# SOME CHEMICAL CONSTITUENTS OF TEN-YEAR-OLD AMERICAN SYCAMORE AND BLACK LOCUST GROWN IN ILLINOIS

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ABSTRACT

Research was initiated to determine the effects of site (upland, bottomland) and tree origin (seedling, coppice) on the chemical composition of wood of two, ten-year-old hardwood species grown in Illinois. Ten-year-old black locust (*Robinia pseudoacacia* L.) and American sycamore (*Platanus occidentalis* L.) were evaluated for alcohol-benzene extractives, hot-water extractives, one-percent NaOH extractives, Klason lignin, holocellulose, and alpha-cellulose. Black locust had statistically higher alcohol-benzene extractives, hot-water extractives, and alpha-cellulose content than sycamore. A relationship between wood density and alpha-cellulose exists. Black locust yielded a higher mean Klason lignin value, and sycamore yielded a higher mean one-percent sodium hydroxide value but the difference was not significant at the $\alpha = 0.05$ level. The effects of site and origin were inconsistent for the different chemical properties. Black locust appears to be a favorable species for a variety of chemical constituents and can be successfully grown under different silvicultural methods.

*Keywords:* American sycamore, black locust, alpha-cellulose, bottomland, coppice, extractives, holocellulose, Klason lignin, seedling, upland.

Wood is a variable, complex substance. The principal constituents of wood are the polysaccharides, lignin, and extractives. It is possible that fast-grown hardwood biomass can replace, or at least supplement, traditional sources of raw materials for products that petroleum and timber now provide. In addition, wood may be converted into useful chemical compounds such as ethanol, furfural, benzene, various phenolics and sugars, rayon, and other chemicals.

Wood extractives are found as surface deposits on cell lumens and inside cell walls (Panshin and DeZeeuw 1970). The main function of extractives in living trees is as food reserves (fat, fatty acids, sugars, and starch), protectants (terpenes, resins, phenolic compounds), and as plant hormones such as phytosterols (Rydholm 1965). The extractive content of domestic woods has been shown to vary from 2–25% of dry weight depending on species (Summitt and Sliker 1980). The recent discovery of the

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possible cancer cure, taxol, from extractive tests performed on Pacific yew (*Taxus brevifolia* Nutt.) has renewed interest in wood extractives.

Alpha-cellulose, hemicellulose, and lignin are the major components of a woody cell wall, while the middle lamella consists largely of lignin. Wood is a composite material, and all three constituents determine its mechanical strength properties.

Black locust (*Robinia pseudoacacia* L.) is a shade intolerant, fast-growing species. It is a very adaptive species that will grow on a wide variety of sites, especially old fields of failed agricultural practice and other cleared areas. Black locust is often planted in southern Illinois after an area has been strip-mined because it will grow rapidly, stabilize the soil, and add nitrogen. American sycamore (*Platanus occidentalis* L.) is an intermediate to intolerant species that commonly grows in bottomland sites and along stream banks. Sycamore thrives in the moist bottomland sites and is one of the largest trees in the eastern hardwood forest.

Previous research has shown great potential for using short-rotation juvenile hardwood as raw material for industrial fuels, organic fuels, and chemicals (Chow et al. 1980; Chow and Rolfe 1983; Murphey 1961; Olson and Carpenter 1985). There is a lack of information on the chemical characteristics of 10-year-old black locust and American sycamore.

## OBJECTIVES

The basic experimentation conducted for this study was the chemical analysis of wood of two 10-year-old juvenile hardwood species obtained from different sites and origins at the Illinois Agricultural Experimental Station, Dixon Springs, Simpson, Illinois. These two species were selected because of their potential for rapid-growth plantation silvicultural systems. The objectives of the investigation were as follows: 1. to determine the wood chemical constituents of two juvenile hardwood species; 2. to determine the effects of different sites (bottomland and upland) and tree origin (coppice and seedling) on the chemical constituents of wood of two juvenile hardwood species.

## EXPERIMENTAL DESIGN AND ANALYSES

The main goal of the experiment was to determine a favorable species for wood chemical constituents. The treatment means were undertaken by a randomized complete block design (RCB) and considered as separate factors (Steel and Torrie 1980; Box et al. 1978). The overall analysis of the RCB was a 2 × 2 × 2 factorial, for each dependent variable. Particular attention was given to ensure homogeneity in all treatment groups. It was assumed that samples were normally distributed with different means and with a common variance. The linear model used for explaining sources of variation was established to be the following:

$$Y_{ijkl} = \mu + R_i + A_j + B_k + C_l + AB_{jk} + AC_{jl} + BC_{kl} + ABC_{jkl} + \epsilon_{ijkl} \quad (1)$$

where

$Y_{ijld}$ = any observed value for a chemical property, $\mu$ = overall mean, $A_j$ = site effect, $B_k$ = tree origin effect, $C_l$ = species effect, $R_i$ = replication effect, $\epsilon_{ijkl}$ = experimental error, i = 1–12 replicates, j = 1–2 levels of factor A, k = 1–2 levels of factor B. l = 1–2 levels of factor C.

Treatment levels for the site factor (A) were established to be the following: $A_1$ = bottomland, $A_2$ = upland. Treatment levels for the tree origin factor (B) were established to be the following: $B_1$ = coppice, $B_2$ = seedling. Treatment levels for the species factor (C) were considered to be as follows: $C_1$ = black locust (*Robinia pseudoacacia* L.), $C_2$ = American sycamore (*Platanus occidentalis* L.) All combinations of letters in the model represent interaction effects of the independent variables.

All statistical analysis was conducted using SAS programming methods (SAS 1989) in conjunction with analysis of variance (Steel and Torrie 1980; Box et al. 1978) on the University of Illinois at Urbana-Champaign mainframe computer system. The significance of each factor and factor interactions were determined at both the $\alpha = 0.05$ and $\alpha = 0.01$ levels, and F-tests were constructed using Type III Sum of Squares.

The tree factor was considered to be nested with the species, origin, and site factors since two trees for each species were harvested for all combinations of origin (seed, coppice) and site (bottomland, upland). Therefore, the mean square error (MSE) term was specified in the analysis of variance to be tree (species × origin × site) and an overinflated estimate of experimental error ($\epsilon$) was avoided.

TABLE 1. *Average height and diameter of experimental trees.*[a]

| Species | Site | Origin | Tree ht. (m) | DBH (mm) |
|---|---|---|---|---|
| Black locust | upland | seedling | 5.6 | 103 |
| | upland | coppice | 8.1 | 128 |
| | bottomland | seedling | 8.1 | 119 |
| | bottomland | coppice | 11.4 | 103 |
| American sycamore | upland | seedling | 6.7 | 112 |
| | upland | coppice | 9.1 | 94 |
| | bottomland | seedling | 8.4 | 105 |
| | bottomland | coppice | 11.4 | 109 |

[a] Each value is an average for two trees.

## MATERIALS AND METHODS

Trees were harvested in February of 1992 from the University of Illinois' Dixon Springs Agricultural Center (DSAC) in the Shawnee National Forest in southern Illinois. The plots at DSAC have limited agricultural value because they are characterized by low soil depth, low productivity, and susceptibility to erosion. The soil at DSAC is a Gransburg silt loam (Majerus and Rolfe 1987).

Two 10-year-old trees from each of four experimental plots were felled, limbed, and bucked for each species. Single-species plots were categorized according to site (upland, bottomland) and tree origin (seedling or coppice). The age of the original trees that were coppiced was 2 years. The spacing for all trees was 71 mm by 142 mm. A total of eight experimental plots were visited in order to obtain all four possible combinations between site and origin for both species. Felled trees were measured for total tree height and diameter at breast height (DBH) (Table 1). Depending on the height of the tree, individual trees were bucked into 4 to 7 sections. Each section measured approximately 1.5 m in length. They were transported to the University of Illinois Wood Chemistry Laboratory for further processing. A disk, comprised entirely of sapwood, was removed from each section of the log. The specific gravity (SG) of each log was determined in accordance with ASTM D-143 (ASTM 1993). The bark from each disk was removed with a pocketknife. The debarked disks from each sample tree were cut on a band saw into a mixture of small pieces that could be fed into a Wiley Mill. All material was ground in the Wiley Mill until it would pass through a No. 40 mesh sieve (425-$\mu$m) yet also be retained on a No. 60 mesh sieve (250-$\mu$m). The ground wood material from all sections of each sample tree was first thoroughly mixed and then kept in a separate bottle.

### *Test methods*

All chemical analysis and procedures followed the methods described in the standards of the Technical Association of the Pulp and Paper Industry (TAPPI) and the American Society for Testing and Materials (ASTM) as shown in Table 2. Six tests were made for each

TABLE 2. *Test methods.*

| Chemical properties | TAPPI[a] | ASTM[b] |
|---|---|---|
| Extraction: | | |
| Hot-water | — | D1110 |
| 1-percent sodium hydroxide | T4m | D1109 |
| Alcohol-benzene | T6m | D1107 |
| Pentosan | T19m | — |
| Lignin | T13m | D1106 |
| Holocellulose | — | D1104 |
| Alpha-cellulose | — | D1103 |

[a] TAPPI 1970.
[b] ASTM 1993.

TABLE 3. *Comparison of average chemical constituents and specific gravity of 10-year-old trees and lumber.*

| Chemical constituent | Black locust Age-10 | Black locust Lumber[1] | Sycamore Age-10 | Sycamore Lumber[1] |
|---|---|---|---|---|
| Alcohol-benzene extractives (%) | 3.2 | 5.1 | 1.4 | 3.4 |
| Hot-water extractives (%) | 5.3 | 5.2 | 3.7 | 3.1 |
| One-percent NaOH extractives (%) | 19.1 | 21.5 | 21.7 | 23.5 |
| Klason lignin (%) | 20.0 | 29.4 | 18.5 | 21.5 |
| Holocellulose (%) | 75.3 | 63.7 | 79.6 | 67.9 |
| Alpha-cellulose (%) | 63.0 | 45.0 | 55.4 | 36.0 |
| Specific gravity (green vol. & oven-dry weight basis) | 0.51 | 0.66[2] | 0.38 | 0.46[2] |

[1] Source: Lee (1981).
[2] Source: U.S. Forest Products Laboratory (1987).

replicate and each combination of independent variables.

## RESULTS AND DISCUSSION

Wood extractives can be isolated from wood by extraction with various organic solvents. Each solvent will isolate different chemical compounds based on polarity. Therefore, it is recommended to use multiple methods of extractive content determination in order to truly assess a species' extractive content. The solvents selected for this research included alcohol-benzene, hot-water, and one-percent sodium hydroxide (1% NaOH).

### *Alcohol-benzene extractives*

The main components of alcohol-benzene extractives are certain phenolic substances (including sterols, tannins, and phlobaphenes), waxes, fats, resins, select organic acids (namely resin acids, and amino acids, and other compounds such as pigments (Browning 1963).

Results in Tables 3 and 4 show that black locust had significantly higher alcohol-benzene extractive content than that of sycamore (3.2% compared to 1.3). There was no significant effect attributable to site or origin. However, the species × origin and site × origin interactions were highly significant (Table 4). The upland trees yielded an overall mean of 2.4% compared to the 2.1% from the bottomland trees. The trees of coppice origin gave a grand mean alcohol-benzene extractive content of 2.2%, and the seed trees were slightly higher with a value of 2.3% (Table 4). The alcohol-benzene extractive content for individual trees is presented in Table 5.

Extractives are considered to be a product of physiological growth (Kramer and Kozlowski 1979). Any factor, such as site of tree origin, that favors growth rate should also increase

TABLE 4. *Analysis of variance (F values).*

| Source of variation | df | Extractives: Alcohol-benzene | Extractives: Hot-water | Extractives: 1% NaOH | Lignin: Klason lignin | Polysaccharides: Holo-cellulose | Polysaccharides: Alpha-cellulose |
|---|---|---|---|---|---|---|---|
| Species | 1 | 68.9** | 9.7* | 4.5 | 1.0 | 42.4** | 7.9* |
| Origin | 1 | 0.1 | 0.1 | 24.2** | 4.6 | 18.3** | 0.5 |
| Site | 1 | 3.4 | 8.1* | 1.5 | 1.0 | 0.00 | 1.9 |
| Species × origin | 1 | 17.9** | 0.2 | 3.6 | 0.00 | 0.7 | 0.8 |
| Species × site | 1 | 2.0 | 0.9 | 1.3 | 1.3 | 2.0 | 0.2 |
| Site × origin | 1 | 12.6** | 8.7* | 0.1 | 1.1 | 0.1 | 6.8* |
| Species × site × origin | 1 | 0.1 | 2.5 | 6.5* | 0.1 | 5.2 | 0.1 |
| Tree(species × site × origin) | 8 | | | | | | |

Note: ** denotes significance at the $\alpha = 0.01$ level. * denotes significance at the $\alpha = 0.05$ level. F crit df (1,8) = 5.32 at $\alpha = 0.05$. F crit df (1,8) = 11.26 at $\alpha = 0.01$.

TABLE 5. *Mean values of all extractive tests for all possible combinations of species, site, and origin.*[1]

| Species | Site | Origin | Alcohol-benzene (%) | Hot-water (%) | 1% NaOH (%) |
|---|---|---|---|---|---|
| Black locust | upland | seedling | 3.6 (10.5)[2] | 5.1 (15.7) | 18.4 (4.3) |
| | upland | coppice | 3.4 (6.4) | 6.86 (12.0) | 20.3 (3.6) |
| | bottomland | seedling | 3.7 (8.3) | 5.4 (14.0) | 18.4 (10.4) |
| | bottomland | coppice | 1.9 (14.1) | 3.1 (18.7) | 19.3 (4.8) |
| American sycamore | upland | seedling | 0.6 (15.4) | 3.8 (15.1) | 21.6 (2.9) |
| | upland | coppice | 2.2 (19.0) | 4.5 (20.0) | 21.7 (5.5) |
| | bottomland | seedling | 1.2 (21.3) | 3.5 (16.6) | 21.1 (5.1) |
| | bottomland | coppice | 1.4 (15.0) | 3.0 (14.3) | 22.2 (3.7) |

[1] All mean values represent percentages of oven-dry wood weight and 12 tests.

[2] Numbers in parentheses are coefficient of variation (CV%). In which, $CV\ (\%) = \frac{\text{standard deviation}}{\text{mean}} \times 100$.

extractive content by any method of extractive content determination. However, the results show that the plots varied in terms of rank of alcohol-benzene extractives. The plots with the greatest growth rate did not show the highest alcohol-benzene extractive content (Table 5).

The alcohol-benzene extractive content of mature hardwoods has been reported to be approximately 2% (Murko 1970; Rydholm 1965). Three-year-old black locust has been shown by Chow et al. (1987) to have an alcohol-benzene extractive content ranging from 2.6%–5.3%, and sycamore displayed a lower value ranging from 2.1%–3.5%.

### *Hot-water extractives*

The major components of hot-water extractives include tannins, gums, sugars, coloring matter, and starch (ASTM 1993).

Again, black locust (5.3%) had higher average hot-water extracted content than sycamore (3.7%) (Table 3). However for this particular test, the juvenile sycamore trees had a higher hot-water extractive content than commercial sycamore lumber (Table 3).

This was the only method of extractive content determination in which site was found to be a significant variable (Table 4). The hot-water extractives content of the upland sites (5.0%) was significantly higher than that of the bottomland sites for coppiced trees (3.8%) (Tables 4 and 5). The growth origin factor was not significant in the analysis of variance. The coppice trees (4.5%) were slightly higher than the seed trees (4.3%) for hot-water extractives (Tables 4 and 5). Again, no relationship exists between growth rate and extractive content based on the extractive yield of the individual plots (Table 6).

Freeman and Peterson (1941) reported that

TABLE 6. *Mean values of Klason lignin, holocellulose, and alpha-cellulose for all possible combinations of species, site, and origin.*[1]

| Species | Site | Origin | Klason lignin (%) | Holocellulose (%) | Alpha-cellulose % |
|---|---|---|---|---|---|
| Black locust | upland | seedling | 20.7 (7.4)[2] | 71.3 (1.5) | 64.1 (1.9) |
| | upland | coppice | 19.6 (8.5) | 78.1 (4.2) | 57.1 (1.6) |
| | bottomland | seedling | 22.0 (6.4) | 77.4 (0.9) | 61.6 (2.6) |
| | bottomland | coppice | 17.8 (7.0) | 74.5 (1.2) | 69.1 (4.3) |
| American sycamore | upland | seedling | 17.9 (8.3) | 78.4 (1.8) | 59.6 (4.7) |
| | upland | coppice | 16.8 (5.7) | 79.7 (3.3) | 48.3 (3.3) |
| | bottomland | seedling | 21.4 (3.5) | 77.4 (0.9) | 55.3 (2.7) |
| | bottomland | coppice | 18.1 (9.8) | 83.2 (1.7) | 58.6 (2.4) |

[1] All mean values represent percentages of oven-dry wood weight and 12 tests.

[2] Numbers in parentheses are coefficient of variation.

the North American hardwoods contain an average of about 2% hot-water extractives. Three-year-old black locust has been shown to possess a hot-water extractive content of 7.5%–10.4%, and sycamore of the same age showed a range of 6.7%–7.8% as reported by Chow et al. (1987).

*One-percent sodium hydroxide extractives*

The major constituents of 1% NaOH extractives are fatty acids, and low molecular weight carbohydrates consisting mainly of hemicellulose and degraded cellulose (ASTM 1993).

This was the only method of extractive content determination in which black locust was not found to be significantly greater than sycamore (Table 4). In fact, sycamore (21.7%) obtained a greater 1% NaOH extractive content than black locust (19.1%) (Table 4).

The average value of matter soluble in caustic soda in upland trees was about the same as that in the bottomland trees by a margin of 20.5% to 20.3%, respectively. Therefore, the site factor was not significant in the analysis of variance (Table 4). The trees of coppice origin gave a mean of 20.9%, which was slightly greater than the 19.9% for the seed trees, and the origin factor was found to be highly statistically significant in the analysis of variance (Table 4). The individual plots showed very little variation for 1% NaOH extractive content (Table 5).

Commercial lumber of North American hardwoods contains an average of about 20%, 1% NaOH extractives (Chow and Rolfe 1983). Chow et al. (1987) found 3-year-old black locust to contain a 1% NaOH extractive content of 25.7%–28.5%, and sycamore showed means ranging from 27.1%–30.5%. It is interesting to note that the sycamore at DSAC has shown a higher 1% NaOH extractive content than black locust at a tree age of three and again here at a tree age of ten.

*Klason lignin*

The analysis of variance showed no significant sources of variation (Table 4). Black locust did show a slightly higher mean Klason lignin content of 20.0% as compared to 18.5% for sycamore (Table 3). Previous research by Lee (1981) showed black locust lumber to possess a Klason lignin content of 29.4%, and sycamore lumber was found to be 21.5% (Table 3). In this study, the lignin content of the bottomland sites (19.6%) was slightly higher than for upland sites (18.5%). Trees of seed origin possessed a mean Klason lignin content of 20.3%, and coppice trees gave a value of 17.8%. The bottomland-seedling plots gave the highest Klason lignin content for both species (Table 6).

A shortcoming of this method of lignin content determination is that extraneous materials which might remain insoluble along with the lignin must be removed before the acid treatment. There is no precise separation of lignin from all other wood components that has been found possible, and the product isolated as lignin is of necessity defined by the procedure of isolation. The ideal delignification would result in a complete removal of lignin without chemical attack of the polysaccharide portion. Presently there is no delignification procedure that can satisfy this scenario (Fengel and Wegener 1984). The Klason method is ideal for softwoods but is less favorable with hardwoods because of their high content of acid soluble lignin.

It is essential that standard testing procedures be strictly adhered to when performing lignin content experimentation. The content of lignin in wood is truly a direct product of the methodology utilized. Comparisons between slightly different methods for lignin content determination are of little significance.

*Holocellulose*

This was the only chemical test in which sycamore was found to have significantly higher value than black locust (Tables 3 and 4). Sycamore yielded a mean holocellulose content of 79.6%, and black locust gave a value of 75.3% (Table 3). Both of these values are well above the values for commercial lumber of sycamore (67.9%) and black locust (63.7%) (Lee 1981). The species and tree origin factors were both highly significant in the analysis of variance (Table 4).

### *Alpha-cellulose*

Black locust had a significantly higher mean alpha-cellulose content of 63.0% as compared to sycamore 55.4% (Tables 3 and 4). The mean alpha-cellulose values obtained by this project are far greater than those of commercial lumber of the same species. Previous research by Lee (1981) showed black locust lumber to possess a mean alpha-cellulose content of 45.0%, and sycamore lumber yielded a value of 36.0% (Table 3).

The analysis of variance revealed that black locust (63.0%) had a significantly higher alpha-cellulose content than sycamore (55.4%) (Table 4). The other factors, site and origin, failed to show a statistical difference (Table 4). The species failed to show a consistent rank for alpha-cellulose content based on origin and site (Table 6).

The plots with the most rapid growth (bottomland-coppice) (Table 1) showed the modest alpha-cellulose content. Alpha-cellulose is the main component of the woody cell wall. The cell wall is the principal factor in determining the density of a particular species (Haygreen and Bowyer 1989). Therefore, a fast-growing species should have a low wood density and a low percentage of polysaccharides. The bottomland-coppice site for black locust yielded the second highest alpha-cellulose content for all four black locust plots (Table 6). The corresponding sycamore plot gave the second highest alpha-cellulose content for all of the sycamore plots (Table 6).

### *Total polysaccharide material*

The experimental procedures used allow for a summative analysis for total polysaccharide structural material. On an oven-dried basis, the holocellulose and lignin contents can be combined to form the total structural wood material. The sum is rarely 100% mainly due to the alcohol-benzene extractive content and, to a lesser degree, the destructive nature of the testing. Nonetheless, summative analyses are important in that they indicate the suitability of the procedures used, and can have implications for the pulping industry. In addition, the total polysaccharide material does not consider alpha-cellulose, which is part holocellulose. A small portion of residual lignin remains with the holocellulose portion of wood. This lignin can solubilize during the holocellulose determination and can cause errors up to 9% in the summative analysis of wood (Fengel and Wegener 1984).

In this study, the black locust contained a total polysaccharide structural material content value of 94.9%. Sycamore yielded a value of 98.1%.

## CONCLUSIONS

Based on the test results, the following summaries can be made: (1) black locust possessed higher three extractive contents than sycamore; (2) black locust and sycamore were equal in Klason lignin and holocellulose contents; (3) black locust produced more alpha-cellulose per unit of volume of biomass; (4) tree origin and site have less effect on the wood chemical constituents than the species.

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