia pseudoacacia L.*	bark	130	35	40	1	3.17	2.31	0.86	0.63
Cold Water	100	*Robinia pseudoacacia L.*	HW	20	24h	-	-	1.00	-	-	-
Cold Water	100	*Robinia pseudoacacia L.*	HW	30	24h	-	-	1.10	-	-	-
Cold Water	100	*Robinia pseudoacacia L.*	SW	30	24h	-	-	2.20	-	-	-

Table 3

Total amount of extractives in different types of material with using different types of solvents. Data shows amount of extracts gain from n cycles – together. (HW – heartwood, SW – sapwood)

Solvent	Volume of Solvent [ml]	**Speciment** Wood Spieces Source		**n-Cycles** Number of Cycles	Boiling Temp. Set [°C]	Boiling Time [min (hour)]	Final Cooling Temp. [°C]	Cooling Time [min]	Extractive Content [%]
Acetone	100	*Robinia pseudoacacia L.*	bark	4	75	32	55/40	1	7.11
Acetone	100	*Robinia pseudoacacia L.*	HW	3	75	32	55/40	1	4.40
Benzen	100	*Robinia pseudoacacia L.*	bark	4	92	22	55/40	1	6.22
Benzen	100	*Robinia pseudoacacia L.*	HW	3	92	22	55/40	1	5.04
Cyklohexan	100	*Robinia pseudoacacia L.*	bark	4	95	10	55/40	1	4.54
Cyklohexan	100	*Robinia pseudoacacia L.*	SW	4	95	10	55/40	1	1.30
Cyklohexan	100	*Robinia pseudoacacia L.*	HW	4	95	10	55/40	1	1.35
Ethanol	100	*Robinia pseudoacacia L.*	HW	3	95	22	55/40	1	8.07
Ethanol	100	*Robinia pseudoacacia L.*	SW	3	95	22	55/40	1	3.56
Hot Water	100	*Robinia pseudoacacia L.*	HW	3	130	30	55/40	1	7.25
Hot Water	100	*Robinia pseudoacacia L.*	bark	4	130	30	55/40	1	6.95
Cold Water	100	*Robinia pseudoacacia L.*	HW	1	20	24h	-	-	1,00
Cold Water	100	*Robinia pseudoacacia L.*	HW	1	30	24h	-	-	1.10
Cold Water	100	*Robinia pseudoacacia L.*	SW	1	30	24h	-	-	2.20

CONCLUSION

This work specified the differences in amount of extractive content, possible to gain out of *Robinia pseudoacacia* L. heartwood, sapwood and bark by using different types of solvents. Acetone, Benzene and Cyclohexan soluble extractives of black locust were found to be higher in bark than in other wood components, while Ethanol and Hot water shows higher results in heartwood.

These results represent first investigations of extractive content of *Robinia pseudoacacia* L. and their dependence on various conditions (position in trunk, locality, other types of solvents and their mixtures).

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