Comparative study between Federer and Gomez method for number of replication in complete randomized design using simulation: study of Areca Palm (*Areca catechu*) as organic waste for producing handicraft paper

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Abstract. The part of Areca Palm (*Areca catechu*) that economical is the seed. It is commercially available in dried, cured and fresh forms, while the fibre is usually thrown away. Cellulose fibers from agricultural waste can be utilized as raw material for handicraft paper. Laboratory research showed that Areca palm fibre contained 70.2% of cellulose, 10.92% of water, and 6.02% of ash. This indicated that Areca palm fibre is very potential to be processed as handicraft paper. Handicraft paper is made of wastepaper or plants which cointain celluloce to produce rough-textured paper. In order to obtain preferred sensory quality of handicraft paper such as color, fiber appearance and texture as well as good physical quantity such as tensile strength, tear resistance and grammage, the addition of wastepaper to provide secondary fibre and sometimes adhesive are needed in making handicraft paper. Handicraft paper making was one alternative to treat the solid waste and to reduce the use of wood fiber as paper raw material. The aim of this study is to compare the two most famous method, i.e. Federer and Gomez Method, for calculate the number of replications. This study is preliminary research before do the research in order to get the best treatment to produce handicraft paper. The Gomez method calculates fewer replications than the Federer method. Based on data simulation the error generated using 3 replicates of 0.0876 while using 2 replicates of 0.1032.

1. Introduction

The areca palm is one of plants that has not received serious attention, compared to other palm trees. In Indonesia, this plant is widely found in Sumatra (Aceh, North Sumatra and West Sumatra), Kalimantan (South Kalimantan and West Kalimantan), Sulawesi (South Sulawesi and North Sulawesi) and Nusa Tenggara (West Nusa Tenggara and East Nusa Tenggara) [1]. Areca palm is still limitedly utilized. This can be seen in the use of its seeds which is only eaten with betel and it is only used in some traditional events served as an ingredient which become a habit for generations in some specific

areas in Indonesia. In other words, parts of areca palm which are frequently used so far are the leaves, stems and seeds [2].

Meanwhile, a part of areca palm that is areca coat nut is often wasted and its utilization is still low. Areca coat nut (Areca catechu L.) is included as waste containing cellulose. Laboratory analysis showed that the areca coat nut contains 70.2% cellulose content, 10.92% water, 6.02% ash. Given that fact that the current utilization of areca coat nut is still limited in number, so innovations are needed to take advantage of areca coat nut such as utilize it as an art paper material.

The potential pulp market in Indonesia is quite high and this can be seen from the pulp and paper industry which numbers keep increasing by time. Indonesia is in the 9th rank having 3.6% global capacity of pulp production after China, USA, Japan, Germany, Canada, Finland, Sweden, and South Korea. It can be seen from the number of pulp exports in 2010 which reached 2.6 million tons [3]. The pulp and paper industry in Indonesia is still far from the expectation, and the need of pulp for art paper in the country reaches 4 million tons/year [4].

Pulp is a paper slurry derived from the separation of cellulose fibers from lignin that comes from fibrous materials both wood and non-wood through some processing [5]. The purpose of the pulping process is to degrade lignin and to separate it from cellulose by minimizing the occurrence of cellulose damage. It can be concluded that the quality of the pulp is affected by the small amount of lignin present in the pulp, the smaller the lignin content in the pulp, the better the quality. The statement is also supported by some research on pulp stating that good pulp is the one that has the least lignin content because lignin will affect the physical properties and quality of the produced pulp [6].

In the pulping process, there are several methods of manufacture which are mechanically, chemically and semi-chemically. The optimal pulping process for non-timber plant fibers is the alkaline process using NaOH, NaOH solution can solubilize the lignin in the fibers. The chemical pulping process is carried out by developing a soda process, an alkaline cooking process with NaOH solution. NaOH solution can degrade fibrous bonds therefore the formation of pulp is accelerated [7]. In general, using NaOH results dark fiber, bleaching (brightening) is necessary to be done using H2O2 solution.

The presence of NaOH, both in the pulp and paper industry is one of the most important components. From the research conducted by Saenah [4], the best conditions of NaOH use of pulp and kenaf characteristics are 12% and 14%. In addition, a research by Onggo and Tri Astuti[8] stated that the effect of pulping process on the color of art paper from reedstakes 10% NaOH as optimal result. Based on the above research, appropriate use of NaOH may vary depending on the type of material. So in this study we will look for proper NaOH concentration to produce quality pulp with lignin content, expected cellulose from midrib and nipah leaf.

Research on areca fiber used as art paper is conducted in two stages. The first stage was to find out the proper concentration of NaOH and the quality of the pulp produced from the pin fiber which in the process is done chemically by the process of soda using NaOH solution. The best treatment in the first stage is used in the second stage of research, which was the manufacture of art paper from pulp of areca coat nut.

One of the interesting physical properties of paper is its tensile strength. The tensile resistance is defined as the paper's resistance to the tensile force acting on both ends of the path to break, expressed in units of force per unit of test width (kN/m)[9]. Tensile strength is important for assessing the strength of paper when stretched and subjected to force on each side other than that tensile strength plays a significant role in the formation of pulp into sheets of paper[10].

Research that involves treatment to produce the response requires a proper experimental design to produce research with reliable conclusions. There are three important elements in the Experiment Design that are treatment, environmental control and replication[11]. The repeat function in an experiment is to provide a means of estimating experimental errors (error = random = residual = error), reducing experimental or research errors and allowing for a more rigorous and precise estimation [12].

There are several formulas in calculating the number of replicates in the experimental design. This paper describes the comparative method of Federer and Gomez Methods in counting replications in the experimental treatment of adding NaOH concentration in pulp fiber manufacturing based on the results of the tests that have been done. The purpose of this comparative study was to find the number of replications with the smallest error for designing advanced research which is planned to use 8 treatments ie Newspapers: (Pulp of areca coat nut + NaOH) = 90: 10, Newspapers: (Pulp of areca coat nut + NaOH) = 80: 20, Newsprint paper: (Pulp of areca coat nut + NaOH) = 70: 30, Newsprint: (Pulp of areca coat nut + NaOH) = 60: 40, Cardboard Paper: (Pulp of areca coat nut + NaOH) = 90: 10, Cardboard: (Pulp of areca coat nut + + NaOH) = 80: 20, Cardboard: (Pulp Shell + Semi + NaOH) = 70: 30, Cardboard Paper: (Pulp of areca coat nut + NaOH) = 60:40. Before that, the data simulation using the result test of tensile strength from eight materials was processed (Table 1). Data simulation is done to estimate what method that produce smaller error.

2. Materials and Methods

This research was a preliminary research prior to the research on paper tensile strength test with 8 treatments. This study uses comparative studies to compare two methods of counting the number of replications. The bootstrap resampling method is used to simulate the error comparison obtained using Federer Method and Gomez Method. The data used for resampling is a paper tensile strength test data from paper with 6 treatments and 2 types of paper circulating on the market used as control.

2.1. The Federer Method

The number of samples according to Federer's formula[13] follows the following formula:

$$t(r - 1) > 15 \tag{1}$$

where t = number of treatments and r = number of replications

2.2. The Gomez Method

According to Gomez[14], Test F should be counted only if the degrees of error free are six or more. Where the degree of error in Complete Randomized Design is calculated by $t(r - 1)$.

3. Results and Discussion

The data used for the simulation is the data of tensile strength test of paper using from 6 types of pulp maker and two types of art paper as control. In tensile strength test of art paper, there are 6 samples tested that are, first, areca paper without additional filler with bleaching treatment using H_2O_2. Second, areca paper with 24 hour drying treatment. Third, betel paper plus re-use paper filler with 50:50 ratio. Fourth, areca paper with boiling material for 1 hour. Fifth, betel paper plus used cardboard filler with 50:50 ratio. Sixth, betel paper plus filler used newspaper with 50:50 ratio. All samples have been through cooking process for 120 minutes using 20 % NaOH. The sample used as a control is a white art paper from a straw obtained from one of the paper stores sold in Malang.

The tensile strength test process was conducted using Brazillian Test instrument. The testing process is carried out in the following steps: first, all the samples tested along with the control sample being tested are measured regarding its width respectively. Based on these measurements, the results obtained is that each paper has a length of 8 cm with a width of 4 cm, so a paper area was 32 cm^2 each. The second step was the measurement of the applied tensile force carried out using a micrometer tool. The tensile force was the maximum value on the micrometer reading of the dial when the paper is pulled until the sample is broken or torn at the time of the test in kilogram force. In Apriani study (2016) it is stated that the average yield of tensile strength of corn stem fiber art paper ranged between 0,213 - 0,554 (kgf/mm^2). The mean value of the highest tensile strength of 0.554 kgf/cm^2 was obtained from the composition of corn stalk raw materials and scrap paper with ratio : 25%:75% and cooking time of 180 minutes. The average value of the lowest tensile strength is 0.213 kgf/mm^2 on the composition of the raw material of corn and sracp paper with ratio 100%:0%, with a cooking time of 60 minutes. The higher the composition of corn stalks used in making art paper, the tensile strength of

the resulting paper tends to decrease. It is suspected that the lignin content of corn stalk pulp is higher compared to waste paper, resulting in the tensile strength of the paper being low. The data on paper tensile strength test in the preliminary study is presented in Table 1, where each ingredient is added NaOH with a concentration of 20% based on material weight.

Table 1. Paper Tensile Strength Testing**.**

Material	Cross-sectional area (cm^2)	F (kgf)	P (F/A) (kgf.cm^{-2})
Bleached areca nut paper	32	2.2	0.069
Boiled dry areca nut without glue	32	0.4	0.013
Material	Cross-sectional area (cm^2)	F (kgf)	P (F/A) (kgf.cm^{-2})
50% re-use: 50% areca nut	32	1.3	0.041
Boiled areca nut (for 1 hour)	32	1.4	0.044
50% Cardboard : 50% areca nut	32	3.7	0.116
50% newspaper : 50% areca nut	32	1.2	0.038
White paper	32	1.3	0.041
Red paper	32	12.3	0.384

Data P in the column used for simulation. Data on resampling results with eight treatments are presented in Table 2 and Table 3.

Table 2. Result of resampling using 3 replications.

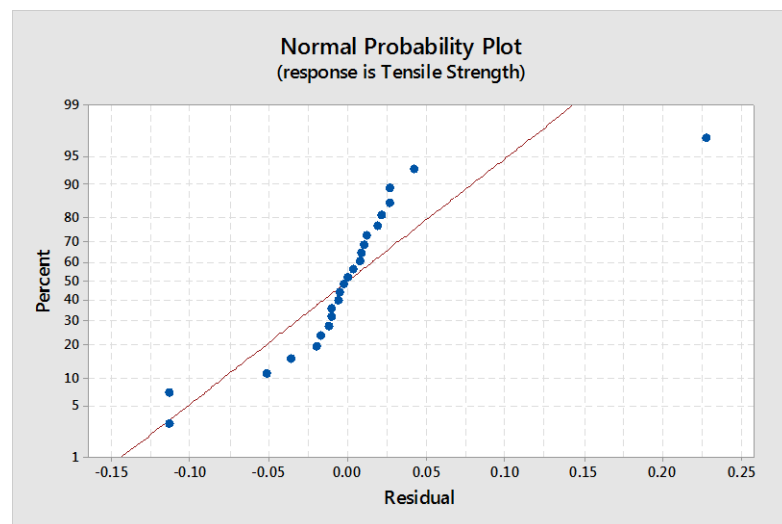| Replication	Treatment							
	T1	T2	T3	T4	T5	T6	T7	T8
1	0.013	0.013	0.041	0.116	0.038	0.384	0.013	0.069
2	0.013	0.038	0.038	0.038	0.069	0.044	0.044	0.038
3	0.044	0.041	0.044	0.116	0.044	0.044	0.041	0.116

Figure 1. Normal Probability Plot of Residual Using 3 Replication

Table 3. Result of resampling using 2 replications

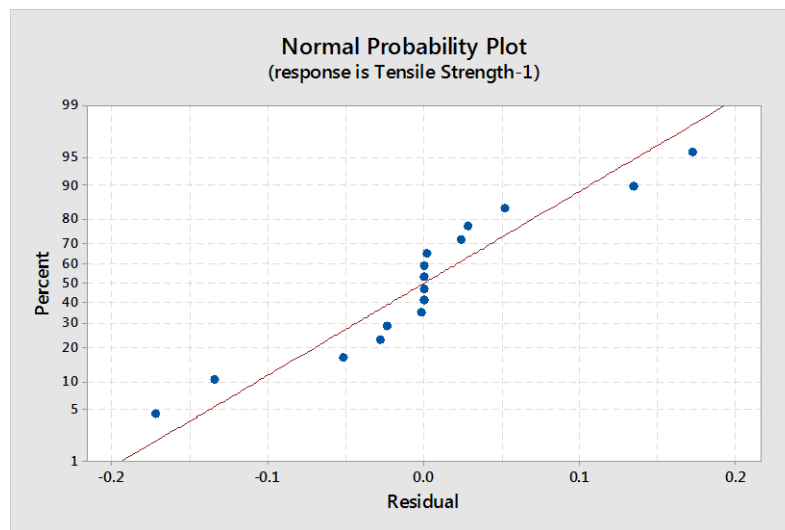| Replication	Treatment							
	T1	T2	T3	T4	T5	T6	T7	T8
1	0.116	0.041	0.013	0.038	0.384	0.069	0.384	0.116
2	0.069	0.041	0.116	0.041	0.384	0.013	0.041	0.384

Figure 2. Normal Probability Plot of Residual Using 2 Replication

3.1 The Federer Method
In the follow-up study, there are 8 treatments planned so that according to the recalculation formula using Federer method, at least 3 replications are obtained. Analysis of variance shows that sum square error of 0.08678 was obtained.

3.2 Gomez Method
Using the Gomez method, the replication can be reduced in number because the recalculating formula in the Gomez method requires $t(r-1) \geq 6$, so it can be determined that using 2 replications on each treatment is still valid to do. Analysis of Variance shows that the sum square error of 0.1032 was obtained.

From the results it is obtained that the higher the number of replications then the error becomes smaller. This is consistent as stated by Montgomery that the replication is used to minimize the error [2]. The researcher can adjust the recalculation method using various considerations. For example in terms of price of raw materials in making the sample, the length of the production process, and so forth. But the researchers have to note that when replication is taken with the extra-large number, the error will bigger than the reasonable of replication [15].

4. Conclusion
The Gomez method calculates fewer replications than the Federer method. Error produced using 3 and 2 replicates were 0.0876 and 0.1032, respectively. The next study is focusing on the amount of replication to minimize the error.

References
[1] Pertanian dan Perkebunan Indonesia. Retrieved from https://id.wikipedia.org/wiki/Pertanian_dan_Perkebunan_di_Indonesia. [Accessed on 6 December 2017]
[2] Balai Penelitian Tanaman Palma 2010 Prospek Pengembangan Tanaman Pinang Sulawesi
[3] Pusat Kebijakan Perdagangan Luar Negeri Badan Pengkajian dan Pengembangan Kebijakan Perdagangan 2015 Indonesia Export Target Analysis (In Indonesia) Kementrian Perdagangan. Jakarta
[4] Endah A 2016 The Influence of Raw Material Composition and Length of Cooking Time on Drag Strength on Art Paper Making from Corn Stover and Used Paper Waste (In Indonesia)

J.Mekanika dan Sist.Termal **1 2** 38-42

[5] Saenah E 2002 Effect of Soda Dose on Abaca Pulp and Pulp Kenaf Pulping Soda-Antaquinon Characteristics (In Indonesia) Skripsi Jurusan Kimia FMIPA Universitas Brawijaya Malang

[6] Fengel D dan Wegener 1995 *Wood, Ultrastructure Chemistry and Reactio*ns (In Indonesia) Penerjemah H. Sastrohamidjojo Gajah Mada University Press. Yogyakarta

[7] Malo B A 2004 *Membuat Kertas Seni dari Pelepah Pisang* (Making Art Paper from Banana Bark) (In Indonesia) Kanisius Yogyakarta

[8] Onggo H dan Tri Astuti J 2004 Effects of Sodium Hydroxide and Hydrogen Peroxide Against Rendeman and Pulp Color from Leaf Fiber (In Indonesia) *J. Tolok Ukur Pemanfaatan Serat Alam Bagian Proyek Penelitian dan Pengembangan Otomotif, Transportasi, dan Energi LIPI*

[9] Giancoli D 2000 *Physics for Scientists & Engineers Third Edition* Upper Saddle River: Prentice Hall

[10] E J Pavlina and C J Van Tyne 2008 Correlation of Yield Strength and Tensile Strength with Hardness for Steels *J. of Mat. Eng. and Perform* **17** 6

[11] Component of an Experimental Study Design. Retrieved from https://stats.libretexts.org/Core/Experimental_Design/Analysis_of_Variance/Components_of_an_experimental_study_design. [Accessed on 6 December 2017]

[12] Montgomery C D 2002 *Design and Analysis of Experiment* John Wiley and Son, Inc United State of America

[13] Federer W T 1967 *Experimental Design, Theory and Application* Oxford and IBH Publ. Co New Delhi

[14] Gomez and Gomez 1995 *Statistical Procedures for Agricultural Research* John Wiley and Son, Inc United State of America

[15] Ihwah A 2012 Bootstrap in Censored Data to Estimate The Regression Coefficient on The Cox Regression Models (In Indonesia) Thesis of Postgraduate in Mathematics Gadjah Mada University Yogyakarta