SITE, SPACING, TREE PORTION, AND SPECIES INFLUENCE ASH AND EXTRACTIVES CONTENT OF FIVE JUVENILE HARDWOODS[1/]

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Abstract.--Three year old short-rotation trees of Elaeagnus umbellata, Alnus glutinosa, Robinia pseudoacacia, Populus deltoides, and Platanus occidentalis were established on Illinois' marginal land that was not suitable for food production. Test results indicated that all independent variables (site, spacing, wood portion, and species) of the silvicultural practices caused significant influences on the dependent variables (Ash or mineral, alcohol-benzene extractives, one-percent sodium hydroxide extractives, and hot-water extractives content). Populus deltoides was found to have the highest ash and extractives content among the five juvenile hardwood species. Test results indicated that the five juvenile hardwoods examined could serve as a raw material for the chemical industries.

Keywords: short-rotation, biomass, juvenile hardwoods, black locust, black adler, sycamore, autumn olive, and cottonwood chemicals, ash, extractives, site, spacing.

INTRODUCTION

The chemical components of wood profoundly affect the utilization of wood varying widely with wood species, the portion of the tree, and the age of the tree, because wood is not a uniform substance. Thus the species with high cellulose content and low lignin content are usually preferred for pulp and paper making. The species rich in extractive materials could have a higher potential for production of certain chemicals such as tannin, rosin and rubber.

Ash is the residue that remains after complete combustion of the wood material at 575 ± 25°C. It is composed primarily of inorganic substances such as calcium oxide (CaO) and silica (SiO_2), and small percentages of alumina (Al_2O_3), iron oxide (Fe_2O_3), and magnesium oxide (MgO) (Karchesy 1979).

Generally, ash is considered an undesirable material in most industrial situations. Though the ash content is a small percentage of the total wood substance, the juvenile woods may have ash contents several times that of mature woods, and may therefore require extra attention by maintenance when used as a fuel or as a raw material for the pulp and paper industry. Ash will accumulate in furnaces and can interfere with the combustion process. This will reduce combustion efficiency, and increase hydrocarbon emissions (Tillman et al. 1981). The increase in use of wood as a fuel may cause an ash disposal problem. According to Tillman (1978), this problem may be eliminated since ash is useful as a soil conditioner and fertilizer when supplemented with nitrogen.

Wood extractives are extraneous components, not essential to the wood cell wall. They are found as surface deposits on cell lumina, or entering completely into the cell walls. The wood extractives include a wide variety of classes of organic compounds, but can be divided into two major groups, namely the resins and phenols. The resins, or oil fraction, consist mainly of free resin acids, fatty acids and fatty

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acid esters, terpenes, and waxes. The phenols consist of a wide range of substances including the tannins and lignins (Wenzl 1970). Other important extractives include some sugars, volatile hydrocarbons, and proteins. Though the extractives are generally a small percentage of the total wood volume, they have a large influence on many wood properties. Extractives affect the odors, colors, and fungal and insect resistance of wood, and are directly related to permeability, specific gravity, and hardness of wood.

Extractives are removed from the wood by different kinds of chemical solvent on the basis of polarity. Therefore, each organic solvent will remove a select portion of the total extractives. In the recovery of waste liquors from pulping process, chemicals such as acetic acid, formic acid, tall oil, and methanol are produced depending on the pulping process. Tall oil is used in the manufacture of rubber and plastics, soaps and detergents, printing inks, metal working compounds, vinyl plasticizers, fabric softeners, paper size, epoxy coatings, paints and varnishes, asphalt emulsifiers, adhesives, drilling muds, and textile processing chemicals. Tall oil and fatty acids are also used in the flotation of various metals reported by the Westvaco Paper Company, 1986.

Logging slashes could be used for extraction of polyphenols, glycosides, and carbohydrates reported by Murkos (1970). The study of the chemical composition of wood and bark of alder trees indicated that both the wood and bark could be valuable sources for tannins. Usually, the bark is rich in extractives and mineral substances. Therefore, it appears that with a large portion of bark, trees have greater potential than wood alone for chemical conversion. Chow and co-workers (1980) found that heat value of one-year-old sycamore wood is positively related to its extractives content.

The primary objective of this study was to determine the ash and extractives content of five species of three-year-old plantations of short-rotation hardwoods. The objective of this study was two-fold: (1) to determine the selected chemical properties of five species of juvenile hardwood and (2) to determination the effects of species, wood material portion, tree spacing, and planting site on selected chemical properties of five plantation-grown juvenile hardwoods.

MATERIALS

Bundles of whole tree specimens were received from the plantation sites at the Dixon Springs Agricultural Center, southern Illinois. The trees were harvested and bundled according to species, and the site and spacing variables of the growth plots. All trees were 3 years old. All 5 species were grown on both bottomland and upland sites using both narrow and wide spacings. The wide spacing indicated in this study meant 12 x 18 inches, and the narrow meant 9 x 9 inches.

Weights of each bundle were recorded. Branchwood was then removed from each main stem and the branchwood and stemwood portions were each weighed for determination of the branch to stemwood ratio.

To prepare the wood portion, bark was removed from sections of the base, center, and top of randomly chosen stems of each bundle. These bark free stem sections were then cut into small cross-sectional disks, and ground in a Wiley mill to pass a 60 mesh screen. For the mix portion, sections of stemwood from the base, center, and top were cut and ground to pass a 60 mesh screen. Randomly chosen branchwood was then ground to particles and mixed with the stemwood particles in the original stem-to-branch ratio.

EXPERIMENTAL DESIGN

A Randomized Complete Block (R.C.B.) design was used in the factorial analysis to determine the effects of the silvicultural treatment combinations on each dependent variable (ash content, alcohol-benzene extractives, hot water extractives, and 1 percent sodium hydroxide extractives content). The treatments or independent variables were site (factor A), spacing (factor B), wood portion (factor C), and species (factor D).

Site:
- A1 = bottomland
- A2 = upland

Plantation spacing:
- B1 = narrow (9 x 9 inch)
- B2 = wide (12 x 18 inch)

Tree portion:
- C1 = mixture of bark, branches, and stemwood
- C2 = wood

Species:
- D1 = autumn olive (*Elaeagnus umbellata* Thumb.)
- D2 = black alder (*Alnus glutinosa* L.)
- D3 = black locust (*Robinia pseudoacacia* L.)
- D4 = eastern cottonwood (*Populus deltoides* Bartr.)
- D5 = sycamore (*Platanus occidentalis* L.)

Thus the overall analysis of the R.C.B. was a 2 x 2 x 2 x 5 factorial, for each dependent variable.

The following linear model for the R.C.B. was used for explaining sources of variation:

$$Y_{ijklm} = \mu + R_i + A_j + B_k + AB_{jk} + C_l + AC_{jl} + BC_{kl} + ABC_{jkl} + D_m + AD_{jm} + BD_{km} + CD_{lm} + ABD_{jkm} + ACD_{jlm} + BCD_{klm} + ABCD_{jklm} + E_{ijklm}$$

where,

Y_{ijklm} = any observed value for a chemical property
μ = overall mean
R_i = block effect (replications)
A_j = site effect
B_k = spacing effect
C_l = wood portion effect
D_m = species effect
E_{ijklm} = experimental error
i = 1-6 replicates
j = 1-2 levels of A
k = 1-2 levels of B
l = 1-2 levels of C
m = 1-5 levels of D

All combinations of letters in the model represent interaction effects of the independent variables.

All statistical analyses were carried out using computer software procedures described in the Statistical Analysis System (SAS 1981) for Linear Models (Murko 1970). Procedures used included the General Linear Models for the factorial analysis, Duncan's multiple range test for multiple comparisons among means, and the least significant difference (LSD) procedure for the t-test multiple comparisons between means.

TEST METHODS

All experimental procedures were carried out according to the American Society for Testing and Materials (ASTM D 1102, D 1107, D 1109 and D 1110, 1982) and the Technical Association of the Pulp and Paper Industry (TAPPI). Six tests were run for each combination of independent variables represented in the study. Extractive contents were determined using three different solvents. They were: hot-water, alcohol-benzene (1 part 95% ethanol, 2 parts distilled benzene), and one percent sodium hydroxide.

RESULTS AND DISCUSSION

Table 1 shows moisture contents and specific gravity of sample material.

Table 1. Specific Gravity and Moisture Contents of Sample Material

		Specific Gravity		Moisture Content2/	
Species		Stem Wood	Stem Wood with Bark	Stem Wood	Stem Wood with Bark
Autumn olive	NB1/	.72	.62	7.3	7.6
	NU	.69	.70	7.1	7.4
	WB	.67	.71	8.2	9.3
	WU	.72	.78	10.6	15.0
Black alder	NB	.47	.52	9.2	10.1
	NU	.46	.52	7.4	7.8
	WB	.44	.55	7.9	8.6
	WU	.43	.48	7.0	8.5
Black locust	NB	.52	.62	12.8	12.6
	NU	.61	.69	9.5	8.4
	WB	.54	.53	10.8	10.4
	WU	.46	.55	10.2	9.5
Eastern cottonwood	NB	.39	.42	8.0	8.2
	NU	.43	.43	7.0	7.0
	WB	.39	.48	8.6	8.1
	WU	.38	.43	10.1	9.1
Sycamore	NB	.70	.66	7.9	8.0
	NU	.55	.52	9.5	9.3
	WB	.50	.52	10.2	11.3
	WU	.52	.51	8.0	9.5

1/ N = narrow, W = wide, B = bottomland, U = upland.
2/ Prior to determining moisture content, trees were air dried at 72° F. indoors for three weeks.

Table 2. Ash and extractives content of five hardwood species

Species	Portion	Site	Spacing	Ash %	Extractives: Alcohol Benzene %	Extractives: Hot-water %	Extractives: 1% NaOH %
Autumn olive	Mixture	B	N	1.20 (2.75)1/	3.49 (10.26)	8.78 (7.06)	30.85 (1.42)
			W	1.35 (1.62)	2.54 (8.55)	10.05 (3.06)	34.40 (1.40)
		U	N	1.70 (1.05)	2.12 (43.15)	9.84 (10.69)	34.61 (0.92)
			W	1.45 (2.97)	2.55 (6.26)	10.38 (3.16)	33.88 (1.49)
	Wood	B	N	0.99 (2.10)	0.57 (40.30)	6.80 (2.13)	24.65 (1.73)
			W	1.04 (4.15)	2.57 (18.14)	8.04 (7.00)	25.62 (1.11)
		U	N	1.17 (2.05)	0.30 (62.99)	7.39 (6.66)	25.28 (0.84)
			W	1.04 (1.26)	1.84 (19.37)	9.51 (3.27)	28.56 (1.06)
Black alder	Mixture	B	N	2.25 (2.94)	7.32 (4.17)	13.06 (1.13)	42.40 (1.15)
			W	1.88 (1.49)	6.51 (30.35)	14.64 (0.98)	44.07 (1.04)
		U	N	1.53 (1.50)	4.35 (13.07)	12.12 (1.59)	36.30 (2.51)
			W	1.64 (1.29)	11.65 (9.20)	12.73 (1.59)	41.71 (1.37)
	Wood	B	N	1.00 (3.23)	4.84 (10.50)	9.17 (1.12)	31.81 (3.03)
			W	1.09 (1.32)	8.83 (3.86)	12.14 (1.17)	34.27 (0.95)
		U	N	0.71 (1.69)	2.64 (17.22)	8.90 (0.72)	29.65 (4.31)
			W	0.94 (2.74)	2.41 (22.90)	7.59 (9.56)	30.53 (0.90)
Black locust	Mixture	B	N	2.27 (3.80)	5.85 (6.46)	10.54 (3.65)	35.59 (1.46)
			W	2.01 (1.81)	3.00 (21.81)	12.00 (3.16)	36.10 (0.90)
		U	N	2.27 (0.95)	5.82 (8.16)	11.00 (3.41)	36.92 (1.62)
			W	3.11 (1.47)	4.19 (17.83)	14.50 (1.30)	37.72 (0.95)
	Wood	B	N	0.75 (8.09)	2.57 (8.88)	7.54 (4.00)	27.89 (0.62)
			W	0.72 (0.72)	5.30 (18.46)	8.42 (4.85)	25.72 (2.15)
		U	N	1.02 (4.07)	2.62 (6.59)	10.35 (1.64)	26.80 (1.83)
			W	0.91 (1.99)	4.01 (33.46)	9.45 (2.29)	28.50 (0.93)
Eastern cotton-wood	Mixture	B	N	2.93 (1.79)	12.64 (5.41)	19.11 (1.48)	46.93 (0.83)
			W	2.16 (0.85)	13.30 (5.49)	15.22 (2.75)	40.06 (2.22)
		U	N	3.33 (0.92)	8.14 (8.79)	16.76 (1.18)	42.83 (1.23)
			W	3.44 (0.94)	8.98 (12.35)	19.30 (0.58)	44.32 (1.07)
	Wood	B	N	1.03 (8.75)	4.04 (12.86)	9.08 (1.56)	34.12 (0.44)
			W	0.63 (4.37)	8.99 (9.64)	6.84 (4.06)	28.13 (0.97)
		U	N	1.47 (1.12)	3.42 (11.73)	6.54 (9.23)	29.69 (1.59)
			W	1.81 (1.52)	4.93 (12.59)	7.91 (3.54)	30.72 (0.76)
Sycamore	Mixture	B	N	1.32 (1.76)	5.24 (6.77)	8.85 (3.07)	34.49 (0.54)
			W	1.38 (0.74)	3.07 (33.17)	8.77 (4.26)	33.98 (1.94)
		U	N	1.52 (0.81)	3.05 (8.39)	7.06 (3.39)	29.80 (1.05)
			W	1.50 (1.36)	2.78 (7.83)	9.23 (12.42)	31.04 (1.10)
	Wood	B	N	0.83 (1.83)	3.50 (6.18)	9.02 (1.93)	30.50 (0.68)
			W	1.02 (1.55)	3.25 (8.57)	6.70 (8.83)	30.08 (1.04)
		U	N	1.52 (1.28)	3.18 (37.08)	6.96 (3.47)	27.81 (1.64)
			W	1.23 (0.89)	2.13 (31.36)	7.78 (9.12)	27.06 (2.33)

1/ Values in parenthesis are coefficient of variation.

Ash content:

Ash contents of mature North American hardwoods have been reported to range from about 0.2 to 0.9 percent, rarely exceeding one percent (Brown et al. 1952). Ash contents for all combinations of independent variables in this study are shown in Table 2. Ash content for the wood portion of these juvenile woods were 0.85, 0.93, 1.06, 1.15, and 1.24 percent for black locust, black alder, autumn olive, sycamore, and eastern cottonwood respectively, about 2 times that reported for mature wood (Chow et al. 1983). The mixture portion gave higher values of 1.42, 1.43, 1.83, 2.41, and 2.96 for autumn olive, sycamore, black alder, black locust, and eastern cottonwood, respectively. These values range from 3 to 6 times that reported for mature woods.

All independent variables such as site, spacing, portion, and species, had a significant effect on ash content at the .05 alpha level for both the LSD t-test and Duncan's Multiple Range test for differences between means (Tables 3, 4). For the variable of site, upland juvenile hardwoods had the higher mean ash content with a value of 1.67 percent, while the bottomland young trees had an average ash content of 1.39. Wood portion showed large differences with mean values of 2.01 and 1.05 percent for the mixed and wood portions respectively. Means for species were all significantly different. Eastern cottonwood had the highest mean ash content while autumn olive had the lowest. Mean values were 2.10, 1.63, 1.38, 1.29, and 1.24 percent for eastern cottonwood, black locust, black alder, sycamore, and autumn olive, respectively.

Table 3. Summary of Duncan's Multiple Range Test by Site, Spacing and Portion

	Site	Spacing	Portion
Ash Content	A2 A1	B1 B2	C1 C2
Extractives			
Alcohol Benzene	A1 A2	B2 B1	C1 C2
Hot-water	A2 A1	B2 B1	C1 C2
1% NaOH	A1 A2	B2 B1	C1 C2

1/ Significance level 0.05 degree of freedom 195.
2/ Means with same line are not significantly different.
3/ Left to right are highest to lowest means.
4/ A_1 = bottomland, A_2 = upland, B_1 = narrow spacing, B_2 = wide spacing, C_1 = mixture, C_2 = wood.

In the analysis of variance, all main effects and interaction effects were significant for type I and type III sum of squares, with the exception of the site and portion interaction (Table 5).

The higher value of ash content for the mixture over that of wood can be explained by the inclusion of the bark. It was reported that bark tends to have a higher ash content than wood (Tillman et al. 1981). Juvenile wood tends to have a higher ash content than the mature wood of the same species. This may also be due to the need for large quantities of mineral substances necessary for growth in the juvenile stages of wood development.

Table 4. Duncan's Multiple Range Test by Species

Ash content	S4	S3	S2	S5	S1
Extractives:					
Alcohol-Benzene	S4	S2	S3	S5	S1
Hot-water	S4	S2	S3	S1	S5
1% NaOH	S4	S2	S3	S5	S1

1/ Significane level = 0.05, degree of freedome = 195.
2/ Left to right are highest to lowest means.
3/ S1 = Autumn olive, S2 = Black alder, S3 = Black locust, S4 = Eastern cottonwood, S5 = Sycamore.

Table 5. Factorial Analysis1/

Independent Variable	Ash Content	Extractives: Hot-water	Extractives: 1% NaOH	Extractives: Alcohol-Benzene
A (Site)	HS	N	HS	HS
B (Spacing)	HS	HS	HS	HS
A x B	HS	HS	HS	N
C (Wood portion)	HS	HS	HS	HS
A x C	N	HS	N	HS
B x C	HS	HS	HS	HS
A x B x C	HS	HS	N	HS
D (Species)	HS	HS	HS	HS
A x D	HS	HS	HS	HS
B x D	HS	HS	HS	HS
A x B x D	HS	HS	HS	HS
C x D	HS	HS	HS	HS
A x C x D	HS	HS	HS	HS
B x C x D	HS	HS	HS	HS
A x B x C x D	HS	HS	HS	HS

1/ HS = significant difference at 1% level; N = no significance at 5% level.

Alcohol-Benzene Extractives

Alcohol-benzene soluble material consists mainly of resins, fatty acids, waxes, colorings and some phenolic compounds. Alcohol-benzene extractive content for mature hardwoods has been reported to be about 2 percent (Murko 1970, Rydholm 1964).

Values of alcohol-benzene extractive content for all combinations of independent variables are shown in Table 2. Extractive contents for the wood portion were 1.32, 3.02, 3.63, 4.68, and 5.34 percent for autumn olive, sycamore, black locust, black alder, and eastern cottonwood, respectively. The mean of 3.6 percent is about two times that generally reported for mature woods. The mixture portion had higher values of 2.68, 3.53, 4.71, 7.46, and 10.76 percent for autumn olive, sycamore, black locust, black alder, and eastern cottonwood, respectively. The mean of 5.83 percent is about three times that reported for mature woods.

The higher extractives content for the mixture portion over that of the wood can be explained by the inclusion of the bark and sapwood in the test material. The growing portion of the stem contains larger amounts of sugars and fatty acids. Bark contains larger quantities of resins, waxes, and tannic acids compared to wood (Haygreen et al. 1982).

All means of levels of independent variables: site, spacing, portion, and species, were significantly different at the .05 alpha level for both the LSD t-test, and Duncan's Multiple Range test for differences between means (Tables 3 and 4). The bottomland site trees had a higher extractive content than upland trees and values of 5.37 and 4.06 percent were measured. For the variable spacing, the wide spacing had a mean value of 5.14 percent and the narrow spacing had a mean value of 4.28 percent. For the variable of tree portion, the mixture contained more extractives than the wood. They had mean values of 5.83 and 3.60 percent, respectively. All species means were significantly different with values of 2.00, 3.28, 4.17, 6.70, and 8.05 percent for autumn olive, sycamore, black locust, black alder, and cottonwood, respectively.

In the analysis of variance, the site and spacing interaction was not a significant factor (Table 5). All other terms in the model were important in explaining variation. Both the portion and species factors were most important.

Hot-Water Extractives

Hot-water extractives include free sugars, free polysaccharides, inorganic salts, tannins, fatty acids and coloring matter (ASTM 1982). North American hardwoods contain an average of about 2 percent hot-water extractives (Freeman et al. 1941).

The wood portion had an average of 8.31 percent hot-water extractives, with values of 7.59, 7.61, 7.94, 8.94, and 9.45 percent for eastern cottonwood, sycamore, autumn olive, black locust, and black alder, respectively. These values range up to 4 times those reported for mature wood. The mix portion gave higher values with an average of 12.0 percent hot-water extractives. Results were 8.48, 9.76, 12.01, 13.14, and 17.60 percent for sycamore, autumn olive, black locust, black alder, and eastern cottonwood, respectively, ranging from 3 to 8 times higher than that reported for mature woods. The reasons for higher values of hot-water extractives for the mixture portion over those observed for wood were similar to those for alcohol-benzene extractives, in that the bark and sapwood are included in the mix. The juvenile wood may contain larger amounts of extractives relative to mature wood because these materials may be important in early stages of growth.

For both the LSD t-test and Duncan's Multiple Range test for differences between means, site means were not significantly different at the .05 alpha level (Tables 3 and 4). However, tree portion means were significantly different. Wood/bark mixtures had the higher value of 12.20, while the wood had the lower value of 8.31 percent. Species differences all varied significantly with values of 8.05, 8.85, 10.48, 11.30, and 12.60 percent for sycamore, autumn olive, black locust, black alder, and eastern cottonwood, respectively.

One Percent Sodium Hydroxide Extractives

One percent sodium hydroxide extractives consist mainly of fatty acids, waxes, resins, essential oils, and some lignin and low molecular weight hemicelluloses. Commercial lumber of North American hardwoods contain an average of about 20.18 percent 1% NaOH extractives (Chow et al. 1983).

One percent NaOH extractive contents for the wood portion were 26.03, 27.23, 28.86, 30.66, and 31.56 percent for autumn olive, black locust, sycamore, eastern cottonwood, and black alder, respectively (Table 2). The mean of 28.9 is about one and a half times that reported for mature hardwoods. The mixture portion had higher values of 32.32, 33.44, 36.58, 41.12, and 43.40 percent for sycamore, autumn olive, black locust, black alder, and eastern cottonwood, respectively. The mean of 37.37 percent is nearly 2 times that reported for mature wood. The reasons for higher values of one percent sodium hydroxide extractives for the mixture over those for the wood are similar to those for alcohol benzene and hot-water extractives, in that the bark and sapwood are included in the mix. Likewise, the juvenile wood may contain larger amounts of extractives relative to mature wood because these materials may be important in early stages of growth.

For the variable portion, the mixture contained more extractives than the wood with

mean values of 37.37 and 28.87 percent, respectively. All species means were significantly different with values of 29.73, 30.59, 31.91, 36.34, and 37.03 percent for autumn olive, sycamore, black locust, black alder, and eastern cottonwood, respectively.

In the analysis of variance, the site and portion, and the site and spacing and portion interactions were not significant factors. All other terms in the model were important in explaining variation (Table 5).

SUMMARY AND CONCLUSIONS

1. Based on the results of this study, we can conclude that site, tree spacing, wood material portion, and species influenced the chemical properties of five species of juvenile hardwoods.

2. Comparison of dependent variables between bottomland and upland sites showed all means with the exception of the hot-water extractives content, varied significantly. Trees grown on bottomland sites exhibited significantly higher alcohol-benzene extractives, and one-percent sodium hydroxide extractive contents. Trees grown on upland sites exhibited significantly higher ash contents.

3. Comparison between narrow spaced and wide spaced tree plantations showed that the dependent variables varied significantly. The narrow spaced trees exhibited significantly higher values for ash content. Wide spaced trees gave significantly higher alcohol-benzene extractives, hot-water extractives, and one-percent sodium hydroxide extractives.

4. In the comparison of the wood material portion, the mixture portion means varied significantly from those of the wood portion for all dependent variables. The mixture portion contained significantly higher amounts of ash, and alcohol-benzene extractives.

5. In comparisons among species, eastern cottonwood was found to have the highest ash content, alcohol-benzene extractives, hot-water extractives, and one-percent sodium hydroxide extractives.

6. When comparing the wood portion of juvenile hardwoods with accepted values for mature hardwoods, juvenile wood had higher amounts of ash, alcohol-benzene extractives, hot-water extractives, and one-percent sodium hydroxide extractives.

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