

Influence of Cooking Variables on the Soda and Soda-Ethanol Pulping of *Nypa Fruticans* Petioles

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Abstract: The influence of cooking variables on the soda and soda-ethanol pulping of *Nypa fruticans* petioles is evaluated. The raw materials were characterised to know its properties. The solubility in cold and hot water, 1% NaOH and ethanol-benzene were determined. Lignin, cellulose and ash content were also estimated. The effect of the pulping time (10 to 120 minutes), temperature (120°C and 150°C), concentration (8% and 12%) and cooking liquor (soda and soda-ethanol) on the pulp yield and Kappa number of *Nypa fruticans* petiole pulps were investigated. Pulping was done in the laboratory digester. The results of the physico-chemical analysis were within the range of those reported elsewhere for some non-woody pulping raw materials except solubility in cold and hot water was higher. Higher pulp yield and higher Kappa number were obtained with shorter pulping time (10 minutes) while longer pulping time results in lower pulp yield and Kappa number. At both concentrations and temperatures, soda liquor gave lower pulp yield with higher Kappa number while soda-ethanol gave higher pulp yield with lower residual lignin. Generally, pulp yield and Kappa number decreases with the increase in the temperature, concentration concentration of the cooking liquor and time.

Key words: *Nypa fruticans* Petioles, Soda, Soda-Ethanol, Pulp Yield and Kappa Number.

INTRODUCTION

Agricultural fibres constitute an alternative to wood as raw material for making pulp on account of their high growth rate and adaptability to various soil types. In Brazil where sugarcane is abundantly grown crop, bagasse is available for paper making, beside other non-wood materials such as wheat straw and various agricultural residues (Usta *et al.*, 1999).

There is a growing interest in using agricultural residues such as rice straw for pulping and paper-making. As future worldwide fibre shortages are predicted, agricultural fibres are believed to be a potential substitution for wood fibres in certain paper applications. Rice straw as an agricultural residue has been used in pulp and paper production for a long time and remains one of the major raw materials in many countries (Usta *et al.*, 1999).

The European Union has supported research on several non-wood species with high biomass production, that can be planted in areas made available from agriculture and that are adequate for different industrial uses (Van Dam *et al.*, 1994). The most frequent raw materials are straw, in particular wheat and rice straw and sugar cane bagasse (MacLeod, 1988). The E.U. consumption of pulps from annual plants is around 400,000 tonnes of which about one-quarter is imported (Van Dam *et al.*, 1994). The pulping of annual plants differs substantially from wood pulping. In most cases, delignification proceeds very fast and the resulting pulps have low residual lignin content.

Several processes have been developed, yet all based on chemical pulping technology. Soda, kraft and neutral sulphite semi-chemical (NSSC) pulping are suitable for producing pulps from straw and other non woody raw materials. Lime process is also still utilized for pulping. Among all pulping methods, soda and modified soda processes are superior to others. However, one of the most serious problems associated with straw chemical pulping is high silica content of fibres, which makes conventional recovery difficult (Pan and Leary, 2000, Utne and Hegborn, 1992). Organosolv pulping of straw has been evaluated by a number of investigators. (Kennedy *et al.*, 1996, Gullichsen and Fogelholm, 2000). Organosolv (solvent-based or solvolysis) pulping is a chemical pulping method in which delignification of the biomass (wood or non-wood) is done in an organic solvent or solvent plus water system. Most advantages of organosolv pulping of straw are low Kappa number of the resultant pulp, low impacts of environmental problems (as known to be an environmentally friendly pulping process) and the silica of straw does not accumulate in the system nor retained in the pulp (Rowell *et al.*, 1994). In order to improve optical properties of unbleached pulp or obtain bright (white) paper, bleaching treatment of pulp is usually done. Bleachability of non woody pulps with respect to wood pulps are easy, in addition, organosolv pulps were easily bleached than other chemical pulps (Rowell *et al.*, 1994).

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Moreover, soda-organosolv pulping is the other alternative method on soda pulping. There are several soda-organosolv pulping studies using non wood materials as rice straw (Pourjoozi *et al.*, 2004; Rodríguez *et al.*, 2008a; Rodríguez *et al.*, 2008b), empty fruit bunches (EFB) (Rodríguez *et al.*, 2008c), hemp (Gumuskaya *et al.*, 2007; Tjeerdsma *et al.*, 1994; Zomers *et al.*, 1995), reed (Shatalov and Pereira, 2002), sugarcane bagasse (Moriya *et al.*, 2005; Sanjuan and Gomez, 1991), wheat straw (Xu *et al.*, 2006; Goel *et al.*, 2000), cotton stalks (El-Sakhawy *et al.*, 1995), cereal straw (Tschirner *et al.*, 2002) and jute (Sahin, 2003). The main purpose of all modifications is to produce high quality pulp.

In general, organosolv pulping has low effect on carbohydrate degradation and has good selectivity in the delignification reaction (Muurinen, 2000). Organo solvent that have been used for pulping include; alcohols, acetone, anthraquinone, ethylene glycol, acetic acid, formic acid etc. Alkaline-ethanol pulping of cotton stalks was investigated by Akgül and Tozluolu, (2010). Ethanol-based pulping of *Cynaracardunculus* L. was examined by Oliet *et al.*, (2005). Base-catalyzed organosolv pulping of jute was carried out by Sahin, (2003). Cellulosic pulp from *Leucaena diversifolia* by soda-ethanol pulping process was investigated by López *et al.*, in 2011. There is a remarkably increase in pulp yield using ethanol pulping as compared to organic acid. Using soda and soda-ethanol pulping methods will solve the environmental problems associated with sulphite and kraft pulping.

In this work, delignification of *Nypa fruticans* petioles by soda and soda-ethanol was investigated at different cooking temperature, time and concentration of the cooking liquor and characterization of the resultant pulp by determining its Kappa number which indicates the extent of delignification and the amount of the bleaching agent that would be needed to bleach the pulp.

MATERIALS AND METHODS

The raw materials (*Nypa fruticans* petioles) used in this work were collected at Oron Beach, in Oron Local Government Area of Akwa Ibom State. The samples were dried in the air to reduce its moisture content; it was then cut into chips of about 2cm by 2cm. Moisture contents of the air dried chips were determined to know the weight of the dry matter. Part of the chips were ground and used for chemical analysis, while the remaining chips were used for pulping studies.

Specific Gravity and Fibre Dimensions:

Specific gravity signifies the density, workability or the strength properties of the pulping raw material. The specific gravity was determined using representative samples. The moisture content of the air dried sample chips were determined in order to permit description of the specific gravity on the basis of air dry matter. The chips where tie with a thread of known weight, the volume of the chips where determined by immersing it in a graduated measuring cylinder. The increase in volume after the sample was submerged was taken, the difference in volume before and after specimen immersion was taken as the volume of the sample.

Fibre dimensions of *Nypa fruticans* petioles were determined by cutting the samples into thin strip/filaments using razor blade. The filaments were soaked in a glass bottle containing (50/50. v/v) 80% acetic acid and 30% hydrogen peroxide for one week. It was later put in the oven for 48h with sequential shaking in mechanical shaker. The fibres were washed with distilled water to neutral pH. The fibre length and fibre width were determined using digital camera microscope, model: xsz 2003, at Forest Research Institute (FRIN) Ibadan, Oyo State.

Chemical Characterization:

The chemical characteristics of *Nypa fruticans* petioles were determined in accordance with TAPPI standard designated as follows:

- Solubility in hot and cold water - T207 cm99.
- Solubility in 1% NaOH - T212 om 02.
- Solubility in ethanol benzene -T204, cm 97.
- Lignin content -T222 om 98.

Cellulose content was determined using Kurschner-Hoffer cellulose method, (Kurscher and Hoffer, 1931).

Pulping Experiment:

Soda liquor was prepared from a standard concentrated solution of sodium hydroxide by series of dilution with de-ionized water. Soda-ethanol liquor was prepared by mixing equal volume of the respective solution of sodium hydroxide with a mixture of ethanol/water, 60/40 (v/v). The samples were pulped in the 10- litre stainless steel laboratory digester fitted with the heater and temperature controller. The chips of *Nypa fruticans* petioles were weighed and charged into the digester with the required amount of cooking liquor at liquor to solid ratio of 10:1. The cooking variables considered were temperatures (120°C and 150°C) and cooking time (10, 30,

60, 90 and 120 minute), which was then maintained throughout the experiment. The resultant pulp was washed thoroughly with tap water and the pulp yield was determined gravimetrically after drying to constant weight in the oven at 105°C. Kappa number of the pulp was determined in accordance with TAPPI standard designated T236 om 99.

RESULTS AND DISCUSSIONS

Nypa fruticans petioles were characterized and the results obtained are presented in Table 1 below.

Table 1: Physico-Chemical Characterization of the Raw Materials.

Solubility in cold water (%)	8.12
Solubility in hot water (%)	11.17
Solubility in 1% NaOH (%)	32.16
Solubility in ethanol-benzene (%)	2.38
Specific gravity	0.62
Lignin (%)	19.85
Cellulose (%)	42.22
Ash (%)	4.06
Fibre Length (mm)	1.06
Fibre diameter (µm)	12.00

Table 2: 8% Soda Pulping of *Nypa fruticans* Petioles.

Temperature (°C)	120	120	120	120	120	150	150	150	150	150
Time to Temp. (Min.)	25	22	24	27	30	42	36	35	34	35
Time at Temp. (Min.)	10	30	60	90	120	10	30	60	90	120
Pulp Yield (%)	47.61	46.85	45.43	43.26	41.65	33.04	28.28	26.24	26.16	23.23
Kappa Number	36.5	35.7	35.6	35.9	30.5	28.3	19.9	14.3	14.0	11.1

Table 3: 8% Soda-ethanol Pulping of *Nypa fruticans* Petioles.

Temperature (°C)	120	120	120	120	120	150	150	150	150	150
Time to Temp. (Min.)	25	22	24	27	30	42	36	35	34	35
Time at Temp. (Min.)	10	30	60	90	120	10	30	60	90	120
Pulp Yield (%)	51.90	49.02	47.92	47.06	46.40	34.56	32.83	31.08	29.95	27.50
Kappa Number	36.0	35.4	32.3	31.2	22.7	19.0	18.1	14.1	12.4	10.5

Table 4: 12% Soda Pulping of *Nypa fruticans* Petioles.

Temperature (°C)	120	120	120	120	120	150	150	150	150	150
Time to Temp. (Min.)	25	22	24	27	30	42	36	35	34	35
Time at Temp. (Min.)	10	30	60	90	120	10	30	60	90	120
Pulp Yield (%)	46.95	44.02	40.22	39.46	35.26	30.89	25.80	23.69	21.85	20.73
Kappa Number	36.3	35.7	35.2	35.2	31.7	28.2	18.2	14.3	10.2	8.3

Table 5: 2% Soda-ethanol Pulping of *Nypa fruticans* petioles.

Temperature (°C)	120	120	120	120	120	150	150	150	150	150
Time to Temp. (Min.)	25	22	24	27	30	42	36	35	34	35
Time at Temp. (Min.)	10	30	60	90	120	10	30	60	90	120
Pulp Yield (%)	51.60	49.47	47.18	46.47	45.50	30.87	28.74	27.74	24.76	23.86
Kappa Number	33.9	32.6	30.6	31.7	29.8	17.7	13.4	6.7	6.5	2.3

Discussion:

Specific Gravity:

Nypa fruticans petioles were characterized with high value of specific gravity. The specific gravity as presented in Table 1 was 0.77 which fall within the range 0.3-0.8 reported by Lee *et al.*, 1994, for bamboo. The high specific value is an indication of high liquor consumption by the sample chips and high pulp yield.

Fibre Characteristics:

The fibre length and fibre diameter of *Nypa fruticans* petiole as presented in Table 1 were 1.06mm and 12µm respectively. The fibre length was, was lower than that of hardwood and softwood (Grace and Malcolm, 1993). it was also lower than that of wheat straw, (1.5mm) and bagasse, (1.0-1.5mm), bamboo (2.7-4mm), (Pande,1998), Rice straw (1.48mm), reed grass (1.5mm) (Hunter, 1988) and Jute (2.5mm), kenaf (2.74mm) (Hunter, 1988). The fibre width of *Nypa fruticans* petiole was very small, nearly about 12 µm, which was lower as compared to wood and other common non-wood fibre (approximately 32.0-43.0µm and 18.0-30.0µm for soft- and hardwoods, respectively) (Rydholm, 1976). However, it is longer than that of esparto (9.0 µm) and rice straw (11.0 µm). The fibre length of any plant is important because there is a strong relationship between the fibre length and the strength properties of the resultants pulp (Ververis *et al.*, 2004).

Chemical Composition:

The solubility in cold water (22.36%) and hot water (22.61%) is an indication of high content of tannins, gums, sugar, colouring matter and starch. The solubility in ethanol-benzene (2.51%) indicates the measure of the waxes, fats, resins, oils and tannins and certain ether-insoluble components and 1% sodium hydroxide solubility (6.09%) indicates the degree of fungus decay that can take place in a given wood or non-woody sample. The higher soluble content is also an indication of easy access and degradation of the sample in weak alkali (Kristova *et al.*, 1998). It therefore means that the soda liquor concentration should be low in order to preserve the cellulose content and enhance good pulp yield. However, the results of, ash, cellulose and lignin content as presented in Table 1 are within the range of those reported by Hunter (1988), for others non wood raw materials. High cellulose contents indicates high pulp yield and high lignin content indicate high liquor consumption

Influences of Cooking Variables:

The results and the pulping conditions are presented in Tables 2 to 5. The cooking variables considered in this work were; temperature and time, type and concentration and pulping liquors.

Effects of Cooking Temperature and Time:

As presented in figure 1 and 2, higher pulp yield and higher Kappa number were obtained at the lower temperature of 120°C, while lower pulp yield and lower Kappa number were obtained at the higher temperature of 150°C. This implies that, high rate of degradation of cellulose and delignification occurs at elevated temperature. During pulping both lignin and cellulose are dissolved at different rates. This rate is much accelerated by increasing the temperature (Iglesias *et al.*, 1996). There was a general decrease in the pulp yield and Kappa number due to increases in the pulping time at constant temperature.

Effects of Concentration and Pulping Liquor:

Effect of concentration and pulping liquor is illustrated in figure 1 and 2. Higher concentration of the pulping liquor results in the lower pulp yield and Kappa number. This is because the higher the concentration of the cooking liquor, the higher the rate of delignification and degradation of cellulose. Although there was a remarkably increase in the pulp yield when soda-ethanol pulping was carried out, this was due mainly to the fact that organic solvent has the tendency of reducing the rate at which soda degrades the cellulose fibres.

However, the type of pulping liquor has greater effect on the yield and Kappa number *Nypa fruticans* petioles. At both temperatures soda-ethanol gave higher pulp yield, while soda pulping gave lower pulp yield. The predominant degradation reaction in soda condition is the peeling reaction in which some glucose units are removed from cellulose molecule and decreases its degree of polymerization (DP) (Muurinen, 2000). The chips pulped with an aqueous mixture of alcohol and sodium hydroxide are easier to defibre than those pulped with soda. Moreover, organo-solvent pulping has lower effect on carbohydrate degradation and has good selectivity in the delignification reaction (Muurinen, 2000). The lower pulp yield of soda pulping is due to the high rate of degradation of cellulose during pulping.

On the other hand, Kappa number entails the amount of residual Klason lignin of the pulp. Residual Klason lignin indicates the amount of bleaching agent that will be needed to bleach the pulp. At all temperatures and concentrations, soda pulping gave higher value of residual Klason lignin which implies lower rate of delignification whereas at the same conditions, soda-ethanol gave lower value of residual Klason lignin which in turns implies higher rate of delignification. This is because organic solvent reduces the surface tension of the pulping liquor promoting the penetration of the alkali into the chips and the diffusion of the breakdown products of lignin from the chips to the liquor. Simultaneously, ethanol also degrades lignin and prevents it from condensing, this result in the low residual lignin obtained with soda-ethanol pulping.

Conclusion:

The increasing demands for paper and environmental concerns have increased the need for non-wood pulp as a low-cost raw material for papermaking. This has also led to the developing of alternative pulping technologies that are environmentally benign. Annual plants and agricultural residues appear to be well suited for producing papermaking pulp due to the fact that they are abundant and renewable resource.

The result of the chemical composition of *Nypa fruticans* petioles indicates its suitability as a pulping raw material. Extractive components are within the acceptable range for the non-woody pulping raw material. It has been observed that soda pulping lead to higher rate of cellulose degradation and high residual lignin. Organosolv pulping processes are alternatives to conventional pulping processes and have environmental advantages. Adding ethanol into soda pulping liquor resulted in an increase in pulp yield and reduction in Kappa number. The positive effect of ethanol in pulping was brought about by prevention of cellulose degradation and high rate of delignification. Temperature, concentration and cooking time also exert positive effect on the pulp yield and Kappa number.

However, since the *Nypa fruticans* petioles have short fibre length and width, the pulp obtained can be mixed with long fibres from soft wood or other non woods to produce paper with good strength properties. It can also be utilized for the preparation of cellulose derivatives.

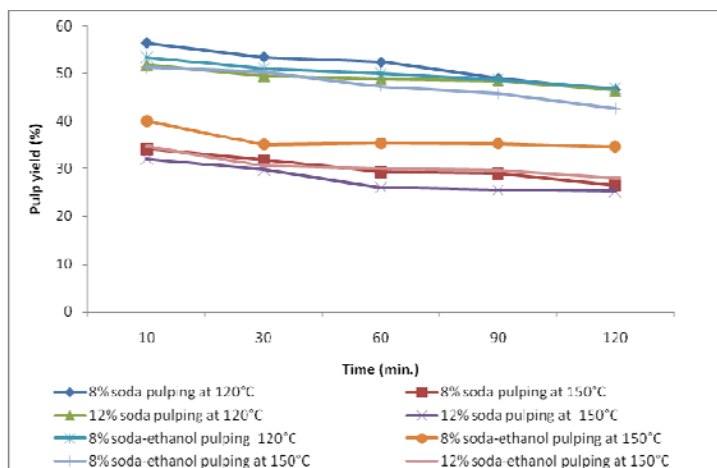


Fig. 1: Plot of pulp yield against time showing influence of cooking variable on the pulp yield of *Nypa fruticans* petiole.

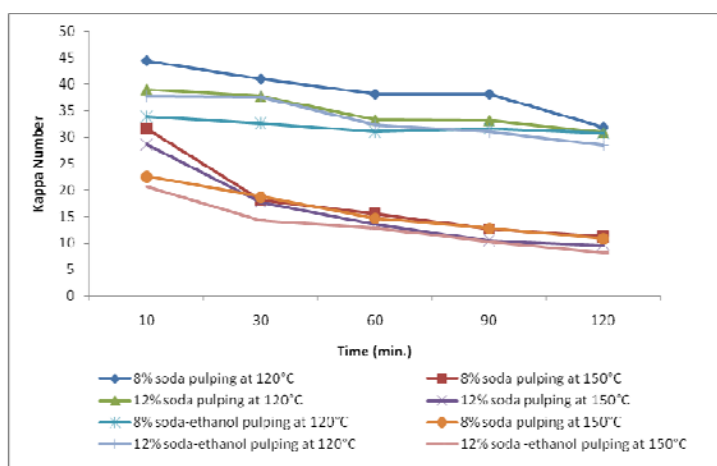


Fig. 2: Plot of kappa number against time showing influence of cooking variable on Kappa number of *Nypa fruticans* petiole

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