

A Study of Chemical Constitution of Lignin of Leucaena Leucocephala

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Abstract— The study was carried out on *Leucaena leucocephala* of different age groups to learn more about its chemical composition by isolating lignin via solvent isolation technique. The effect of plant's age on its chemical constitute were also investigated. As a cheap and fast-grown resource with superior physical and mechanical properties compared to most wood species, *Leucaena leucocephala* offers great potential as an alternative to softwood that is usually used in pulp and paper industry.

Key words: Lignin, Solvent Isolation, Softwood

I. INTRODUCTION

The pulp and paper sector in India represents one of the energy demanding sectors. Not only this, it also is responsible for causing tarnishing effects on the environment. However, increases in productivity through the implementation of better, efficient and cleaner technologies may help in bringing about economic, environmental, and social development goals. For this reason the Indian pulp and paper industry is in continuous search of possible new raw material to meet the ends. In this work we have tried to analyze the potentials of *Leucaena leucocephala* to replace the traditional raw material used in pulp and paper industry.

II. MATERIAL & METHOD

A. Sample Collection:

The samples of three different ages (2yrs, 3yrs, and 5 yrs) of *Leucaena leucocephala* were collected from different places near Bhopal and Vidisha district of Madhya Pradesh, India. Collection of the samples was done in the month of April-May 2012. The wood was finely grinded in the ball mill and screened through the 30-60 mesh.

B. Proximate Chemical Analysis:

Proximate analysis of the wood sample were done as per TAPPI standard T-207, 0S-75[1], T-204, 0S-76[2], T- 5m-59 [3],T-212 0S-58[4], T-223 0S-71 84[5], T-22-05-74[6], 0S-76, T-15[7-10].

| S.No | Particulars of Proximate Chemical Analysis | Leucaena Leucocephala | | | Average |
|------|--|-----------------------|------------|-----------|---------|
| | | Two Year | Three Year | Five Year | |
| 1 | Ash Content | 2.31 | 2.74 | 3.45 | 2.833 |
| 2 | Pentosan Content | 13.93 | 14.88 | 17.57 | 15.46 |
| 3 | Cold Water Solubility | 1.05 | 1.98 | 3.04 | 2.02 |
| 4 | α Cellulose Content | 39.9 | 46.8 | 49.9 | 45.53 |
| 5 | Hot Water Solubility | 2.50 | 3.40 | 5.94 | 3.94 |
| 6 | Ether Solubility | 5.94 | 7.35 | 8.25 | 7.18 |

| | | | | | |
|----|----------------------------|-------|-------|-------|-------|
| 7 | Alcohol Benzene Solubility | 4.46 | 5.05 | 5.19 | 4.9 |
| 8 | 1% NaOH Solubility | 18.56 | 20.55 | 23.19 | 20.76 |
| 9 | Methoxyl Content | 4.65 | 5.08 | 5.60 | 4.96 |
| 10 | Lignin Content | 19.53 | 21.50 | 23.30 | 15.54 |
| 11 | Acetyl Content | 2.30 | 2.7 | 3.4 | 2.8 |
| 12 | Holo Cellulose Content | 67.61 | 75.07 | 78.07 | 73.58 |

Table 1: Proximate Chemical Analysis *Leucaena Leucocephala*

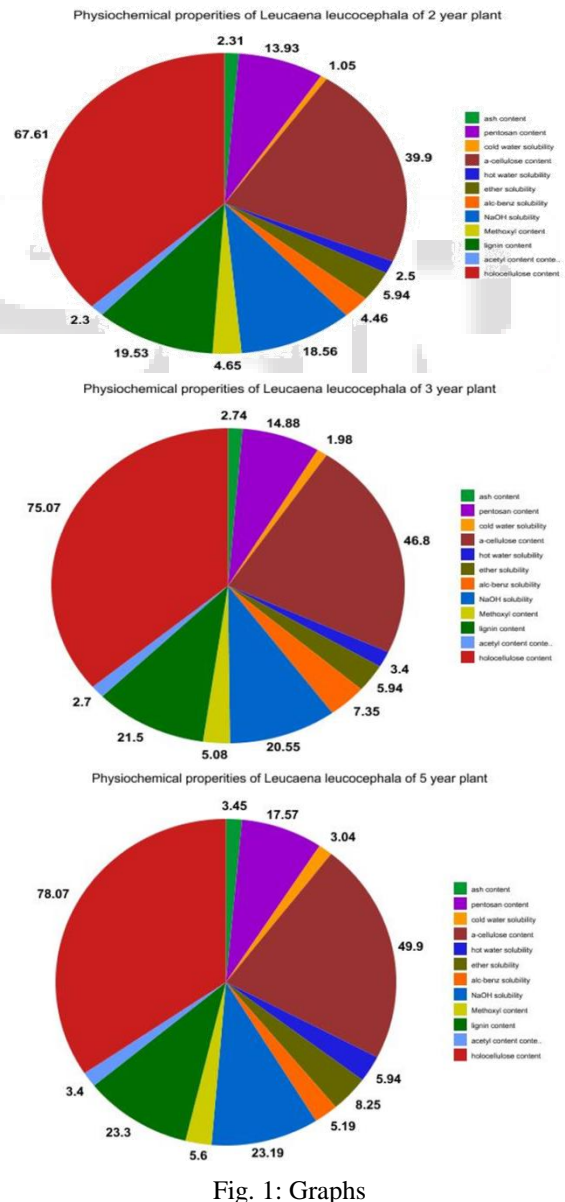
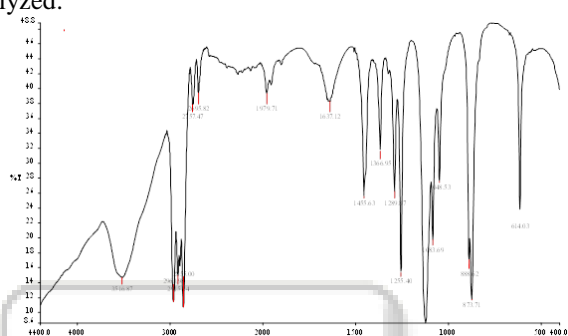


Fig. 1: Graphs

III. PREPARATION AND EXTRACTION OF MILLED WOOD LIGNIN

The isolation of lignin was done by the method suggested by Bland [11-12], Nascimento[13] and Morais[14]. However some changes were made as per the samples.. A 100 mesh ground wood was extracted with benzene-ethanol (2:1) and water. It was then extracted by 0.1 M cold sodium hydroxide solution in order to remove residual polyphenols. This resulting extract-free meal was then treated with 2% acetic acid until the pH was 4. It was then dried at 80C for 10 hours. It was followed by successive extraction by chloroform and then with dioxane:water (9:1) for couple of days. The dioxane was evaporated and the relatively wet meal was treated with 2% NaOH and filtered. The filtrate contains lignin which was then precipitated using 2% acetic acid. The precipitate was then filtered and dried and analyzed.



| Carbon | Hydrogen | Oxygen | Methoxy OCH ₃ | C ₉ Formula | Formula Weigh |
|--------|----------|--------|--------------------------|--|---------------|
| 56.4 | 5.6 | 38.0 | 22.5 | C ₉ H _{7.75} O _{2.32} (OCH ₃) _{1.64} | 203.71 |

Table 2: Element Composition and C₉ Formula Leucaena Leucocephala

| S. No. | Species | OCH ₃ | | CO per | Total OH per | Phenolic OH |
|--------|-----------------------|------------------|-------------------------|---------------------|---------------------|-------------------------|
| | | % | per C ₉ unit | C ₉ unit | C ₉ unit | per C ₉ unit |
| 1. | Leucaena Leucocephala | 22.5% | 1.64 | 0.21 | 13.76 | 0.34 |

Table 3: Reactive Groups of Mild Wood Lignins Leucaena Leucocephala

In order to predict the use of *Leucaena leucocephala* as a alternative raw material for pulping, it is desirable to compare its physiochemical properties with other raw materials that are being frequently used in pulp and paper industry.

| S. No | Particulars of Chemical Analysis | Hardwood E. Grandis | Softwood Piums Spp. (Pine) | Leucaena Leucocephala (Subabul) |
|-------|----------------------------------|---------------------|----------------------------|---------------------------------|
| 1. | Ash Content % | 02.87 | 04.45 | 02.83 |
| 2. | Cold Water Solubility% | 02.19 | 02.98 | 02.02 |
| 3. | Hot Water Solubility% | 04.59 | 07.53 | 03.94 |
| 4. | Ether Solubility% | 14.00 | 1 to 5.5% | 07.18 |
| 5. | Alcohol Benzene Solubility% | 03.11 | 06.45 | 04.90 |
| 6. | 1% NaOH Solubility% | 17.90 | 17.88 | 20.76 |
| 7. | Pentosan Content% | 11.60 | 12.03 to 14.03 | 15.46 |
| 8. | Klason Lignin Content% | 29.60 | 26.71 | 21.44 |
| 9. | Holo Cellulose Content% | 59.80 | 71.61 | 73.58 |
| 10. | α Cellulose Content% | 44.30 | 48.94 | 45.50 |
| 11. | Acetyl Content% | 00.79 | 01.15 | 02.80 |
| 12. | Methoxly Content% | 07.20 | 05.17 | 05.11 |

Table 4: Comparisons

VII. CONCLUSION

- 1) The ash content indicates the inorganic matter present in the wood. The lesser the ash content the better is the pulp. The mean ash content of *Leucaena leucocephala*

Fig 4: Ftir of Lignin of Leucaena leucocephala

IV. CHEMICAL ANALYSIS:

A series of experiments were carried out to study the chemical constitution of lignin of samples of LEUCAENA LEUCOCEPHALA were analyses for the following parameters. It includes elemental chemical analysis and determination of carbonyl groups, methoxyl group, total hydroxyl groups, phenolic hydroxyl groups and furfural content as per standard TAPPI method.

V. RESULTS AND DISCUSSION:

Molecular formula of lignin of *Leucaena leucocephala*: The polymeric structure of lignin as described by Freudenberg was based on various phenylpropanyl alcohol monomeric units (also called lignols or phenylpropanoid units) attached through both ether and carbon-carbon bonds [15]. The different types of phenylpropanoid units in lignin are guaiacylpropane, syringylpropane, and p-hydroxylpropane. To elucidate the type of phenylpropane unit in the lignin of *Leucaena leucocephala* it is customary to find out its molecular formula, which is referred as C₉ formula i.e number of Hydrogen, Oxygen and Methoxy group per 9 carbon. The C₉ formula was found to be C₉H_{7.75}O_{2.32}(OCH₃)_{1.64} suggesting, phenylpropane units are mainly composed of syringyl units and some guaiacyl units with no p-hydroxyl units which is in-fact a common feature of most hardwoods lignins.

VI. COMPARISON OF PROXIMATE CHEMICAL ANALYSIS OF HARDWOODS & SOFTWOOD WITH STUDY SAMPLE (% ON DRY BASIS)

is 2.83% which is in accordance with the results of Dutt & Tyagi[16]who did the work on *Eucalyptus* species. Similarly Lal[17] et al reported ash content in pine to be 4.45 %.

- 2) Cold water soluble content of *leucaena leucocephala* was 2.02% while hot water soluble content was found out to be 3.94%. These results synchronize with the results of Sharma[18] et al their study being performed on *Eucalyptus*.
- 3) Alcohol-benzene extractable substances can precipitate and retard drainage on a paper machine due to blocking of openings in a Fourdrinier wire and leaving stains in the resulting paper sheets. Alcohol-benzene content was observed to be (4.9%) in our study material. For various pinus species the same parameter ranges from 4.68% to 6.45% Sari[19] et al
- 4) Ether soluble content which informs about extraneous components present in the wood sample was found to be 7.18% in *Leucaena leucocephala*. It was clearly seen that the mean values so obtained of ether solubility is less than that of pine *Gonulasa & Ucar* [20].
- 5) Alkali solution extracts out low-molecular-weight carbohydrates consisting mainly of hemicellulose and degraded cellulose in wood and pulp, Pettersen.[21]. It is useful in indicating how quickly the wood can decompose due to biological and physiochemical factors Procter and Chow[22]. Hence as the wood decays or the percentage of the alkali-soluble material increases, indicating length of storage of a raw material in a wood yard. The experimental studies showed that the sample has alkali solubility of 20.76%, while that of *eucalyptus* ranged from 12.2% upto 19.9% depending upon the section of tree analyzed Miranda[23] et al while that of pine ranges from 20.01 to 27.46% *Gonultas and Ucar*[20].
- 6) Pentosan content in pulp indicates the retention or loss of hemicellulose in general during pulping and bleaching processes, and since hemicellulose contributes to the strength of paper pulps, high pentosan content is desirable.
In a similar experiment Haddad [24] et al found out that pentosan content of pine 12.3 to 15.53% while that of *eucalyptus* Since, pentosan content (15.46) falls well within the range of observed values and hence pulp derived from it, can be used for commercial purpose.
- 7) Pulp properties such as color, bleachability are associated with the lignin content. The lignin content of *Leucaena leucocephala* is about (23.30%), it shows that *Leucaena leucocephala* is a good raw material for the pulp and paper industry.
- 8) Holocellulose is the lignin-free total carbohydrate fraction of extractive free wood and comprises both the true cellulose and hemicelluloses of wood. The more the holocellulose the better is the wood sample. Dutt and Tyagi [16] found that holocellulose content in *eucalyptus* was 55.6-67.8% while that of pine ranges between 67.60% to 72.30% *Khattak & Mahmood* [25]. *Leucaena holocellulose* observed in this research was 73.58% justifying the use of *leucaena* in pulp and paper industry. α - cellulose content has been widely used to evaluate pulps for various purposes, such as aging characteristics and response to refining operations.
- 9) The α - cellulose % of *Leucaena leucocephala* is about 45.5 which enables it to quantify as a good raw material to make high strength papers. Acetyl group are combined with polysaccharide portion of non woody plant while methoxyl group are present in the lignin and they characterize lignin. Acetyl content of *Leucaena* was 02.80% while methoxyl content was found to be 05.11%.
The above study revealed that with the age of *Leucaena leucocephala* its α -cellulose, holocellulose and pentosan content was found to increase, which in general is the primary requirement of pulp and paper industry. However, along with them, the lignin content was also found to amplify. Hence, in order to derive pulping conditions that result in better yield production of cellulose from *Leucaena leucocephala* a complete kinetic study of its delignification process is very necessary.

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