

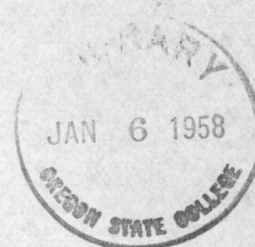
1.

The Chemical Analysis of Lodgepole Pine
to Further its Utilization.

by

Myrno A. Madden

A Thesis
Presented to the Faculty
of the
School of Forestry
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In Partial Fulfillment
of the Requirements for the Degree
Bachelor of Science
June 1941

Approved:

Professor of Forestry

Permanized
OLD RELIABLE
SCHOOL OF FORESTRY
OREGON STATE COLLEGE
CORVALLIS, OREGON

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WAG CONTENT

Introduction

The presence of conditions that drain our woods is bound to reduce the kinds of trees that are considered the best. In choosing the topic, "Chemical Analysis of Lodgepole Pine to further its Utilization", I had two perspectives in view. One, the use of Lodgepole pine to replace diminishing species; and second, the use of Lodgepole pine in a field all its own.

The present day usage of Lodgepole pine is rather limited to local areas in which case it is used as firewood, poles, ties, and lumber. The reason for such small usage is due to the fact that the major properties of this tree are surpassed by the other trees that are apparently plentiful. It is with this small usage in mind, that I chose to analyze this wood, so that in the future it may have its place among other trees.

There has been little done in the line of study on Lodgepole pine, as there has been no necessity to make such a study. A few experimenters have run strength tests which are closely related to the length of the fibers. I found no experiments concerning the composition of this wood.

The "Methods Used at the Forest Products Laboratory for the Chemical Analysis of Pulps and Pulpwoods" was used as a laboratory manual throughout this analysis.⁽¹⁾

Obtaining and Preparing the sample for Analysis

The sample of wood I used was taken from a thicket of Lodgepole pine (*Pinus Contorta*, Douglas) located about two miles southeast of Newport, Oregon. The sample was one from an 8 inch tree about 40 feet high, and showed signs of a strong ocean breeze in that there was slight compression wood. The three foot sample was taken about $4\frac{1}{2}$ feet above the ground. The sample was taken from between knot growths, and as a result was free of knots.

After taking the bark from the sample, it was sawed into sawdust with a small circular electric driven saw. The sawdust so made was run through a hammer mill. The resulting sample ranged from material retained on a 40 mesh screen to material passing through 100 mesh. The sample I used was that retained on an 80 mesh screen and passing a 60 mesh. To be sure of a representative sample, the course material was run through the hammer mill again and again until all the sample passed a 60 mesh screen.

The sawdust sample was allowed to dry 24 hours after sawing. This prevents matting of the sawdust in the hammer mill, and clogging of the screens.

Procedure Used in the Analysis

The procedure precedes each individual experiment and is not especially important to this part of the thesis.

Discontinued

Tabulated Results of My Analysis

Determination	Percentage
2. Ash	0.24
3. Cold Water Solubility	2.65
4. Hot Water Solubility	3.36
5. 1% NaOH Solubility	14.37
6. 10% KOH Solubility	20.84
7. Ether Solubility	0.91
8. Alcohol-benzene Solubility	4.14
9. Lignin	26.21
10. Holocellulose*	60.96
11. Cellulose*	56.47

* -- The Holocellulose and Cellulose determinations are a little low, as the chlorine lightly attacks the cellulose in the removal of lignin.

Interpretation of the above Results

Ash:

Without this analysis, a person would have no idea of the amount of ash in Lodgepole pine. If a tree has a large ash content, there is possibilities of utilizing the ash for its potash content as well as its other constituents. The potash could be used for fertilizer. In the case of the Lodgepole pine, the ash content is low, being 0.24%.

Ash from other trees that have been analyzed, is composed of the following compounds: K_2O , P_2O_5 , CaO , MgO , Fe_2O_3 , SO_3 , SiO_2 , and Na_2O .⁽²⁾

Further analysis of ash probably will in the future be correlated with the growth of trees. Just what part these

inorganic compounds play is not definitely known.

Water Solubilities:

These values give an idea regarding the percentage of tannins and carbohydrate materials that are present in the wood. These tannins and carbohydrates are extraneous substances and do not form a part of the wood structure. Woods having high percentages of extraction generally manifest erratic shrinking on drying. The erratic shrinking is more apt to be true if the water-soluble materials are present in the cavities of the wood fibers and ray cells. Those extractives will increase the specific gravity of the wood but not the volume or shrinkage. If the soluble materials are high in tannins, the wood will show specific reaction toward iron. (3)

I did not get a chance to analyze for tannins as time would not permit.

The water soluble material in Lodgepole pine is rather low, showing that erratic shrinking probably will not take place during drying.

In the case of hardwoods, the water soluble materials run as high as 15% as in the case of Mesquite (*Prosopis juliflora*).⁽⁴⁾ It is of interest to note that Western Larch (*Larix occidentalis*) has a 15.30 % solubility in water.⁽⁵⁾

One Percent Caustic Soda Solubility:

This percentage solubility includes the water-soluble material and also a portion of the more soluble hemicelluloses.

In the sample is left the least soluble hemicelluloses which are thought to be of importance in making a plastic.

In the experiment I carried out, there was a loss of almost 15%. This loss would naturally increase the lignin to cellulose ratio. Such an increase is desirable in the production of plastics from wood.

To produce a plastic from wood, the ratio of lignin to cellulose has to be higher than that of ordinary wood. To make such a ratio possible one of two things can be done. Part of the hemicelluloses and cellulose can be removed by action of an acid or base; or, additional lignin can be added. To this must be added a plasticizing agent as the high temperatures necessary to plasticize lignin alone is sufficient to cause deterioration.

It has been found that the lignin to cellulose ratio very largely determines the properties of the finished material, other things being equal. A low ratio of lignin to hydrolyzed cellulose will have high strength values and low water resistance, where as the opposite effects will be obtained in a higher lignin cellulose ratio. (6)

With the knowledge of the effectiveness of the 1% NaOH solubility, this might be used as a pre-treatment of wood to make it usable as a raw material for a plastic.

"A chief point of interest in these plastic compositions is their cheapness. It is estimated that these molding powders can be made for 3 to 4 cents a pound by acid-hydrolysis process and 5 to 6 cents a pound by the aniline-hydrolysis process, a cost which should make them readily available for large-scale molding. At the present time, work upon these materials is just emerging from the experimental scale. The ultimate use of these materials

will depend entirely upon their properties when compared with plastics already in use." (6)

10% KOH:

This is a test more drastic than the 1% NaOH solubility, removing considerable more of the hemicelluloses. It indicates to some extent the quality of the constituents in the wood. This test applies more to pulps in which case a high solubility indicates a low grade of cellulose.

Ether and Alcohol-Benzene Solubilities:

These solubilities show the presence of oils, fats, resins, and waxes in the wood. Woods with high resin contents are not desirable for pulping by use of the Sulfite Process. The solubilities I received were not too high, but high enough to keep this wood from being used in a sulfite mill under the present conditions. This wood could be pulped very easily by the craft or soda process in that the resins are esterified during the cook and do not show on the paper or pulp sample. In comparison to other pulp woods of the conifers, the Lodgepole has a short fiber. The following table⁽⁷⁾ is given to show the comparison of fiber lengths.

Tree	Average length
Engelmann spruce (<i>Picea engelmannii</i>)	5.71 mm.
Sitka Spruce (<i>Picea sitchensis</i>)	2.87
Lodgepole Pine (<i>Pinus contorta</i>)	2.63
White fir (<i>Abies concolor</i>)	4.63
Lowland White fir (<i>Abies grandis</i>)	4.14
Longleaf Pine (<i>Pinus palustris</i>)	5.53
Douglas fir (<i>Pseudotsuga taxifolia</i>)	2.68
Western Hemlock (<i>Tsuga heterophylla</i>)	3.04

1 mm. equals about 1/25 of an inch

Just because Lodgepole pine has the shortest average fiber still does not shut it out of the paper and pulp industry. The fact remains that the greater part of the book papers are made from short fibered woods such as cottonwood. Also a wood that has a high alpha cellulose content is just as good in explosives, whether it has a long fiber or a short one.

The materials extracted by ether, and alcohol-benzene, are extraneous substances and do not form any part of the wood structure. They increase the specific gravity of the wood. Higher percentages of these soluble materials will tend to make a wood of given specific gravity shrink less than wood of the same specific gravity having no ether or alcohol-benzene extractives. (3)

The ether extractives of Lodgepole pine were low, and the alcohol-benzene extractives were about one half that extracted from Ponderosa pine.

Lignin:

Lignin is a substance that is in combination with the cellulose. This represents the noncarbohydrate content of the extractive-free wood substance.

The percentage I received is just about the same as that for all other pulp woods now in use. The cedars and redwood have about 3 to 8% more lignin than do the regular pulp woods. This might in part be the reason for the great durability of the cedars and redwood over the ordinary pulp-woods, in resisting decay.

The amount of lignin in lodgepole pine is sufficient to compete with all other woods in the line of lignin for plastics with the exception of the cedars and redwood.

Holocellulose:

Holocellulose is the white lignin free solid composed of the cellulose and hemicellulose fractions of the wood.⁽¹⁾ Wood substance in this form has received wide recognition in that all the carbohydrates of the wood are present. This gives the chemist an opportunity to study all the carbohydrate components of the wood. I did not have time to analyze this material and break it down into its individual constituents.

The yield I received is probably low due to the action of the chlorine on the holocellulose.

Cellulose:

Cellulose is a carbohydrate having the following formula: $(C_6H_{10}O_5)_x$. It is probably the most important constituent of the wood at the present time, being important in the paper and pulp industry as well as other industries that are as large or larger than the paper and pulp industry.

In the sample that I tested, the cellulose content was about the same as that of Long leaf pine. The spruces are 4% higher plus or minus. I think Lodgepole pine can well compete in the amount of cellulose.

10% Sodium Hydroxide Cook:

The results of the cook showed over 50% of cellulose.

This percentage shows me that Lodgepole pine really has 11. possibilities in the future. The lignin removed by such a cook could be used in some plastic, providing some efficient system can be figured out to recover the soda chemicals.

Alpha Cellulose Content:

I run an alpha cellulose determination on the above soda cook. According to my results, the 50 plus percent of cellulose was 95% alpha cellulose. This figure might be in error due to remaining lignin that could have remained in the sample of cellulose analyzed.

Conclusion

1. Lodgepole pine is a wood that can be used in the pulp and paper industry to take the place of other woods when they are depleted. Also I feel it could compete with other woods at the present time.
2. Lodgepole pine has a possible future in the line of plastics in that it has the desired qualities plus its plentiful supply that is available.
3. The solubilities tend to show this wood to be one that is not subject to erratic shrinking.
4. This wood is probably very susceptible to decay in that its lignin content is below that of cedars and redwood.

Recommendations

1. Further work should be done on the alpha cellulose content of this wood, as it really has possibilities.
2. A study should be made to definitely correlate the present laboratory findings with conditions existing in industry.

Procedure and Results obtained in the Analysis of
Lodgepole pine.

1. Moisture Determination

The purpose of this determination is to find the percentage of moisture in the wood sample, as all calculations are based on oven dry weight.

	No. 98	No. 55
Wet weight of sample and cruc.	21.4908	20.1855
Weight of empty cruc.	19.2603	18.0729
Weight of air dry sample	<u>2.2305</u> gms.	<u>2.1126</u> gms.
Wt. of oven dry sample and cruc.	21.3104	20.0147
Wt. of empty cruc.	19.2603	18.0729
Wt. of oven dry sample	<u>2.0501</u>	<u>1.9418</u>
Air dry wt. of sample	2.2305	2.1126
Oven dry wt. of sample	2.0501	1.9418
Total wt. of water lost	<u>0.1804</u>	<u>0.1708</u>

Sample No. 98 -- $\frac{0.1804 \times 100}{2.2305} = 8.08\%$ Moisture in the sample
based on air dry wt.

Sample No. 55 -- $\frac{0.1708 \times 100}{2.1126} = 8.08\%$ Moisture in the sample
based on air dry wt.

2. Ash Determination

About a 2 gram sample of prepared sample is incinerated on a tared dish.

	No. A	No. B	No. c
Wt. of sample and cruc.	11.1416	11.3318	9.8792
Wt. of a cruc.	9.5997	9.2588	8.3233
Wt. of air dry Samp.	<u>1.5419</u>	<u>2.0730</u>	<u>1.5559</u>

Sample corrected for moisture:

No. A -- $1.5419 \times 8.08 = 0.1244$	$1.5419 - 0.1244 = 1.4175$
No. B -- $2.0730 \times 8.08 = 0.1650$	$2.0730 - 0.1650 = 1.9080$
No. c -- $1.5559 \times 8.08 = 0.1257$	$1.5559 - 0.1257 = 1.4302$

	No. A	No. B	No. c
Wt. of sample and cruc.	9.6034	9.2634	8.3263
Wt. of a cruc.	9.5997	9.2588	8.3233
Wt. of oven dry samp.	0.0037	0.0046	0.0030

Percentage ash:

$$\text{No. A -- } \frac{0.0037 \times 100}{1.4175} = 0.26\% \text{ Ash}$$

$$\text{No. B -- } \frac{0.0046 \times 100}{1.9080} = 0.24\% \text{ Ash}$$

$$\text{No. c -- } \frac{0.0030 \times 100}{1.4302} = 0.21\% \text{ Ash}$$

Average percentage ----- = 0.24% Ash in sample

3. Cold Water Solubility

	No. 1	No. 2
Wt. of WB. and air dry sample	27.6667	26.8971
Wt. of Weighing Bottle	25.6661	24.9365
Wt. of air dry sample	2.0006 gms.	1.9606 gms.
Wt. of WB., Cruc., and Sample	41.3294	46.7790
Wt. of WB., and Cruc.	39.7384	45.0265
Wt. of oven dry ext. sample	1.7910 gms.	1.7525 gms.

Samples corrected for moisture:

$$\begin{array}{ll} \text{No. 1 -- } 2.0006 \times 8.08 = 0.1616 & 2.0006 - 0.1616 = 1.8390 \\ \text{No. 2 -- } 1.9606 \times 8.08 = 0.1584 & 1.9606 - 0.1584 = 1.8012 \end{array}$$

Percentage solubility:

$$\text{No. 1 -- } \frac{0.0480 \times 100}{1.8390} = 2.61\% \text{ water soluble}$$

$$\text{No. 2 -- } \frac{0.0487 \times 100}{1.8012} = 2.70\% \text{ water soluble}$$

Average percentage ----- = 2.65% water soluble material

4. Hot water solubility (1)

Approximately 2 gms. of air dry material are extracted with 100 ml. of distilled water. The sample and water are placed in a 200 ml. Erlenmeyer flask and this in turn is placed in a boiling water-bath for a period of 3 hours.

	No. 1	No. 2
Wt. of sample and WB.	27.6844	26.8907
Wt. of WB.	25.6664	24.9371
Wt. of air dry sample	<u>2.0180</u> gms.	<u>1.9536</u> gms.
Wt. of WB., cruc., and sample	41.5338	46.7624
Wt. of WB., and cruc.	39.7397	45.0285
Wt. of oven dry extracted sam.	<u>1.7941</u> gms.	<u>1.7339</u> gms.

Samples corrected for moisture:

No. 1 -- $2.0180 \times 8.08 = 0.1631$	$2.0180 - 0.1631 = 1.8549$
No. 2 -- $1.9536 \times 8.08 = 0.1579$	$1.9536 - 0.1579 = 1.7957$

Percentage solubility:

No. 1 -- $\frac{0.0608 \times 100}{1.8549} = 3.29\%$ hot water soluble
No. 2 -- $\frac{0.0618 \times 100}{1.7957} = 3.44\%$ hot water soluble

Average percentage ----- = 3.36% water soluble material

5. One Percent Caustic Soda Solubility (1)

Approximately two grams of air-dry sample are placed in a 250 ml. beaker. To this is added 100 ml. of 1% NaOH. The beaker is covered with a watch glass and then placed in a bath of boiling distilled water. The contents are occasionally stirred for a period of 1 hour.

	No. 1	No. 2
Wt. of weighing B. and sample	20.1396	21.2396
Weight of weighing bottle	18.0737	19.2648
Wt. of air dry sample	<u>2.0659</u> gms.	<u>1.9748</u> gms.
Wt. of WB., cruc., and sample	41.5291	46.4163
Wt. of WB., and cruc.	39.9054	44.8598
Wt. of dry extracted sample	<u>1.6237</u> gms.	<u>1.5565</u> gms.
Wt. of air dry sample	2.0659	1.9748
Wt. lost due to moisture	0.1669	0.1596
Oven dry wt. of sample	<u>1.8990</u> gms.	<u>1.5565</u> gms.

	No. 1	No. 2
Wt. of original sample (Oven dry)	1.8990	1.8152
Wt. of extracted sample (Oven dry)	<u>1.6237</u>	<u>1.5565</u>
Wt. soluble in NaOH (1%)	0.2753 gms.	0.2587 gms.

Percentage solubility:

$$\text{No. 1 -- } \frac{0.2753 \times 100}{1.8990} = 14.50 \% \text{ Soluble}$$

$$\text{No. 2 -- } \frac{0.2587 \times 100}{1.8152} = 14.25\% \text{ Soluble}$$

Average percentage ----- = 14.37% solubility in 1% NaOH

6. 10 Percent Potassium Hydroxide Solubility (1)

Approximately two grams of air-dry sample are placed in a 250 ml. Erlenmeyer flask provided with a reflux air condenser. To this is added 100 ml. of 7.14 % NaOH solution. The flask is then heated in a salt bath maintained at 100 degrees Centigrade, for a period of 3 hours. After cooking the excess base is neutralized with glacial acetic acid.

	No. 1	No. 2
Wt. of WB., and air dry sample	36.7714	33.0796
Wt. of WB.	<u>34.7567</u>	<u>30.9649</u>
Wt. of air dry sample	2.0147 gms.	2.1147 gms.
Wt. of WB., cruc., and sample	46.3319	41.0406
Wt. of WB., and cruc.	<u>44.8580</u>	<u>39.5103</u>
Wt. of extracted sample	1.4739 gms.	1.5303 gms.
Wt. of Air dry sample	2.0147	2.1147
Wt. lost due to moisture	<u>0.1628</u>	<u>0.1708</u>
Oven dry wt. of original sample	1.8519 gms.	1.9439 gms.
Wt. of original sample oven dry	1.8519	1.9439
Wt. of extracted sample oven dry	<u>1.4739</u>	<u>1.5303</u>
Wt. of material extracted	0.3780 gms.	0.4136 gms.

Percentage solubility:

$$\text{No. 1} \text{ -- } \frac{0.3780 \times 100}{1.8519} = 20.42\% \text{ soluble}$$

$$\text{No. 2} \text{ -- } \frac{0.4136 \times 100}{1.9439} = 21.27\% \text{ soluble}$$

$$\text{Average percentage} \text{ -----} = \underline{\underline{20.84\% \text{ soluble in } 7.14\% \text{ NaOH}}}$$

7. Ether Solubility (1)

Approximately 2 grams of air-dry sample are extracted in a soxhlet apparatus with ethyl ether for a period of 8 hours. The ether is kept boiling briskly all during the extraction.

	No. 1	No. 2
Wt. of WB., cruc., and sample	41.6050	46.9290
Wt. of WB., and cruc.	39.5069	44.8570
Wt. of air dry sample	<u>2.0981</u> gms.	<u>2.0720</u> gms.
Wt. of WB., cruc., and Sample	41.4206	46.7419
Wt. of WB., and cruc.	39.5069	44.8570
Wt. of oven dry ext. sample	<u>1.9137</u> gms.	<u>1.8849</u> gms.
Wt. of air dry sample	2.0981	2.0720
Wt. of moisture in sample	<u>0.1695</u>	<u>0.1674</u>
Wt. (oven dry) of original	<u>1.9286</u> gms.	<u>1.9046</u> gms.
Wt. of original sample oven dry	1.9286	1.9046
Wt. of extracted sample oven dry	<u>1.9137</u>	<u>1.8849</u>
Wt. lost in extraction	<u>0.0149</u> gms.	<u>0.0197</u> gms.

Percentage solubility:

$$\text{No. 1} \text{ -- } \frac{0.0149 \times 100}{1.9286} = 0.77\% \text{ soluble}$$

$$\text{No. 2} \text{ -- } \frac{0.0197 \times 100}{1.9046} = 1.04\% \text{ soluble}$$

$$\text{Average percentage} \text{ -----} = \underline{\underline{0.91\% \text{ soluble in ethyl ether}}}$$

8. Alcohol-Benzene Solubility

Approximately 2 grams of air-dry material are extracted for a period of 8 hours in a 33 percent alcohol and 67 percent benzene solution (by volume).

	No. 1	No. 2
Wt. of WB., Cruc., and sample	46.8040	41.5406
Wt. of WB., and Cruc.	44.8566	39.5082
Wt. of air dry sample	1.9474 gms.	2.0324 gms.
Wt. of WB., cruc., and sample	46.5706	41.3011
Wt. of WB., and cruc.	44.8566	39.5082
Weight of oven dry sample (extracted)	1.7140 gms.	1.7929 gms.
Wt. of original air dry sample	1.9474	2.0324
Wt. of moisture in sample	0.1573	0.1642
Wt. of original sample oven dry	1.7901 gms.	1.8682 gms.
Wt. of original sample oven dry	1.7901	1.8682
Wt. of extracted sample oven dry	1.7140	1.7929
Wt. of extracted material	0.0761 gms.	0.0753 gms.

Percentage solubility:

$$\text{No. 1} \text{ -- } \frac{0.0761 \times 100}{1.7901} = 4.25\% \text{ soluble}$$

$$\text{No. 2} \text{ -- } \frac{0.0753 \times 100}{1.8682} = 4.03\% \text{ soluble}$$

Average percentage ----- = 4.14% soluble in alcohol-benzene

9. Lignin Determination (1)

Approximately 2 grams of air-dry material are extracted in a soxhlet apparatus with 95% ethyl alcohol for a period of 4 hours. This is followed by a similar extraction with alcohol-benzene for a period of 4 hours. The sample is then extracted for 3 hours in 400 ml. of hot water in a hot water bath. The sample is then dried. The dried material is transferred from the crucible to a beaker. The sample is then divided into fine particles, and triturated with 40 ml. of 72.0% sulfuric acid.

The acid is added in small quantities, and the reaction temperature must remain under 20 degrees Centigrade. The reaction is allowed to take place for two hours. This mixture is then diluted with 1,503 ml. of distilled water. This makes the concentration of the sulfuric acid exactly 3%. This is then boiled for 3 hours.

	No. 1	No. 2
Wt. of WB., cruc., and sample	37.0916	36.2757
Wt. of WB., and cruc.	35.0684	34.1080
Wt. of air dry sample	<u>2.0232</u> gms.	<u>2.1677</u> gms.
Wt. of WB., cruc., and sample	36.8944	36.0200
Wt. of WB., and cruc.	35.0689	34.1080
Wt. of extracted sample	<u>1.8255</u> gms.	<u>1.9120</u> gms.
Wt. of original air dry sample	2.0232	2.1677
Wt. of moisture in sample	0.1635	0.1751
Wt. of original sample oven dry	<u>1.8597</u> gms.	<u>1.9926</u> gms.
Wt. of WB., cruc., and ext. sample	36.8944	36.0200
Wt. of Wb., cruc., and remaining part of sample	35.1203	34.1715
Wt. of ext. sample used	<u>1.7741</u> gms.	<u>1.8485</u> gms.
Wt. of WB., cruc., and lignin	35.5402	34.6143
Wt. of WB., and cruc.	35.0677	34.1080
Wt. of oven dry lignin	<u>0.4725</u> gms.	<u>0.5063</u> gms.

To get every figure on the same basis so I can figure percent, I used the ratio.

$$\begin{aligned} \text{No. 1} &-- 1.8579 : 1.8255 :: x : 1.7741 \\ x &= 1.8073 \text{ gms.} \end{aligned}$$

$$\frac{0.4725 \times 100}{1.8073} = 26.14 \% \text{ lignin}$$

$$\begin{aligned} \text{No. 2} &-- 1.9926 : 1.9120 :: x : 1.8485 \\ x &= 1.9264 \text{ gms.} \end{aligned}$$

$$\frac{0.5063 \times 100}{1.9264} = 26.28\% \text{ lignin}$$

Average percentage ----- = 26.21% lignin in sample

10. Holocellulose Determination (1)

Holocellulose, (2) is a white lignin-free solid of the cellulose and the hemicellulose fractions of wood.

Approximately 2 grams of air-dry sample is extracted in a soxhlet apparatus for 8 hours. The first 4 hours the sample is extracted with ethyl alcohol. The next 4 hours the sample is extracted with alcohol-benzene. This extraction is followed by extracting the sample in 400 ml. of hot distilled water for a period of 3 hours. The sample is then chlorinated until the sample is white, and the lignin solvent no longer appears colored. The solvent used was alcohol-monoethanolamine (95% alcohol containing 3% of monoethanolamine).

	No. 1	No. 2
Wt. of WB., cruc., and sample	36.1736	37.0866
Wt. of WB., and cruc.	34.1110	35.0709
Wt. of air dry sample	<u>2.0626</u> gms.	<u>2.0157</u> gms.
Wt. of WB., cruc., and sample	35.8927	36.8272
Wt. of WB., and cruc.	34.1110	35.0709
Wt. of extracted sample	<u>1.7817</u> gms.	<u>1.7563</u> gms.
Wt. of original air dry sample	2.0626	2.0157
Wt. of moisture in sample	0.1667	0.1629
Oven dry wt. of original sample	<u>1.8959</u> gms.	<u>1.8528</u> gms.
Wt. of cruc., WB., and ext. sample	35.8927	36.8272
Wt. of cruc., WB., and remaining sample in cruc.	34.1578	35.0709
Wt. of sample used	<u>1.7349</u> gms.	<u>1.7563</u> gms.
Wt. of cruc., WB., and Holocellulose	25.5020	27.7560
Wt. of cruc., and WB.	24.3675	26.6384
Wt. of Holocellulose	<u>1.1345</u> gms.	<u>1.1176</u> gms.

To get every figure on the same basis to figure percent,
I used the ratio.

$$\text{No. 1 -- } 1.8959 : 1.7817 :: x : 1.7349$$

$$x = 1.8456 \text{ gms.}$$

$$\frac{1.1345 \times 100}{1.8456} = 61.50\% \text{ Holocellulose}$$

$$\text{No. 2 -- } \frac{1.1176 \times 100}{1.8528} = 60.32\% \text{ Holocellulose}$$

$$\text{Average percentage -----} = \underline{\underline{60.96\% \text{ Holocellulose in sample}}}$$

11. Cellulose Determination (1)

The wood sample extracted with alcohol-benzene is the one used in the cellulose determination. The extracted sample is transferred to a 35 ml. Jena glass crucible. In this crucible the sample is chlorinated. The chlorinated lignin is removed by 2 percent sodium sulfite. The chlorination and removal of chlorinated lignin continues until the sample is white, and the lignin solvent is no longer colored when added to the sample being chlorinated. The sample is then washed with water and then dried.

	No. 1	No. 2
Wt. of original sample oven dry	1.7901	1.8682
Wt. of extracted sample oven dry	1.7140	1.7929
Wt. of WB., cruc., and sample (extracted)	46.5706	41.3011
Wt. of WB., cruc. and remains	44.8884	39.5420
Wt. of sample used	<u>1.6822</u> gms.	<u>1.7591</u> gms.
Wt. of Jena cruc., and cellulose	36.0753	35.1286
Wt. of Jena cruc.	35.0698	34.1079
Wt. of oven dry cellulose	<u>1.0055</u> gms.	<u>1.0207</u> gms.

To get every figure on the same basis to figure percent,

I used the ratio.

No. 1 -- $1.7901 : 1.7140 :: x : 1.6822$
 $x = 1.7575$ gms.

$$\frac{1.0055 \times 100}{1.7575} = 57.21\% \text{ cellulose}$$

No. 2 -- $1.8682 : 1.7929 :: x : 1.7591$
 $x = 1.8329$ gms.

$$\frac{1.0207 \times 100}{1.8329} = 55.74 \% \text{ cellulose}$$

Average percentage -- = 56.47 % cellulose in sample

12. 10 Percent Sodium Hydroxide Cook

This cook was made under the supervision of Dr. Leo Friedman. The digester used has a capacity of 4 liters.

403.09 gms. of oven dry Lodgepole pine sawdust was digested in 2,500 ccs. of 10% NaOH for a period of 1 hour @ 135 degrees Centigrade, after $1\frac{1}{2}$ hours was taken to bring the sample up to this temperature. There was an excess of NaOH to insure a good cook.

Wt. of original oven dry sample	403.09
Wt. of the cellulose	207.66
Wt. of wood lost in cook	<u>195.43</u> gms.

Percentage of wood lost in cook:

$$\frac{195.43 \times 100}{403.09} = 48.48\% \text{ of wood lost in cook}$$

Percentage of Cellulose in wood:

$$\frac{207.66 \times 100}{403.09} = 51.52\% \text{ cellulose in wood.}$$

According to Dr. Leo Friedman, this sample of wood has been under cooked, as the cellulose content is too high. The high figure may be due to remaining lignin, or to lack of degradation of cellulose by sodium hydroxide.

Bibliography

- (1) Bray, Mark W., "Methods Used at the Forest Products Laboratory for the Analysis of Pulps and Pulpwoods", Revised, September 1939.
- (4) Ritter, George J. and Fleck, L. C., J. Ind. Eng. Chem., November 1922.
- (3) Ritter, George J., Letter to Dr. Leo Friedman, Mar. 11, 1941.
- (5) Schorger, A. W., J. Ind. Eng. Chem., 9 (1917) 556
- (2) Schorger, A. W., "Chemistry of Cellulose and Wood", First Edition, (1926) 51.
- (6) Sherrard, E. C., Beglinger, Edward, and Hoff, J. P., Bull. No. R1209, (June 1939) 5.
- (7) Sutermeister, Edwin, "Chemistry of Paper and Pulp Making", Second Edition, (1929) 57.