## Study the Effect of Age on Wood Chemical Compounds of *Eucalyptus Camaldulensis* and *Populus Nigra* Growing in Erbil Governorates

### Sherzad Omar Osman<sup>1</sup>, Heman Abdulkhaliq Ahmed<sup>2</sup>, Arshad Abdulkhalq Yaseen<sup>3</sup>

<sup>1,2,3</sup> Department of Forestry, College of Agriculture, University of Salahaddin, Erbil, Kurdistan Region, Iraq

*Abstract:* In this investigation, the effect of age of Eucalyptus camaldulensis and Populus nigra trees (at two different ages i.e. 4 years old and 8 year old) on chemical compound, for using in pulp and paper industry was studied. Chemical compounds for each age, have been repeated two times and the amount of holocelluloses, hemicelluloses, alpha-cellulose, lignin, and total extractives for 4 years old in E. camaldulensis have been obtained relatively 68.96, 29.80 39.06, 18.89 and 8.69 respectively. Regarding 8 year- old, these results were obtained 70.76, 28.63, 42.31, 21,15 and 9.41 respectively. In terms of 4 year-old of P. nigra trees these result were obtained 72.03, 26.81, 44.88, 21.59 and 6.00 respectively for all chemical compounds. While for 8 year- old the results were 74.37, 25.6, 48.2, 20.72 and 8.35 respectively. These compounds in several ages have been compared with each other. The results showed that the 8 year-old of P. nigra trees are the best one between them because they contain higher amount of cellulose and lower amount of extractive and lignin which be more suitable choice for pulp industry.

Keywords: cellulose; extractives; holocellulose; lignin; Populus and eucalyptus.

#### I. INTRODUCTION

P. nigra which known as Poplar, it is a fast-growing species, can satisfy the growing needs for solid woods; furthermore, it offers a quality raw material for wood-composite manufacturing factories because the end product will benefit from higher compression ratio. Poplar belongs to Salicaceae family (1).

E. camaldulensis and P. nigra are important species in Kurdistan because they can grow very well in various parts of the country, and the wood biomass production are especially excellent for the pulp and paper industries that use E. camaldulensis and P. nigra wood as raw material. The pulp and paper industries look for wood that has a high pulp yield after the Kraft, or soda process. In addition, for raw wood material the factories need low content in lignin and high cellulose.

The main objective of this study is to investigate the feasibility of predicting pulp yield property and chemical composition of E. camaldulensis and P. nigra. The chemical composition values contained both main components such as holocellulose,  $\alpha$ -cellulose, and lignin, one percentage of hemicellulose and minor components such as extractive.

#### II. MATERIAL AND METHOD

#### A. Statistical Methods:

For obtaining data from chemical analysis, factorial statistical design has been used and each sample in any age has been tested three times. Then for analysis the effect of two ages on chemical compounds, one way variance analysis test has been used. For comparing the average of several samples with each other the Duncan statistical test has been used.

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#### B. Sample Preparation of E. camaldulensis and P. nigra:

Seventy samples of E. camaldulensis and P. nigra wood chip were obtained from they were inspected for defects of different particle size. Therefore, the wood chips were ground with Wiley mill. After that, they were screened through the size 40-60 mesh. All wood powder that remains on 60 mesh of screen, its moisture was controlled by drying in oven at 105°C for two days, and then it was cooled in desiccators. After that it was kept in a plastic bag to store the samples.

#### C. Sample Preparation for Chemical Analysis:

#### 1. Analysis of Wood Extractive:

About 10 g of wood powder was extract with Ethanol-Toluene in Soxhlet extraction apparatus after that, the wood powder was transferred to beaker. The excess solvent was removed by washing with 95% ethanol for 4 hour. Then, the excess solvent was removed with suction and the sample was further washing with hot water for 1 hour. The wood powder was filtered and it dried at room temperature after that it was stored in air tight container for further chemical analysis. Then the moisture content in wood was calculated.

The wood powder, at least 4 g were required for determination in duplicate wood powder was weighted into the extraction thimble in the Soxhlet apparatus. It was extracted with 150 ml of solvent that contain the mixture of ethanol (1 volume) and toluene (2 volumes). The extraction apparatus was heated to boiling temperature of solvent. The cycle rate of the solvents was used at least 6 times per hour over 4-5 hour period. Then the flask was removed from the apparatus, and the solvent was evaporated until dryness. The extractive was dried in oven for one hour at  $100\pm5^{\circ}$ C. The residual was weight and calculated by dividing with the weight of wood powder, and multiplied by 100.

#### 2. Analysis of Holocellulose:

About 5 g of the extractive free wood powder was weighed and put into Erlenmeyer flask. The 160 ml of distilled water, 0.5 ml of glacial acetic acid and  $1.5\pm0.1$  g of sodium chlorite were added, respectively. Later on flask was placed on a steam bath, of which the temperature was adjusted about 70-80°C. The flask was heated for 1 hour at the reaction temperature, and the contents were mixed by occasional swirling. Then, without cooling, 0.5 ml of glacial acetic acid was added followed by 1.5 g of sodium chlorite. The heating was continuous for an additional hour. At the end of second and third hours, the additions of acetic acid and sodium chlorite were repeated. At the end of the fourth hour, the flask was placed in an ice bath until the contents have cooled below  $10^{\circ}$ C. The holocellulose was filtered on glass crucible pore No.1 and washed with cool water to remove the color and odor of chlorine dioxide, after that it was dried in the oven at  $100\pm5^{\circ}$ C for 24 hour. Then it was moved to a desiccator, and it was let sit 1 hour and weigh. The holocellulose content were calculated by dividing with weight of wood powder and multiplied by 100 (2 and 3).

#### **3.** Analysis of α-Cellulose:

Calculate the percentage of  $\alpha$  -cellulose on the basis of the oven-dried holocellulose sample, as follows:

 $\alpha$  -cellulose, percent = (W2 /W1) × 100

Where W2 = weight of the oven-dried  $\alpha$  -cellulose residue.

W1 = weight of the original oven-dried holocellulose sample (3).

About  $1.5\pm0.1$  g of holocellulose was put into beaker, and then 75.0 ml of 17.5% NaOH was added. The temperature was controlled at 25°C. The Holocellulose was stirred with the stirrer until it was completely dispersed. Then the stirrer was rinsed for removing the adhered Holocellulose with 25 ml of 17.5% NaOH reagents again. After that, the suspension was stirred with a glass rod and maintained temperature in a water bath at 25°C. After30 min from the first addition of the NaOH reagent, 100 ml of distilled water at 25°C was added to the suspension and stirred thoroughly with a glass rod. The beaker was stood in a water bath for 30 min so that the total extraction time was  $60\pm5$  min. The residual was filtered with a glass filtering crucible pore no.3, after that washing with distilled water until the washing solution became neutral. 40 ml of 10% acetic acid into the crucible was added and it was holed for 5 min, from the time the suction was released; place the cellulose overnight in the oven  $100\pm5^{\circ}$ C. Calculated the  $\alpha$ -cellulose content by dividing with the weight of holocellulose and multiplied by 100 (2 and 3).

#### 4. Analysis of Lignin Content:

Klason lignin gives a quantitative measure of the acid insoluble lignin and is not suitable for the study of lignin structures and some other lignin such as cellulolytic enzyme lignin, or Björkman (milled wood lignin) should be prepared (4). This procedure is a modified version of ASTM D-1166-84. The lignin isolated using this procedure is also called sulfuric acid lignin (3).

#### **III. RESULT AND DISCUSSION**

The average properties of the tested samples are listed in Table 1, including Duncan's mean letters, associated with the levels of significance.

TABLE 1. THE AVERAGE PROPERTIES OF THE TESTED SAMPLES WITH DUNCAN'S MEANS

Factor	Extractives%	Lignin,%	Holocellulose, %	α-cellulose, %
Species				
Eucalyptus camaldulensis	9.05 <sup>a</sup>	20.02 <sup>b</sup>	69.86 <sup>b</sup>	40.69 <sup>b</sup>
Populuse nigra	7.17 <sup>b</sup>	21.16 <sup>a</sup>	73.20 <sup>a</sup>	46.54 <sup>a</sup>
Interaction- Age (year)				
Eucalyptus camaldulensis 4year	8.69 <sup>b</sup>	18.89 <sup>c</sup>	68.96 <sup>d</sup>	39.06 <sup>d</sup>
Eucalyptus camaldulensis 8year	9.41 <sup>a</sup>	21.15 <sup>a</sup>	70.76 <sup>c</sup>	42.31 <sup>c</sup>
Populuse nigra 4year	6.00 <sup>c</sup>	21.59 <sup>a</sup>	72.03 <sup>b</sup>	44.88 <sup>b</sup>
Populuse nigra 8year	8.35 <sup>b</sup>	20.72 <sup>b</sup>	74.37 <sup>a</sup>	48.20 <sup>a</sup>

Wood is essentially composed of cellulose, hemicelluloses, lignin, and extractives. In different wood species, however, their relative composition varies greatly, and also the chemical composition of wood varies quantitatively among tree species (5).



Figure 1. The effect of different tree species on the chemical composition

Hemicelluloses play a crucial role in the bonding capacity of fibers, i.e. the ability to form inters fiber bonds, which gives the paper fiber network its strength (Knowpulp). In contrast to cellulose that is crystalline, strong, and resistant to hydrolysis, hemicellulose has a random, amorphous structure with little strength.

It is easily hydrolyzed by dilute acid or base, but nature provides an arsenal of Hemicellulose enzymes for its hydrolysis (6).



Figure 2. The Effect of Different Age of E. camaldulensis and P. nigra trees on wood chemical composition.

By increasing tree age, cell walls of wooden fibers get thicker and percentage of cellulose increase and higher percentage of cellulose will have a good effect on produced pulp [6 -7].

The DP of cellulose increased constantly with increasing tree age. There is evidence that the DP of cellulose of wood is reduced during aging of a living tree; that is, the DP is highest in cells adjacent to the cambium and decreases toward the pith (8).



#### Figure 3. The Effect of Different Age of E. camaldulensis on wood chemical composition

Chemical compounds percentage of each eucalyptus and populuse Results of statistical analysis and comparing the averages in Duncan method show that by increasing tree age, the amount of holocellulose, cellulose, extractives and lignin increase but the amount of hemicelluloses decrease (Table 1). By increasing tree age, volume of hearth wood increase, percentage of parenchyma death cells increase, these cells at death time ooze organic materials (extractives), the amount of these materials will increase in wood( 6 and 7) In Table 2, increasing tree age leads to increasing the amount of wood lignin. Usually by increasing the ageing mature wood, lignin percentage decrease but in this research by increasing age from 4 to 8 years the amount of lignin has increased. This increasing can have several reasons like mature wood and Lignin percentage in cell walls of young wood is more than mature wood that naturally by increasing the age and increasing mature wood volume, lignin percentage decrease (9- 10-11). By increasing tree age, percentage of cellulose

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increase and higher percentage of cellulose will have a good effect on produced pulp (7). The reason for the pulp yield differences could be due to the alpha-cellulose content of the wood. It has been demonstrated that Kraft pulp yield is directly proportional to the alpha-cellulose of the wood (12).



Figure 4. The Effect of Different Age of P. nigra on wood chemical composition.

Young wood in comparison with mature wood has larger amount of hemicelluloses (13). In hardwood trees by increasing the age, percentage of hemicelluloses decrease and percentage of cellulose increase (11-14). However making decision about the best year of young Populus and Eucalyptus trees harvesting, in this study from the point of chemical compounds, the best year of harvesting is introducing and the last conclusion will be in later researches. After the last research, results of chemical compounds analysis, of Populus trees in 8 year-old because of the point of higher and lower percentage of lignin and Hemicellulose, and a little difference in amount of extractive and in eucalyptus trees at 8 year-old age because of having better properties that is because of the point of higher cellulose that has a great effect on producing pulp and a little difference in amount of lignin with lower ages, is offered as more a appropriate wood for using in pulping industry.

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