

# Production of NSSC Cellulosic Pulp Fibers from Eucalyptus Cameldulensis



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## Abstract

This study has aimed to investigate production of neutral sulfite semi-chemical (NSSC) pulping from eucalyptus camaldulensis in Zabol, Sistan & Balouchestan, Iran. Chemical composition including cellulose, lignin, extractive and ash were measured (48.33%), (28.49%), (5.94%) & (0.65%) respectively, while dimension of fibers including length (0.935mm), diameter (19.13 $\mu$ m), lumen cavity (8.89 $\mu$ m) & cell wall thickness (5.12 $\mu$ m). Cooking conditions were consist of three levels of chemical charge (10, 14 & 18% on O.D weight), constant time of 90min, constant temperature of 170 °C & liquor to wood ratio of 7:1. The pulps were refined to 400 $\pm$ 25mL, CSF & 127gm-2 hand sheets were made. Finally, strength properties of the pulps were compared and evaluated with that of Mazandaran Wood and Paper Industries (MWPI), Iran. The results showed that pulp yield decreases with increase of chemical charge. Analysis of the mechanical properties indicated that the paper from NSSC of eucalyptus at various cooking levels has preference of NSSC pulp of hardwoods produced in MWPI for all strength indices.

**Keywords:** Eucalyptus camaldulensis; Chemical charge; Pulp; NSSC

## Introduction

The use of fast-growing species, such as tagasaste, leucaena, paulownia [1-3]. The fast-growing commercial plantations of Eucalyptus species have nowadays an important role in the fulfillment of the worldwide increasing demand for pulpwood [4]. By 2008, the total area of Eucalyptus plantations, mainly distributed in about one dozen countries spread worldwide, [5] exceeded 19 x 106ha [1]. In fact, the Eucalyptus species are the most important fiber sources for pulp and paper production in South-West Europe (Portugal and Spain), South America (Brazil and Chile), South Africa, Japan, Iran and other countries [6].

Nowadays researchers are engaged in applying those pulping processes which needs less chemical for the production process due to environmental concerns. In this regard, NSSC process, as one of the production processes which observes environmental issues, requires further investigations to optimize the production method along with economic and ecological objectives for reducing the chemical charge and application of the proper species [7]. Neutral Sulfite Semi-Chemical process briefly called NSSC is mainly used to produce high-yield pulps from

hardwoods. Sodium sulfite cooking liquor is used in this process to neutralize organic acids released from lignocelluloses materials during cooking with small amounts of sodium carbonate, sodium hydroxide or sodium bicarbonate [8]. These materials are utilized for initial treatment while separation of the fibers is finally accomplished by mechanical processes. On the other hand, limitations in utilization of wood from forests in northern part of Iran, entails looking for alternative sources from various parts of the country. Eucalyptus, especially camaldulensis are of quick growing species which is expected to be consistent with ecological conditions of Iran. Eucalyptus has special value and importance considering the continental conditions of Iran with large arid areas [9]. Based on global research and experiences in making pulping from eucalyptus wood species [9-14] the purpose of this research was to investigate chemical composition and biometrical properties of the fibers from eucalyptus camaldulensis, production of NSSC pulp from it and finally, comparison of the mechanical properties of the handsheet papers with those samples made in MWPI.

**Experimental**

**Materials**

Three eucalyptus camaldulensis trees from agriculture researches station of Zabol were selected randomly and cut down. Logs of Breast height were cut and prepared from each tree. This region has hot and dry climate such that its yearly maximum temperature reaches 45 °C. Meanwhile, annual precipitation of this region is 41mm and so-called 120-days wind of Sistan is of the dominant phenomena in this region.

**Measurement of chemical composition**

Measuring percentage of cellulose was done through nitric acid method [15], while percentage of lignin, extractives soluble in acetone, and ash content were measured according to TAPPI test methods T222-om-98, T204-om-97 & T211-om-93, respectively.

**Measurement of fibers dimensions**

Franklin’s technique (1954) was used to prepare the samples. Dimensions of the fibers including length and diameter, Lumen width and thickness of cell wall were measured by LeicaQ5000MC Image analyzer apparatus and biometric ratios and coefficient being calculated from the following equations:

$$\text{Slenderness Ratio} = \frac{L}{d} \dots\dots\dots(1) \quad [16]$$

$$\text{Flexibility coefficient} = \left(\frac{c}{d}\right) \times 100 \dots\dots\dots(2) \quad [17]$$

$$\text{Runkel Ratio (tear strength of fiber)} = \left(\frac{2p}{c}\right) \times 100 \dots\dots\dots(3) \quad [18]$$

Where, L=length of fibers, d=diameter of fibers, c= Lumen width of fiber, p=thickness of cell wall.

**Experimental Pulping**

Three amounts of chemicals of sodium sulfite (Na<sub>2</sub>SO<sub>3</sub>) and sodium bicarbonate (Na<sub>2</sub>CO<sub>3</sub>) (10%, 14%, & 18%, on the basis of oven dry mass of eucalyptus) and constant pulping time of 90min were used. For each combination of variables, 3 replica pulp samples were made. A pulping temperature of 170 °C was kept constant. The cooking liquor to eucalyptus (L/W) was at a 7-1 ratio. The cooking trials were performed using an experimental rotating digester (HATTO), with 500 grams of eucalyptus in each trial. Pulping time was measured after reaching 170°C. The time to reach the cooking temperature was adjusted at 30 minutes. At the end of each cooking, the content of the cooking cylinder was

discharged on a 200-mesh screen, and the cooked material was washed using hot water. The remaining liquor was separated by hand-pressing the cooked material. Digester yield was measured by weighing the washed material on top of the screen without defibration. The cooked material was defibrated using a 25cm laboratory single-disc refiner, and then pulp was screened using a set of 2 screens, a 12-mesh screen on top of a 200-mesh screen. The material remaining on the 12-mesh screen was considered as reject (shives), and the fibers that passed the 12-mesh screen but remained on the 200-mesh screen were considered as accept. To estimate the required refining, initial freeness was determined according to TAPPI 227 om-92, and then the pulp was refined to 400±25 mL CSF according to TAPPI 248 om-88, with a PFI Mill. Hand sheets (with basis weight of 127gm<sup>-2</sup>) were made according to TAPPI 205 om-88. Hand sheets were kept in a conditioning chamber at 23 °C & 50% RH for 24 hours. Then, basic weight, caliper, Corrugating Medium Test (CMT), Ring Crush Test (RCT), stiffness and tensile strength index, tear strength index, and the burst strength index of the hand sheets were determined according to TAPPI T410 om-98, T411 om-89, T809-om-99, T818-om-87, T240 om-92, SCAN P11:73, T403 om-91, and T403 om-91 test methods, respectively. One-way Variance Analysis test was used to analyze differences in strength properties of the hand sheets due to change in chemical charge of cooking liquor at confidence level of 95%. Meanwhile, average values of the properties were classified using Duncan multiple range testing.

**Results And Discussion**

**Chemical composition and fiber dimensions**

The percentage of cellulose, lignin, extractives soluble in alcohol-acetone, and ash are summarized in Table 1. Fiber dimensions and biometrical coefficient of eucalyptus are summarized in Table 2. The cellulose content of eucalyptus was found to be 48.3%, which is in the satisfactory range for pulp production. The cellulose content of eucalyptus is more than rice straw (41.20%) [19]and wheat straw (38.20%) [20]. The lignin content of eucalyptus was found to be higher than rice straw (21.90) and Egyptian cotton stalks (22.50) [21]. The Extractives soluble in alcohol-acetone content of eucalyptus was found to be almost similar to rice straw but higher than aspen (2.50%), and lower than wheat straw (7.80%). The ash content of eucalyptus was also low.

**Table 1:** Chemical Composition of Eucalyptus.

Composition	Cellulose (%)	Lignin (%)	Extractives Soluble in Alcohol-Acetone (%)	Ash (%)
Eucalyptus	48.3±1.38	28.5±1.22	5.9±0.35	0.65±0.03

**Table 2:** Results of Fiber Dimension Measurements.

	Length (mm)	Diameter (µm)	Lumen Width (µm)	Cell Wall Thickness (µm)	Runkel Ratio (%)	Slenderness Ratio (%)	Flexibility Coefficient (%)
Eucalyptus	0.935±0.1	19.13±0.9	8.89±0.8	5.12±0.5	115.19	48.88	46.47

Chemical composition of the raw material is one of the most important factors in pulp and paper production. Therefore, identification of chemical composition of eucalyptus has significant importance for predicting properties of the paper made. Cellulose is the most important component of cell wall in pulping process. Strength properties of paper increase with greater percentage of cellulose. Lignin is another component of cell wall which is responsible for connection of fibers to each other. Removing lignin is known as one major objective for any pulping process since once lignin has been solved and removed, cellulose fibers can establish more intermolecular bonds which will lead to higher strength of the paper produced [22].

Fibers were classified into three groups. The first group was considered short fibers with lengths of less than 0.9mm such as hardwood. The second group had an average length between 0.9-1.9mm. The results showed that the average fiber length of eucalyptus was 0.935mm. The third group included fibers longer than 1.9mm [23]. Eucalyptus fibers are shorter than wheat straw (1.73mm) [24]. On the other hand, the cell walls of eucalyptus fibers are thicker than those of aspen (1.93 $\mu$ m) (Law and Jiang 2001) [25] and cotton stalks (3.40 $\mu$ m) [26]. The calculated Runkel ratio for eucalyptus fibers (115.19%) is higher than that of cotton stalks (84%), aspen (23%), and date palm rachis fibers (80%). The slenderness ratio of eucalyptus fibers is 48.88 and is higher than that of cotton stalks (42.35) and aspen fibers (46.15), but the flexibility coefficient of eucalyptus fibers is less

than both cotton stalks (65.31) and aspen (81.44). This indicates good sheet forming potential from these fibers.

Dimensions including Length, diameter, lumen width and cell wall thickness have great effects on physical and mechanical properties of papers. In this regard, length of fibers has a very distinct role on improving strength indices of paper. Generally speaking, an acceptable amount for slenderness ratio for pulping is believed to be higher than 33 [27] such that eucalyptus fibers will be stand at this range.

### Pulping and pulp evaluation

Pulp yield after cooking is one important characteristic which must be measured after pulping process. The results show that a higher chemical charge reduced digester yield. The yield of these pulps varied between the highest values of 74.18% to the lowest value of 67.1%. Statistical analysis indicated that the effect of chemical charge as well as the combined effect of the variables on digester yield was statistically significant at 95% (Table 3). Therefore, to compare the averages, the Duncan multiple range test was used, and the results are shown in each figure using lower case letters. An increase of chemical charge resulted in a decrease of the pulping yield because of lignin and carbohydrate dissolution, especially hemicelluloses (under the influence of sodium sulphite and sodium bicarbonate). Generally, charging 10% chemical led to the greatest yield, and the lowest yield was related to 18% chemical charge as expected [23].

**Table 3:** Analysis of Variance (F-value) of the Results of eucalyptus NSSC Pulping.

	Yield	CMT	RCT	Tensile Strength Index	Stiffness	Tear Strength Index	Burst Strength Index
Chemical	108.7*	1215.1*	123.9*	30997.9*	23511.3*	719.9*	347.6*

Intensity of chemical reactions and delignification is increased with greater amount of chemicals used and the raw material becomes much softer. Thus, fibers will be separated more simply and there would be a better refining. Deniz I et al. [20] have reported that the effect of utilized chemical charge on properties of pulp is very crucial as the pulp yield is reduced with higher amounts of chemicals in the cooking liquor.

Furthermore, results of freeness distinguished that by increasing the percentage of chemical charge, treatment will be intensified, lignin dissolution will be increased, and water passing through pulps will be decreased due to more bonds between fibers. Moreover, greater amounts of chemical will intensify chemical reaction; encourage penetration of chemical into fiber structures; and increase their swelling which will lead to improve refining of the fibers. As a result, needed rotations of the refiner for meeting the desired freeness will be decreased.

The impact of chemical charge on strength properties, including tensile strength index, burst strength index, and tear strength index, was statistically significant at 95% (Table 3). The results showed that a higher chemical charge increased pulp strength property.

Even though the influence of chemical charge on strength indices revealed that higher chemical charges improved the strength values of the pulps, CMT, RCT, tensile index, Stiffness, tear index, and burst index, increased to 217.33KNm<sup>-1</sup>, 1.95KNm<sup>-1</sup>, 54.79Nmg<sup>-1</sup>, 770.22KNm<sup>-1</sup>, 7.43mNm2g<sup>-1</sup> & 3.86kPam2g<sup>-1</sup>, respectively. While the lowest value is related to the paper produced from pulp of MWPI, due to elimination of more lignin.

The most effective factors on mechanical properties are quality and quantity of bonds among fibers, fibers strength and length of fibers [28]. Among these factors, "bonds between fibers" is more important than others. Refining of the pulps introduces a positive effect on strength properties of the papers produced. Comparison of the sample (NSSC pulp of MWPI) (Figure 1) with the papers made from eucalyptus wood shows that the later were refined with higher rotations. Further refinement increases the quality and quantity of bonds between fibers, improves flexibility and thus, more fiber fibrillation than before. So enhanced mechanical properties of the papers made from eucalyptus can be partially attributed to the further refinement of its resultant pulps.

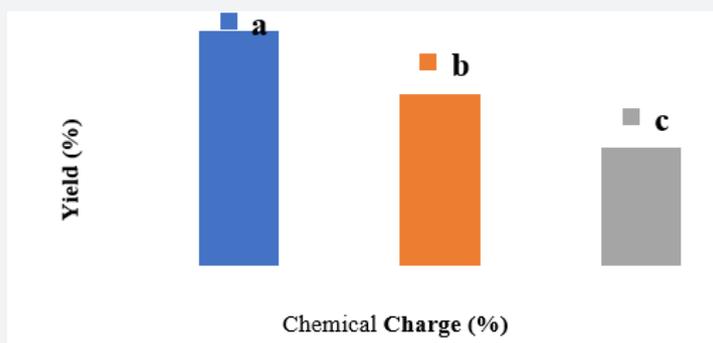


Figure 1: Effect of chemical charge on eucalyptus NSSC pulp yield.

Higher tear index of the eucalyptus paper can be explained by better biometrics coefficients which cause better refinement of the fibers and leads to improved bond of papers prepared from eucalyptus, as the most effective factor on the tear strength index is length of fibers. Increased length of the fibers improves tear strength, since in this case greater force would be required to cut the fibers [29]. In fact, length of fibers, slenderness ratio and Runkel ratio of eucalyptus fibers are almost the same as hardwoods of forests in northern Iran and even its Runkel ratio (115.19 $\mu$ m) is far better than many industrial hardwood species which itself can improve tear index additionally.

Two factors are effective on burst strength properties, namely length of fibers and bonds between fibers. Although increased length of fibers may lead to higher burst index, this

characteristic is more dependent on the bonding between fibers. Increased rotations of refiner will also enhance burst strength to some extent, while additional removing of lignin from pulp and formation of stronger bonds within pulp will also improve burst strength characteristics. The above-mentioned reasons can explain higher burst strength index for the samples produced from eucalyptus at various cooking conditions.

Figures 2-7 show the effects on pulp strength properties under the influence of chemical. The maximum amount of strength indices observed in the NSSC pulp produced by 18 % chemical charge while the minimum amount of them was observed in the NSSC pulp made in MWPI. It can be observed that all average values of resultant papers are categorized in four distinct groups (Figures 2-7).

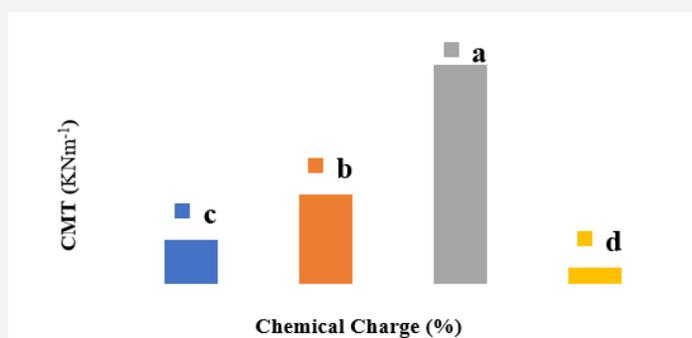


Figure 2: Effect of chemical charge on CMT of the eucalyptus NSSC pulp and NSSC pulp in MWPI.

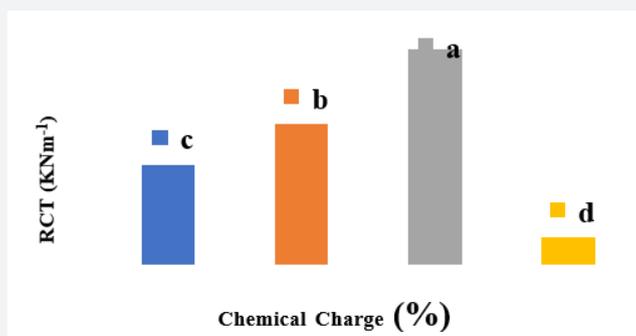


Figure 3: Effect of chemical charge on RCT of the eucalyptus NSSC pulp and NSSC pulp in MWPI.

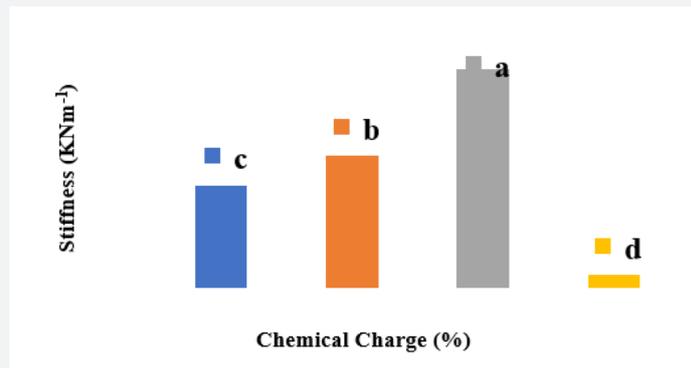


Figure 4: Effect of chemical charge on stiffness of the eucalyptus NSSC pulp and NSSC pulp in MWPI.

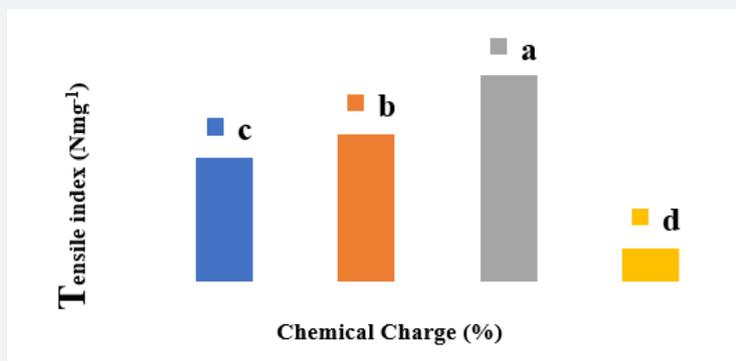


Figure 5: Effect of chemical charge on tensile index of the eucalyptus NSSC pulp and NSSC pulp in MWPI.

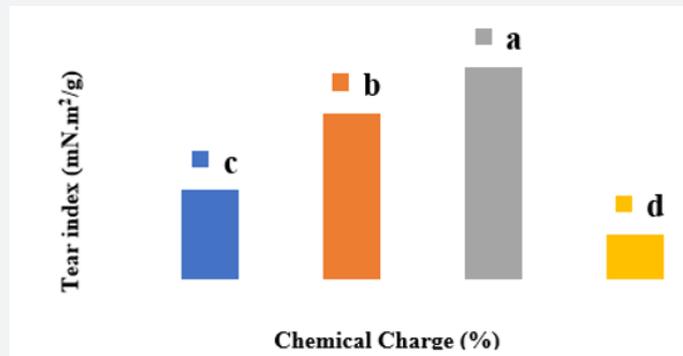


Figure 6: Effect of chemical charge on tear index of the eucalyptus NSSC pulp and NSSC pulp in MWPI.

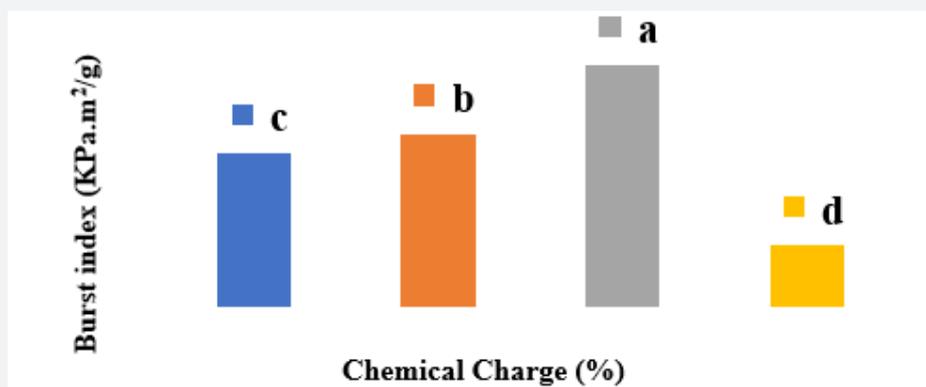


Figure 7: Effect of chemical charge on burst index of the eucalyptus NSSC pulp and NSSC pulp in MWPI.

## Conclusion

- a) The result of chemical content of eucalyptus is in the satisfactory range for pulp production.
- b) The results showed that pulp yield decreases with increase of chemical charge.
- c) Analysis of the mechanical properties indicated that the paper from NSSC eucalyptus at various cooking levels has preference to NSSC pulp of hardwoods produced in MWPI for all strength indices.

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