Chemical Utilization of Western Woods:

Whitz Fir

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ACKNOWLEDGMENTS

Information for this report was gathered first by J. D. Ross and R. K. Ray. Additional references were selected by D. W. Glennie and H. Aft.

INTRODUCTION

This report is designed to summarize published work on chemical properties of white fir, <u>Abies concolor</u> (Gord. and Glend.) Lindl. It is intended to show extent of existing knowledge of chemistry of white fir, and to indicate to experienced readers possible avenues for further memical investigation of the species.

Because some works may have been omitted and because only published works through mid-1960 have been listed, this summary should not be accepted as complete. References were obtained primarily from <u>Chemical Abstracts</u>, 1907 to date, with additional references obtained from abstracted articles.

Characteristics of Growth

White fir is in the genus <u>Abies</u> because in its wood, resin canals and ray tracheids are lacking, end walls of ray parenchyma are nodular, and cross-field pitting is taxodioid $(3)^1$.

The species is one of the most important true firs. The tree grows to moderately large size, 100-150 feet high and 2-3 feet in diameter. Larger trees have been found. White fir is tolerant of shade and may have many branches on the trunk, resulting in a high proportion of knotty wood. The tree grows rapidly when young, but is short-lived; trees over 125-150 years old may be defective. The species grows

Numbers in parentheses refer to literature cited.

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mixed with Douglas fir or pines and seldom, if ever, grows in pure stands.

Bark of mature trees may be 4-6 inches thick at base of tree and is deeply furrowed, hard, and resistant to fire. Foliage is a pale bluishgreen color. Needles are considerably longer than those of other true firs, except grand fir. Wood is white, soft and light. White fir is low in strength and has little resistance to decay. It is somewhat more difficult to dry than species that are better known. This characteristic has resulted in logging other species in the past and leaving white fir uncut. In recent years, white fir has become acceptable as a species for commercial lumber.

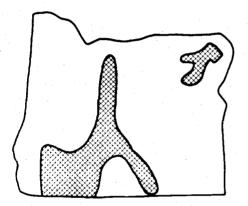


Figure 1. Range of white fir in Oregon.

While the wood is known to be excellent for production of pulp, white fir has not been utilized to much extent. Major stands of the species are located too far from pulp mills on the Pacific coast. However, as supplies of pulpwood decrease, sources of raw material may be sought farther from manufacturing centers.

Natural Range

White fir grows primarily in dry, moderately-high-altitude areas of western and southwestern United States. Largest stands are found i the interior and, to less extent, in the coastal areas of Southern Oregon and Northern California. Smaller amounts are found in most western states. Estimated stands of white fir were reported in 1945 (1).

State	Stand volume,
	Billions board fee
California	30.4
Oregon	5.6
Colorado	3.0
Utah	3.0
Idaho	1.0
New Mexico	1.0
Arizona	0.5
Montana	negligible
Nevada	negligible
Washington	negligible
Wyoming	negligible

Accurate volume of white fir being logged is not available since volumes of all true firs generally are totaled together. However, volume of logs used for lumber, plywood, and pulp from all true firs in the Pacific Northwest in 1957 was estimated to be only 0.5 billion board feet, log scale. This was about 5 per cent of the net volume of 73 billion board feet (2) of all species available in this area.

References to chemical investigations on white fir are listed according to chemical properties of wood, bark, and foliage. Very little has been accomplished in chemical utilization of white fir commercially. For this reason, most references are concerned with laboratory investigations of chemical properties. Aiyar, S. S. "Balsam of White Fir." J. of <u>Am. Pharm. Assoc</u>. 12(587-588). 1923.

> Aiyar reported on oleoresin from white fir, presumably <u>Abies</u> <u>concolor</u> (Goed) Parry. Many years had elapsed from time of collection to time of analysis.

Oleoresin was colorless, but a white frocculent precipitate formed. Oleoresin was soluble in alcohol, acetone, and cold benzene, leaving some white residue. Oleoresin was completely soluble in chloroform and ether.

Steam distillation gave 27.7 per cent volatile oil that was colorless and that had a pleasant odor.

Direct fractional distillation gave 1.4 per cent volatile oil at temperatures up to 154 F and a total of 29.8 per cent volatile oil at 176 F.

Attempts were made unsuccessfully to obtain crystalline resin acid from residue of steam distillation. On extraction of the resin with alcohol, acetone, and alcohol benzene, the Saponification number increased from 120.9 to 143.4

 Carlberg, Gary Lawrence. <u>A Comparison of the Extractives from</u> <u>Four Species of True Firs</u>. Master of Science thesis, Oregon State College, Corvallis, Oregon. May, 1960. See also <u>Tappi</u> 43 (982-988). 1960.

	Extraction solvent					
Species	Ethyl ether	Ethyl alcohol	Hot water	Total		
	Per cent	Per cent	Per cent	Percen		
Abies amabilis	0.22ª	1.40	2.22	3.84		
Abies concolor	. 24	1.83	1.28	3.35		
Abies grandis	.19	1.43	1.20	2.82		
Abies procera	.21	2.25	2.28	4.74		

In a study of extractives from noble fir, grand fir, Pacific silver. fir, and white fir, the extractive contents listed below were found.

^aPer cent of oven-dry weight of unextracted wood.

The sample of white fir was from a disc 7 inches in diameter with 13 annual growth rings. Steam volatiles from these species were obtained in extremely low yield of about 0.002 to 0.005 per cent of the wood. No terpenes were detected by gas chromatography.

Only a fraction of these extracts soluble in both water and ether gave distinctive reactions to color for the species with zinc and hydrochloric acids. This test has been applied to indicate presence of flavones and has produced a light brown solution with Pacific silver fir and grand fir, a light cherry-red solution with noble fir, and a dark red-brown solution and precipitate with white fir. This color response was not obtained when applied directly to wood.

Analysis of extracts by ultra-violet and infra-red adsorption and by paper chromatography indicated strong similarity between the species. Development of a distinguishing chemical test for the four species may be difficult.

Erdtman, H. "Constitution of the Resin Phenols and Their Biogenetic Connections. VIII. Conidendrin (Sulfite Liquor Lactone) and Its Distribution Among Various Conifers." <u>Svensk Papperstidn</u>. 47(155-159). 1944.

Ertman reported on conidendrin found in sulfite-cooked wood. Seven species of <u>Abies</u> were examined, presumably including Abies concolor. Conidendrin was found only in <u>Abies</u> arizonica.

4. Hatch, R. S. and W. F. Holzer. <u>Pacific Coast Pulpwoods</u>. Monograph No. 4, Tech. Assoc. of Pulp and Paper Ind. 1947.

> Hatch and Holzer studied general properties of western pulpwoods, including white fir. It was described as an excellent pulpwood, giving good yields of pulp and being able to be pulped by any process. Charges of white fir in digesters were about 57 per cent lower than those of hemlock because of lower density of white fir. Strength of pulp from white fir also was about 57 per cent lower than was strength of pulp from hemlock. A good balance was maintained between burst and tear with pulp from white fir. Color of pulp from white fir was better than from hemlock and pulp can be bleached.

 Hergert, H. L. and E. F. Kurth. "The Isolation and Properties of Catechin from White Fir Bark." J. Org. Chem. 18(521-530). 1953.

> Hergert and Kurth, while investigating recovery of catechin from bark of white fir, also briefly reported on catechin in wood of white fir. In wood collected near Fort Klamath, Oregon, in 1950,

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extract of crude ether was determined to be 0.12 per cent of weight of oven-dry wood.

 Hyttinen, A. and E. R. Schafer. <u>Groundwood Pulping of White Fir</u> and <u>Corkbark Fir</u>. Report No. 2175, U. S. For. Prod. Lab., Madison, Wisc. 1959.

> Recent study of groundwood pulping of white fir (and corkbark fir) was reported by Hyttinen and Schafer. Pulps from young white fir (48 years old and 14.5 inches in diameter) were stronger, lighter, and longer-fibered than those from older trees (133 years old and 11.3 inches in diameter).

Compared with typical pulps made commercially by groundwood process, pulps from white fir were similar in bursting strength, breaking length, and density. Brightness was about 10 per cent lower and resistance to tearing similar for the old white fir. Resistance to tearing was about 80 per cent higher in young white fir when compared to typical pulps.

 Isenberg, I. H. "Age and the Chemical Composition of White Fir Wood." J. <u>Am. Chem. Soc</u>. 58(2231). 1936.

> Isenberg reported on effect of age on chemical composition of white fir wood collected near Quincy, California. A single section was taken from a tree at height of 12 feet. The section then was divided into five zones by age according to annual rings. The following table gives details.

Rings from		Sap or	Radial width	Area of	Part of	Part of sap
periphery	Symbol	heartwood	of zone	zone	total	or heartwood
No.			<u>In</u> .	$\underline{Sq}/\underline{in}$.	Per cent	Per cent
1-15	Α	S	0.5	23.0	10.1	15.5
16-40	в	S	1.25	60.6	26.8	40.9
41-85	С	S	1.75	64.6	28.5	43.6
86-125	D	н	2.38	57.0	25.1	72.5
126-160	E	H	2.62	21.6	9.5	27.5
1-160	T^{a}	-	8.50	226.8	100.0	-

Measurement of Age Zones

^aTotal of all measurements.

Analytical methods of the U. S. Forest Products Laboratory wer followed to obtain these results:

Zone	Ash	Cold water	Hot water	Ether	Alc-benz.	1% NaOH	Acetic acid
A	0.38	1.46	2.35	0.21	0.92	11.28	1.64
в	0.37	0.51	1.29	0.19	0.96	10.66	1.70
C	0.38	0.21	1.19	0.20	0.94	9.95	1.71
D	0.63	2.16	2.83	0.26	1.74	11.72	1.33
E	0.64	1.55	2.04	0.22	1.49	10.77	1.25
Т	0.43	1.12	1.89	0.23	1.43	11.38	1.64
Caic T.	0.46	1.04	1.83	0.22	1.20	10.79	1.56

Solubility

Composition of Wood

Zone	Methoxyl	Nitrogen	Pentosan	Lignin	Cellulose	Pentosan in cellulose
A	4.68	0.08	8.68	26.62	64.63	6.96
в	4.62	0.06	8.46	27.29	64.56	6.33
С	4.40	0.06	8.62	27.65	64.60	7.18
D	4.68	0.06	8.83	27.31	64.53	5.75
E	5.06	0.06	10,98	28.00	63.65	9.16
Т	4.57	0.06	8.86	27.43	64.47	7.31
Calc T	4.62	0.06	8,73	27.40	64.49	-

Calculated Values for Sapwood and Heartwood

Determination	Sapwood	Heartwood
	Per cent	Per cent
Ash	0.38 ^a	0.63
Cold water sol.	0.53	1.99
Hot water sol.	1.41	2.61
Ether sol.	0.20	0.25
Alcohol-benzene sol.	0.94	1.67
1% NaOH	10.45	11.46
Acetic acid	1.70	1.31
Methoxyl	4.53	4.78
Pentosan	8.56	9.42
Nitrogen	0.06	0.06
Lignin	27.34	27.50
Cellulose	64.59	64.29

^aPer cent of oven-dry weight.

 Isenberg, I. H. and M. A. Buchanan. "A Color Reaction of Wood with Methanol-Hydrochloric Acid." J. of For. 43(888). 1945.

Isenberg and Buchanan noted that a reagent consisting of 25 ml of concentrated hydrochloric acid in 1000 ml of dry methyl alcohol developed a red-to-purple color with species of true fir. Rate of formation of color seemed to depend upon the particular species. Sapwood and heartwood may be distinguished in white fir by rapid and intense development of color in sapwood. Reaction to color may result by formation of anthocyanidin from leucoanthocyanin or related substances, according to D. W. Manson ("The Leucoanthocyanin from Black Spruce Inner Bark." <u>Tappi</u>, 43 No. 1 (59-64). 1960).

This color test is important because indicators of \underline{PH} are not effective in distinguishing sapwood from heartwood in white fir.

 Oregon Forest Research Center. Unpublished work, Project 19Z-5 Sponsored by West Coast Lumbermen's Association.

Studies at the Oregon Forest Research Center agreed with those of Carlberg on chemical tests to distinguish true firs from western hemlock. A solution of equal volumes of methyl alcohol and 5 per cent aqueous sodium hydroxide gave, with few exceptions, a dark-brown stain on either sapwood or heartwood of western hemlock. This same solution gave a yellow, yellow-green, or yellow-brown color with noble fir, grand fir, Pacific silver fir, and white fir. There was no apparent distinction of true fir species.

Ross, J. D. "Chemical Resistance of Western Woods." <u>Proceed-ings</u>, For. Prod. Res. Soc. 1955.

Ross reported on loss in breaking strength of western softwoods when exposed to a number of common acids, bases, and salts. Woods tested were Douglas fir, white fir, noble fir, western red cedar, incense cedar, Port Orford cedar, Sitka spruce, west coast hemlock, ponderosa pine, sugar pine, Idaho white fir, and redwood. Cypress and southern yellow pine were tested for comparison.

White fir gave an unexpectedly good performance on basis of difference in breaking strength before and after treatment with various common chemical solutions.

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 Thickens, J. H. and G. C. McNaughton. <u>Ground Wood Pulp</u>. U.S. Dept. of Agric. Bulletin 343. 1916.

Thickens and McNaughton reported on grinding of spruce for mechanical pulp and on suitability of a number of woods, including white fir (Abies concolor), as substitutes for spruce in ground wood pulp. White fir gave a rather soft, but satisfactory, pulp, particularly when green wood from young trees was used. Pulp from trees 40 inches in diameter was inferior in color, fiber, and yield to that from trees 18 inches and less in diameter.

Color of pulp was light enough to compare favorably with spruce, ranking 10 (young wood) and 15 (old and young mixed) when compared to 22 woods studied. Pulp from white fir ranked 11 in strength properties. When steamed before grinding, dark-brown pulp was suitable for board or cheap-grade wrapping paper. Yield was about 2000 pounds (oven-dry weight) a 100 cubic feet of solid-rossed wood.

U. S. Forest Products Laboratory. <u>Physical Characteristics and</u> <u>Chemical Analysis of Certain Softwoods (Other than Pine) Re-</u> <u>ceived at the Forest Products Laboratory from October 1, 1948</u> <u>to August 7, 1957</u>. Report PP-144.

The U. S. Forest Products Laboratory reported on analysis of white fir with specific gravity of 0.36. Sample of white fir was obtained in California.

Material	Amount
	Per cent
Holocellulose	65.5
Alpha cellulose	49.1
Lignin	27.8
Pentosans	5.5

Composition of White Fir

Extractives^a

1% NaOH	Hot water	Alcohol-benzene	Ether	Ash
12.7	5.2	2. 1	0.3	0.4

^aBased on moisture-free wood.

12.

Wells, S. D. and J. D. Rue. <u>The Suitability of American Woods</u> for Paper Pulp. U. S. Dept. of Agric. Bulletin 1485. 1927.

Wells and Rue reported on pulping of 94 species of wood by soda, sulfate, sulfite, and mechanical processes. White fir was among those species tested for all but the soda process. Other names in use for this species were balsam fir, silver fir, white balsam, bastard pine, and blue fir. Oven-dry weight of a cubic foot, green volume, was 22 pounds. Fiber length was 3.5 mm.

Sulfate process

White fir reduced readily to pulp by the sulfate process. The unbleached pulp was very strong. The yield of the oven-dried pulp that was strong was 48-53 per cent of the oven-dry weight of the chips. From 38 to 43 per cent of the pulp yield was pulp for bleaching. From 20 to 30 per cent of bleaching powder of standard strength was required for bleaching white fir.

Pulp from white fir produced by the sulfate process is suited for high-grade kraft wrapping papers and fiber board.

Sulfite process

With the sulfite process, white fir also reduced readily. The unbleached pulp had excellent color and strength. Fibers of white fir were larger and somewhat coarser than were fibers of spruce. Pulp from white fir was easily bleached. The yield of the ovendry pulp was 45-50 per cent of the oven-dry weight of the chips. The bleaching requirement was from 10 to 15 per cent of bleaching powder of standard strength.

The sulfite process produces pulp from white fir suitable for news, wrapping, book, and high-grade printing papers.

Mechanical process

White fir reduced readily by the mechanical process. The pulp produced had excellent color and standard strength. Pulps of better quality are usually produced from young trees than from old trees because of the susceptibility of this species to heart rot. From 15 to 20 per cent more power was required to produce white fir pulp by the mechanical process than was needed for spruce.

Pulp produced by this process is suited for practically all uses requiring ground wood.

CHEMICAL PROPERTIES OF BARK

 Blasdale, W. C. "On Heptane from Coniferous Trees." J. <u>Am</u>. <u>Chem. Soc</u>. 23(163-164). 1901.

Blasdale, while examining exudations from various Pacific coast conifers, reported on white fir.

"Abies Concolor var. Lowiana - (Exudation) obtained at Lake Tahoe from the cavities in the outer bark. It is a light yellow oleo-resin which closely resembles Canada balsam (from <u>Abies Canadensis</u>) and could undoubtedly be used as the equivalent of it. It yielded 20% of a terpene-like liquid, most of which distilled between 155° and 160°."

Data covering this sample were reported as follows:

	mple	Boiling	S p. Gr.	Index of	Sp.	Iodine
	No.	Point	at 15 ⁰	Refraction at 15 ⁰	Rotation	Absorption
Ŭ	5	155-160 ⁰	0.8578	1.4738	-7 ⁰ 91	very high

 Hergert, H. L. "Chemical Composition of Cork from White Fir Bark." For. Prod. J. 8(335-339). 1958.

> In recent studies of the cork fraction of white fir bark, Hergert determined the composition to be (oven-dry basis): 26 per cent extractives (wax, catechin, epicatechin, tannin, phlobaphene and carbohydrates); 18.8 per cent hydroxy fatty acids (hydroxy myristic acid, hydroxy arachidic acid and others); 26.7 per cent phenolic acid (a complex polyphenol closely resembling the phlobaphene present in the extractive fraction); 4.7 per cent lowmolecular-weight phenols (principally ferulic acid); 8.2 per cent cell-wall carbohydrates (probably cellulose since glucose was the main sugar obtained on acid hydrolysis); and 15.6 per cent lignin.

> The cork fraction in air-dried pulverized bark could be separated effectively by suspending the particles in water at 60 to 85 C and skimming off the cork. Only partial separation of Douglas fir bark was obtained under similar conditions. Hergert noted that hydroxy fatty acids have commercial potential as low-temperature lubricants.

^{3.} Hergert, H. L. and E. F. Kurth. "The Chemical Nature of the Extractives of White Fir Bark." Tappi 36(137-144). 1953.

Hergert and Kurth reported on extractives in white fir bark, collected in Southern Oregon and Northern California during 1950. Authors' summary follows:

"The extractive content of white fir bark ranged from 17 to 22% of the dry weight of the bark. The hexane extractive, a lightbrown wax obtained in about a 2.5% yield, was composed of free lignoceryl alcohol and behenic acid, and of combined lignoceryl alcohol, phytosterol, unsaturated alcohols, combined behenic acid, and unsaturated acids. Benzene extraction of the hexaneextracted bark residue produced a dark brown wax, in about a 1% yield, that was composed of lignoceryl alcohol, phytosterol, behenic acid, 13-hydroxy myristic acid, hexane-insoluble unsaturated acids, and a phenolic acid. By extracting this bark residue with ethyl ether, d-catechin, 1-epicatechin, and 3'-methoxy dihydroquercetin were obtained in about a 2% yield. A phlobatannin was obtained in about a 7% yield by hot water extraction. Alcohol extraction of the solvent and water extracted bark resid due yielded about 2% phlobaphene. The cork fraction, which com prised roughly 40% of the bark and was easily separable by screening hogged bark, contained 6% wax, 6 to 16% catechin, 5% tannin, and 3 to 7% phlobaphene."

 Hergert, H. L. and E. F. Kurth. "The Isolation and Properties of Catechin from White Fir Bark." J. Org. Chem. 18(521-530). 1953.

> Hergert and Kurth investigated recovery and properties of catechin from white fir bark collected near Fort Klamath, Oregon, in July, 1950. Results of ether extraction of several bark fractions from trees of various ages were reported.

General method of isolation was extraction of benzene in bark to remove waxes, extraction of ether to remove crude catechin, and recrystallization of ether extract from hot water. Recovery was also accomplished by direct hot water extraction of the bark, concentration of liquor, and subsequent recrystallization from hot water.

Good yields of d-catechol and 1-epicatechol were obtained by ether or hot-water extraction of white fir bark. Two crystalline forms of d-catechol were obtained, depending upon the concentration of the solution from which crystallization took place. Pentamethyl epicatechol was obtained by a modified clemmensen reduction of pentamethyl dihydroquercetin. Infrared spectra of catechol and its derivatives are presented and interpreted.

Source	Age of Tree ^a	Yield ^b
	Years	Per cent
White fir wood	145	0.12
Whole bark	209	3.80
Inner bark	209	0.07
Outer bark ^C	209	1.28
Cork	209	8.98
Cork	152	5.93
Cork	241	16.62

Yield of Crude, Ether-soluble d-Catechol and 1-Epicatechol

^aAverage age of three tree samples. Samples obtained 3 feet from base of tree. Per cent of oven-dry weight.

CExcluding cork.

Schorger, A. W. "Oils of the Coniferae. II. The Leaf and Twig, and Bark Oils of White Fir." J. Ind. Eng. Chem. 6(809-810). 1914.

Schorger reported on nature of oils recovered from bark of small white fir trees cut for poles. Yield of bark oil ranged from 0.07 per cent to 0.12 per cent.

Approximate composition of bark oils was determined to be:

Component	Part of Oil
	<u>Per</u> <u>cent</u>
Furfural	trace
1-a-pinene	9.0
1-camphene	0.0
l-b-pinene	60.0
1-phellandrene	0.0
Dipentene	12 to 13
Ester as bornyl acetate	2.5
Free borneol	4.5
"Green oil"	5.0
Losses	7.0

CHEMICAL PROPERTIES OF FOLIAGE

 Curkal, H. and J. Janak. "Identification of Some Terpenes in Essential Oils of Conifers by Gas Chromatography." Collection of Czechoslovak Chemical Communications. 25(1967-1974). 1959.

> Curkal investigated the oils obtained by commercial steam distillation of needles of white fir. Analysis by gas chromatography showed presence of seven constituents that were not identified.

 Schorger, A. W. "Oils of the Coniferae. II. The Leaf and Twig, and Bark Oils of White Fir." J. Ind. Eng. Chem. 6(809-810). 1914.

Schorger investigated oils from leaves and twigs of white fir, Abies concolor (Gord.) Parry. Properties of oils of leaves were reported.

	<u> </u>								
			Asid	Ester	Ester No. after	Ace-	Alco	ohol	
		·			1	· ·			
d150	ND 150	Ad25 ^o	No.	No.	acetyl	tate	Free	Total	Yield
						<u>%</u>			<u>%</u>
0.8758	1.4788	-27.94	1.13	12.52	48.88	4.38	10.28	13.73	0.140
0.8738	1.4786	-20.18	i.81	27.34	54.58	9.57	7.65	15.17	0.029
0.8732	1.4786	-21.65	1.43	24.03	52.45	8.41	7.99	14.60	0.050
0.8737	1.4796	-26.87	1.07	20.55	54.22	7.19	9.50	15.15	0.150
0.8720	1.4787	-26.59	1.01	14.48	51.83	5.07		14.55	
0.8745	1.4790	-24.08	1.32	14.80	47.84	5.18	,	13.39	0.095
0.8777	1.4781	-20.11	1.06	18.79	55.51	6.58	·	15.56	
					Mean	6.63	9.39	14.59	0.128

Approximate composition of leaf and twig oils also was reported.

Component	Part of oil
	Per cent
Furfural	Trace
<u>1-a-pinene</u>	12
1-camphene	. 8
1-b-pinene	42
1-phellandrene	15
Ester as bornyl acetate	6.5
Free borneol	9.5
"Green oil"	3
Losses	4

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