

# Sponge Gourd Potential for the Raw Material of Paper and Dissolving Pulp

Nyoman J Wistara<sup>1\*</sup>, Yuhana Rahayu<sup>2</sup>

<sup>1</sup>Department of Forest Products, Faculty of Forestry and Environment, IPB University

<sup>2</sup> Agro Business Division of the Office of Public Assessor Services of the Abdullah Fitriantoro & Partner

\*Corresponding Author: nwistara@apps.ipb.ac.id

## Abstract

Sponge gourd (*Luffa cylindrica*) provides a sustainable and cheap source of lignocellulosic materials potential for the raw material of paper and dissolving pulp. In the present works, sponge gourd fruits 5 and 8 months old were pre-hydrolyzed in an acidic (pH of 5) and neutral (pH of 7) at 165 °C for 3 hours, and successively soda pulped at a maximum temperature of 170 °C for 4 hours with 20% active alkali. The resulting pulp was bleached by the D<sub>0</sub>E<sub>1</sub>D<sub>1</sub>E<sub>2</sub>D<sub>2</sub> method. The bleached pulp was chemically analyzed to evaluate its feasibility for dissolving pulp based on the requirement of SNI 14-03-1989. The pulp feasibility for paper materials were based on the measurement of fiber dimensions, fiber derivative values, and the strength properties of the pulp. It was found that the  $\alpha$ -cellulose content of the sponge gourd coir was high compared to the SNI 14-03-1989. However, it did not satisfy the requirement of dissolving pulp due to its high extractives, ash, silica content, and low viscosity. The fiber of sponge gourd coir was classified into quality class II with intermediate pulp strength. Appropriate treatments should be examined to decrease the content of ash, silica, and extractives of the resulting pulp need to be further evaluated.

**Keywords:** Dissolving pulp, paper, *Luffa cylindrica*, pre-hydrolysis, soda pulping

## Introduction

Dissolving pulp is the raw material of cellulose derivative products (Arnoul-Jarriault *et al.* 2015) with an increasing global demand during the last decade (Liu *et al.* 2016, Gehmayer & Sixta 2011). Dissolving pulp can be derived from cotton, wood, and non-wood cellulosic materials (Ma *et al.* 2011).

Sponge gourd is a lignocellulosic material, possibly potential for the raw material of dissolving pulp. It consists of 5-7 species, two of which have been cultivated, i.e., *Luffa cylindrica* and *Luffa acutangula* (Bal *et al.* 2004). It is considered a sustainable and cheap source of cellulose with an increasing economic value (Mazali & Alves 2005). A recent genetic study by Wu *et al.* (2020)

provides information for developing a high-quality sponge gourd, thus ascertaining its sustainability. Its chemical composition is influenced by its growing site and climate conditions, with the average content of cellulose, hemicelluloses, lignin, extractives, and ash of 55-90%, 8-22%, 10-23%, 3.2%, and 0.4%, respectively (Satyanarayana *et al.* 2007), and its average density of 0.82 – 0.92 g cm<sup>-3</sup> (Tanobe *et al.* 2005). Lignocellulosic material of high cellulose content is a very appropriate raw material for dissolving pulp and paper products.

Dissolving pulp can be prepared by soda, sulfite, and pre-hydrolyzed kraft processes (Barba *et al.* 2002). It should be highly pure, free of hemicelluloses and lignin, with controllable ash content, silicate content, polymerization degree

(Jahan 2009), uniform molecular weight distribution, high brightness, and high cellulose reactivity (Matin *et al.* 2015). An environmentally benign ECF bleaching process can be used to obtain pulp with high purity (Hamzeh *et al.* 2007). Vapor phase hydrolysis (Kauto *et al.* 2007), autohydrolysis (Leschinsky *et al.* 2009), and dilute acid hydrolysis (Al-Dajani *et al.* 2009) preceding pulping process were very useful in decreasing hemicelluloses, lignin, and ash content. The pre-hydrolysis process facilitates pulping and bleaching to produce highly pure pulp (Testova *et al.* 2014). Hydrolysis in an acidic media has decreased pulping yield and brightness but increased the resulting pulp's alkaline solubility (El-Ghany 2009).

Fiber dimension and its derivatives are essential to evaluate the feasibility of lignocellulosic material for pulp and paper products. Fiber derivative value includes the Runkle ratio, felting power, flexibility ratio, Muhlstep ratio, and rigidity coefficient. Fiber derivative values have been successfully applied to evaluate the quality of kenaf fiber as the raw material for paper production (Ververis *et al.* 2004). These authors found that kenaf fiber retained a Runkle ratio and felting power of 0.7 and 105.9, respectively, and assumed that kenaf fibers should result in a paper with good mechanical strength.

Along with kenaf, sponge gourd is a source of lignocellulosic material abundantly available in Indonesia. Therefore, with its high cellulose content (Satyanarayana *et al.* 2007) and its increasing economic value (Mazali & Alves 2005), an evaluation of the sponge gourd's potential as the raw material for dissolving pulp and paper products was done in the present research.

## Materials and Methods

Sponge gourd fruits 5 and 8 months old were procured from the District of Garut, West Java Province of Indonesia. The fruits were peeled to obtain their sponge and air-dried for several days. The sponge was then cut into 3-7 mm lengths. The chips were pre-hydrolyzed either in neutral with demineralized water (pH of 7) or in acidic media with H<sub>2</sub>SO<sub>4</sub> (pH of 5). It was carried out at the maximum temperature of 165 °C with a liquor-to-wood (chips) ratio (L/W) of 12 to 1.

The pre-hydrolyzed chips were then cooked with a soda process at a maximum cooking temperature of 170 °C, L/W of 12, and active alkali of 20% for 4 hours. The kappa number of the resulting pulp was determined following the TAPPI T 236 cm-85 standard procedures. D<sub>0</sub>E<sub>1</sub>D<sub>1</sub>E<sub>2</sub>D<sub>2</sub> bleaching method was used to bleach the pulp, proceeded by pulp washing successively with 1% HCl solution and hot water to neutrality. Table 1 lists the currently applied bleaching conditions.

Table 1 The bleaching conditions of the pulp

Parameter	D <sub>0</sub>	E <sub>1</sub>	D <sub>1</sub>	E <sub>2</sub>	D <sub>2</sub>
ClO <sub>2</sub> , % as Cl <sub>2</sub>	0.22 x KN	-	-	-	-
ClO <sub>2</sub> , %	-	-	1	-	0.5
NaOH, %	-	1.5	-	1.5	-
Consistency, %	10	10	10	10	10
Temperature, °C	60	60	70	60	70
Time, min	60	60	180	60	180

Measurements of fiber length, fiber diameter, and cell wall thickness were carried out by OLYMPUS *fiber optic tester* PTCB-E02 microscope. The fiber sample was stained with safranin on a petri dish and was kept for 12 hours before measurement. Measurement was carried out on 100 fibers at 200 magnifications for fiber diameter and 50 magnifications for fiber length.

Chemical component analysis on bleached pulp was carried out by SNI 14-

0938-1989 specification. The content of  $\alpha$ -cellulose, solubility in 10% and 18% NaOH, extractives, and ash and silicate were determined based on TAPPI 236 cm-85, TAPPI T 203 om-88, TAPPI T 244 om-88, and TAPPI T 211 om-85, respectively. Hand sheets for testing the pulp's mechanical properties were prepared per TAPPI T 205 sp-95. Tensile strength, tearing strength, bursting strength, pulp brightness, and pulp viscosity were determined based on the standard procedures of TAPPI T 494 om-96, TAPPI T 414 om-98, TAPPI 403 om-97, SNI 14-0936-1989, and SNI 14-4733-1989, respectively.

The resulting data were analyzed using a completely randomized design of 2 factors, i.e., two levels of the acidity of the hydrolysis process (pH 5 and 7) and two levels of the age of sponge gourd fruits (5 and 8 months old), each with duple replication. The analysis was carried out using SAS 9.1 for windows software. Duncan MRT analysis was applied to determine the differences in the examined parameters.

## Results and Discussion

### The Yield and the Kappa Number of Unbleached Pulp

The yield of pre-hydrolysis, pulping, and bleaching stages and the kappa number of the unbleached pulp can be seen in Table 2. The yield of pre-hydrolysis of the sponge gourd was in the range of 53.75-65.36%. The hydrolysis acidity and the age of sponge gourd fruits did not influence the hydrolysis and pulping process; however, they controlled the yield of the bleaching stage (at  $\alpha=95\%$ ). The pulp bleaching yield of the 8-month-old sponge gourd fruit was higher than that of the 5-month-old sponge gourd fruit. The resulting yield was considered

low, i.e., in the range of 41.13-49.63%. The bleaching yield of PAU5 and PNU5 was statistically the same, but they were lower than these of PAU8 and PNU8. The bleaching yield of the PAU8 was the highest (49.63%).

The kappa number indicates the residual lignin of the pulp. It determines the number of chemicals used in the pulp bleaching stage. The kappa number of sponge gourd pulp was 9.1-11.1. The hydrolysis acidity and the age of the sponge gourd fruits did not influence the kappa number of pulp (at  $\alpha=95\%$ ).

Table 2 The yield of the pre-hydrolysis, pulping, and bleaching stage and the kappa number of the unbleached pulp

Sample	Yield, %			Unbleached Pulp Kappa Number
	Pre-hydrolysis stage	Pulping stage	Bleaching stage	
PNU5	57.48	43.12	41.13	11.1
PNU8	53.75	52.37	45.76	10.1
PAU5	63.64	48.54	43.16	9.4
PAU8	65.36	54.37	49.63	9.1

Note: P= pre-hydrolysis, N = neutral (pH=7), A= acidic (pH=5), U5= 5 months old, and U8= 8 months old

### Dissolving Pulp Potential of Sponge Gourd

Chemical component analysis was carried out on bleached pulp; the results are listed in Table 3. The purity of dissolving pulp is indicated by its  $\alpha$ -cellulose content, in which the SNI standard requires a minimum of 90.5%. For the present works, the bleached pulp of sponge gourd retained the  $\alpha$ -cellulose content of 91.17-94.74%. Pre-hydrolysis process (Wanrosli *et al.* 2004) and pulping with active alkali of more than 17% (Fengel & Wegener 1984) resulted in pulp with high  $\alpha$ -cellulose content. The content of the  $\alpha$ -cellulose of sponge gourds was not significantly influenced by the hydrolysis acidity and the age of the sponge gourd fruits.

The solubility of pulp in 10% NaOH indicates the content of short-chained cellulose (degraded cellulose), and the solubility of pulp in 18% NaOH suggests the range of hemicelluloses of the pulp. The presence of hemicelluloses is unacceptable in dissolving pulp and other cellulose derivatives. It can be seen from Table 3 that the 10% NaOH solubility of most pulp was higher than that required by SNI (a maximum of 10%), and the 18% NaOH solubility of all pulp was higher than that needed for the SNI (a maximum of 6.5%). At high concentrations, hemicelluloses disturb the preparation of viscose (Gehmayer & Sixta 2011) and bring about brittle rayon yarn (Pari *et al.* 2005). It was found in the present research that the hydrolysis acidity and the age of sponge gourd fruits did not influence the solubility of pulp in 10% and 18% NaOH solutions.

Table 3 Chemical composition of sponge gourd bleached pulp

Component	Sample				SNI 14-03-1989
	PNU5	PNU8	PAU5	PAU8	
$\alpha$ -Cellulose, %	94.40	91.20	94.70	94.40	$\geq 90,50$
10% NaOH, %	9.95	10.83	11.32	10.55	$\leq 10,00$
18% NaOH, %	7.01	7.61	7.15	7.36	$\leq 6,50$
Ash, %	0.18	0.43	0.12	0.23	$\leq 0,15$
Extractive, %	0.40	0.38	0.44	0.32	$\leq 0,30$
Silica, mg/kg	38.50	85.00	28.50	42.00	$\leq 50,00$
Brightness, %	91.00	90.00	91.00	91.00	$\geq 90,00$
Viscosity, mPa.S	5.15	4.75	5.35	5.05	$\geq 18,00$

Note: P = pre-hydrolysis, N = neutral (pH =7), A = acidic (pH = 5), U5 = 5 months old, U8 = 8 months old.

The silica content of the 5-month-old sponge gourd pulp was lower than those of the 8-month-old sponge gourd (at  $\alpha=95\%$ ). Except for pulp obtained from neutral pre-hydrolyzed of eight-month-old sponge gourd (PNU8), the silica content of all pulp was lower than 50 ppm as required by SNI 14-03-1989. The silica content of PAU5, PNU5, and PAU8 was statistically the same but lower than PNU8. The hydrolysis acidity and the age

of sponge gourd fruits did not influence the ash content of its bleached pulp. The ash content of pulp obtained from acidic pre-hydrolysis for the five-month-old sponge gourd (PAU 5) was met by SNI 14-03-1989. High ash and silica content brings about a decreasing strength of pulp because ash and silica hinder the occurrence of inter-fiber hydrogen bonding (Wirawan *et al.* 2010).

Extractives precipitate in the cell wall and cavities without chemical bonding with the primary cell wall components. The solubility of pulp in alcohol-benzene is indicative of its extractive content. Alcohol-benzene dissolves resin, fat, wax, and tannin. It was found in the present works that the hydrolysis acidity and the age of sponge gourd fruits did not influence the content of extractives. It can be seen from Table 3 that the extractives content of sponge gourd bleached pulp was higher than that required by SNI 14-03-1989, which is a maximum of 0.3%. High extractive content in dissolving pulp brings about brown specks in pulp and disturbance in the spinning and screening processes of the rayon yarn (Afriani 2007).

The brightness of the pulp represents the magnitude of light reflected by the pulp relative to that reflected by the reference object (titanium oxide) in %ISO or °GE. The brightness of sponge gourd bleached pulp obtained in the present research was 90-91% ISO and satisfied the SNI 14-03-1989 ( $> 90\%$  ISO) requirement. The effect of hydrolysis acidity and the age of sponge gourd fruits were insignificant on the bleached pulp brightness. Pulp brightness can be improved with peroxide (P) bleaching. Peroxide can deactivate chromophore groups in lignin to prevent light absorption by the pulp (Fengel & Wegener 1984).

Pulp viscosity indicates the degree of polymerization and the degradation degree of cellulose in the pulp. Cellulose degradation decreases the pulp viscosity (Jautsimo 2004) and pulp strength (Wathen *et al.* 2005). Table 3 indicates that pulp viscosity is positively related to cellulose content. However, it was negatively correlated to the brightness of pulp, which agrees with that reported by Jahan *et al.* (2008). The present result indicates that the viscosity of bleached sponge gourde pulp was 4.75-5.35 mPa.S. Unfortunately, far below SNI 14-03-1989 required the minimum pulp viscosity of 18 mPa.S. The effect of the hydrolysis acidity and the age of sponge gourd fruits on pulp viscosity was significant ( $\alpha=95\%$ ). The viscosity of PAU8, PAU5, and PNU5 was statistically the same. However, they were lower than that of PNU8. Degrading cellulose to pre-hydrolysis treatment could decrease pulp viscosity (Xu *et al.* 2006).

### Sponge Gourd Potential for the Raw Material of Paper

Table 4 indicates the quality classification of sponge gourd fiber for pulp and paper

raw material according to the Forest Products Research Institute (LPHH) (Rachman & Siagian 1976). The fiber quality is classified based on the derivative value of the fibers. Even though fiber's derivative values are traditionally instrumental in predicting the papermaking potential of lignocellulosic material, the measurement of fiber property distribution has been claimed to be more principal in representing the quality of pulp (Pulkkinen *et al.* 2006).

The hydrolysis acidity influenced the fiber length, diameter, and lumen diameter of sponge gourd pulp. It can be seen from Table 4 that the currently studied sponge gourd retained longer fiber than that of the Costarican sponge gourd (fiber length of 1.31 mm) reported by Rojas (2007) and narrower fiber diameter than that of the Brazilian sponge gourd (fiber diameter of 233  $\mu$ m) written by Guimaraes *et al.* (2009). The fiber length of the presently investigated sponge gourd was between hardwood (1.16 mm) and softwood (2.72 mm) (Pulkkinen *et al.* 2006). Fiber length affects the strength properties of pulp.

Table 4 The quality classification of sponge gourd fiber for the raw material of pulp and paper

Fiber dimension and the value of its derivatives	Quality Classification of the fiber according to LPHH			Sample			
	I (100)	II (50)	III (25)	PNU5	PNU8	PAU5	PAU8
Fiber length, mm	> 2	1 – 2	< 1.00	1.44	1.34	1.52	1.55
Fiber diameter, $\mu$ m	-	-	-	21.08	23.38	25.03	25.90
Lumen diameter, $\mu$ m	-	-	-	14.13	15.48	17.08	18.70
Cell wall thickness, $\mu$ m	-	-	-	6.95	7.90	7.95	7.20
Runkle Ratio	< 0.25	0.25 – 0.5	> 0.5 – 1	0.98	1.02	0.93	0.78
Felting Power	> 90	50 – 90	< 50	68.61	57.63	60.79	59.88
Muhlstep ratio, %	< 30	30 – 60	> 60	55.02	56.09	53.37	47.88
<i>Flexibility ratio</i>	> 0.8	0.5 – 0.8	< 0.5	0.33	0.34	0.32	0.28
<i>Coefficient of rigidity</i>	< 0.1	0.1 – 0.15	> 0.15	0.67	0.66	0.68	0.72
Interval	450 – 600	225 – 449	< 225	225	225	225	225

Note: P=pre-hydrolysis, N=neutral, A=acidic, U5=5 months old, U8=8 months old

Fiber with a low Runkle ratio is an excellent raw pulp material and can be readily processed into good paper. The present works indicated that the Runkle ratio of sponge gourd fiber ranged from 0.78 to 1.02 and classified as a quality class of III based on LPHH criteria. The acidity of hydrolysis influenced the Runkle ratio. PAU8 retained the lowest Runkle ratio; therefore, it could provide relatively higher tensile and bursting strength.

Felting power mainly influences the tearing strength of pulp. The felting power and the Muhlstep ratio of the sponge gourd fiber in the present research ranged from 57.63 to 68.61 and 47.88-56.09%, respectively. The fibers are classified into quality class II. Even though it has fallen in the same quality class, its felting power was higher than *Acacia mangium* of Papua New Guinea, Queensland, and East Indonesia provenances reported by Syafii & Siregar (2006). The Muhlstep ratio of the sponge gourd fiber was comparable to those written by the just mentioned authors. Felting power is positively correlated to the tearing strength of pulp and paper. The Muhlstep ratio is positively related to the paper strength dependent on inter-fiber bonding such as tensile, folding, and bursting strengths. The hydrolysis acidity and the age of the sponge gourd did not influence the felting power of its fiber. It can also be seen from Table 4 that the decrease in the hydrolysis acidity decreased the Muhlstep ratio.

Based on the rigidity coefficient (0.66-0.72) and flexibility ratio (0.22-0.34), the sponge gourd fiber is classified into quality class III. The coefficient of rigidity increased with increasing the acidity of hydrolysis. Fiber stiffness influences paper formation, in which stiffer fiber will result in a worsened

formation with the consequence of reduced paper strength due to reduced inter-fiber bonding capability. The statistical evaluation indicated that PNU8 pulp retained the highest flexibility ratio. Flexible fiber produces good paper strength because of the increasing relative bonded area.

Table 4 indicates that sponge gourd fiber is classified into quality class II based on the scoring of fiber derivative values. According to LPHH criteria, fiber with a class quality of II will produce a paper with medium tearing, bursting, and tensile strength. Except for cell wall thickness and felting power, all other fiber dimensions and fiber derivative values were influenced by the acidity of hydrolysis. Fiber dimensions and fiber derivative values increased with increasing hydrolysis acidity. Pre-hydrolysis was possibly capable of swelling the fibers.

### **The Strength Properties of Pulp**

The tensile, tearing, and bursting strength of its pulp handsheets was determined to evaluate the papermaking potential of the sponge gourd. The resulting strength properties evaluation of the fibers is listed in Table 5. The acidity of hydrolysis and the age of sponge gourd fruits influenced tensile strength. It was found that pre-hydrolysis in acidic conditions of the five months old sponge gourd (PAU5) resulted in pulp with the highest tensile strength.

Regardless of its equal scoring value (the same quality fiber class), PAU5 retained the highest tensile strength. Fiber derivative values are determined based on the mean of fiber length, fiber diameter, and cell wall thickness. However, fiber property distribution is considered more prominently crucial in determining the

strength properties of pulp (Pulkkinen *et al.* 2006). Furthermore, modification of cellulose and hemicellulose characters could increase inter-fiber bonding quality (Minor and Atalla 1992). The bursting strength of the sponge gourd pulp was only influenced by the age of sponge gourd fruits. The same factors control tensile strength as these bursting strengths. In general, tensile strength is inversely correlated to tearing strength, as shown in Table 5. PAU5 pulp retained the lowest tearing strength.

Table 5 The strength properties of the sponge gourd pulp.

Sample	Burst Index (kPa, m <sup>2</sup> g <sup>-1</sup> )	Tear Index (Nm <sup>2</sup> kg <sup>-1</sup> )	Tensile Index (Nm g <sup>-1</sup> )
PNU5	0.62	4.22	13.98
PNU8	0.35	4.15	6.04
PAU5	0.64	3.07	18.27
PAU8	0.37	4.21	6.02

Note: P=pre-hydrolysis, N=Neutral, A=acidic, U5=5 months old, U8=8 months old.

In general, acid hydrolysis might swell the fiber structure facilitating the impregnation of pulping and bleaching chemicals that significantly influence bleaching yield, pulp viscosity, and pulp strength. Acidic treatment has also been reported to reduce ash content (including the content of silica), such as that found in the demineralization process of oil palm powder (Wistara *et al.* 2021).

### Conclusions

The acidity of hydrolysis and the age of the sponge gourd fruits influenced the silica content, viscosity, bleaching yield, and bursting strength of the resulting pulp. Hydrolysis acidity could swell the pulp, affecting the fiber dimensions and derivative values. Even though the  $\alpha$ -cellulose content of sponge gourd fruit was relatively high according to SNI 14-03-1989, it did not satisfy the raw material requirements for dissolving pulp due to its

high extractives, ash, and silica along with its low pulp viscosity. Treatments to decrease the content of ash, silica, and extractives of the resulting pulp need to be further evaluated. Appropriate pulping conditions could result in pulp with higher viscosity.

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