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# Trial-manufactured of paper like washi by reusing wood and plant waste

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**Abstract:** To make paper from non-wood fiber is important because of protection of forest resources environmentally. Moreover, if paper can be made from familiar plants, it will be useful for both effective utilization of resource and environmental education. In this research, corn shuck and leaf, and half-split chopsticks were soaked into the bleaching agent on market in Japan, with changing concentration of the agent and soaking time, and pulp was manufactured. Relations between the quantity of the agent and the soaking time, or pulp yield were examined. For corn shuck and leaf, the yield decreased linearly with increase of the agent or soaking time, in general. But, for the case of applying much shuck, the relation showed maximum curve. If the agent was very less, paper had yellowish. If the agent was increased, the paper became whiter, but orange tint slightly. Compared with papers on market, created papers had smaller apparent specific gravity and air resistance, and were thicker. The sensory test was conducted using both of created papers and papers on market. Homogeneity impression was associated with many impression factors. Papers which were judged as homogeneous had impressions of high intensity, high artificial degree and not-warmth. Papers with heterogeneous or rough impression brought high ventilation impression for subjects. Individual differences of impression points relevant to visual and tactile were apparently smaller than those which might be regarded as physical properties, in general.

**Keywords:** Paper-making, Half-split chopsticks, Corn shuck and leaf, Japanese paper, Sensory test.

## 1. INTRODUCTION

For use as raw material of bioethanol, demand of corn is growing. Corn shuck, leaf and stem are provided in large quantities after crop of corn. The effective utilization of them has big significance.

Production of paper is considered as an example of the effective utilization. Although such researches are not enough, paper may be made from corn waste. Almost of paper is made from wood nowadays. Since non-wood fiber has low quality and is difficult to obtain stably, paper is rarely made from non-wood fiber industrially. For the purpose of effective utilization of familiar plants, papers made from bamboo grass and butterbur are sold only a little quantity as local products, in Japan. The production of industrial paper which used bamboo as raw materials has been carried out for an effective utilization of local resources (Kawata, 2009).

In Japan, paper-making is often conducted in school education for environment. For example, paper was produced experimentally by using shredder wastepaper, used milk pack and half-split chopsticks, for the purpose of learning about reuse of resources and reduction of waste through paper-making. In addition, paper-making from kenaf (Masuo, Tsuchiya & Shimizu, 1990), butterbur (Takada, Han, Nakata & Nakamura, 2007), stem/ leaf of potato and bark of Japanese cypress had also been attempted. In many of these studies, raw materials were boiled with sodium hydroxide and were pulped. In several studies, particular bleaching agent on market was used for pulpifcation, because it was easy to obtain. Components of the bleaching agent are sodium hydroxide and sodium hypochlorite. However, in any studies, relations of pulp production conditions and pulp yield, or paper quality were not examined enough.

To produce pulp from plant and wood waste, corn shuck/ corn leaf/ half-split chopsticks was soaked in the bleaching agent under various conditions in this study. Then, relationships between the yield and pulping conditions were examined. For papers created and those on market, compression properties, air resistance and hue were measured. The sensory test of papers was also made. Relationships between each impression factor were examined.

## 2. EXPERIMENT

### 2.1. Pulp production

By soaking raw materials into the mixed-solution of the bleaching agent and water under room temperature, we examined whether these became pulp. Dry shuck of corn/ dry leaf of corn/ half-split chopsticks (White birch) in air-dry condition was used for the material. The bleaching agent concentration in the mixed-solution and time of soaking in the solution were changed. In the exploratory experiment, high concentration of the agent resulted in high pulping efficiency if quantity of the agent was constant. But compared with the influence of quantity of the agent, influence of the agent concentration was small. Pulping conditions in this research are shown in Table 1. Raw materials were soaked in the solution for the time which was shown in Table 1. Then, those were ground by mixer for 20 seconds. Pulp was obtained by filtering suspension with the net for catching goldfishes. Pulp was dried at 70 degrees Centigrade for 6 hours, and weight of pulp in oven-dry condition was measured. In each condition, experiments were conducted three times. Yield was obtained by dividing weight of pulp in oven-dry condition by weight of raw material in oven-dry condition.

### 2.2. Paper-making

The silk screen (120 meshes) was pasted on the bottom of the wooden frame for paper-making. The internal dimensions of the frame were 10 cm by 15 cm. Air-dried pulp of 1.2 g was used so that basis weight of paper would be set to 80 g/m$^2$. The pulp were soaked in water for 12 hours or more, and was ground in mixer for 10 seconds to obtain suspension. Suspension was poured into

the frame, and pulp on the silk screen was tried to be uniform distribution. Finally, the layer of pulp was not compressed and was dried at room temperature.

### 2.3. Measurement of the physical properties of papers

The compression properties and air resistance of 23 papers (13 created papers and 10 commercial papers) were measured. Created papers with large deformation or big variation in thickness were not measured properties. Compression properties were measured with three places for each paper. Air resistance was measured at neighborhood of four corner of each paper. Compression properties were measured by KES-FB3 testing apparatuses (KATO TEKKO CO., LTD.). Load area and maximum stress were 2 $cm^2$ and 50 $gf/cm^2$ respectively. Immediately after reaching the maximum stress, load was removed. Those experimental measurements were carried out in the laboratory controlled at the standard condition of 20±2 degrees Centigrade and 65±5 %R.H.. Thicknesses were measured at compression stresses of 0.5 $gf/cm^2$ and 50 $gf/cm^2$, and were expressed as $T_0$ and $T_{50}$ respectively. Total work load (per unit area) until reaching maximum stress ($W$) was measured. Total work load (per unit area) of process removing load was also measured. The hysteresis ($H$) was evaluated by the percentage of the latter divided by the former. The reflectance to the light of 400 to 700 nm was measured. At five places, which were the central part and neighborhood of four corners, reflectance of each paper was measured. Reflectance was measured about both sides.

**Table 1**: Pulping conditions

| | Weight (g) | Bleaching agent and water (ml) | Soaking time |
|---|---|---|---|
| Half-split chopsticks | about 12.6 (3 pairs) | 150 and 0 | 7 days |
| | | 200 and 0 | 7 days |
| | | 250 and 0 | 7, 23 days |
| Corn shuck | 5 | 75 and 225 | 24 hrs |
| | | 100 and 200 | 24 hrs |
| | | 125 and 175 | 12, 18, 24 hrs |
| | | 150 and 150 | 24 hrs |
| | | 200 and 100 | 12, 18, 24 hrs |
| Corn leaf | 3, 5 | 100 and 300 | 24 hrs |
| | | 120 and 280 | 24 hrs |
| | | 150 and 250 | 24 hrs |
| | | 180 and 220 | 24 hrs |

**Table 2**: Papers used for the sensory test

| Created paper | Production conditions of pulp | | |
|---|---|---|---|
| | Weight (g) | Bleaching agent and water (ml) | Soaking time |
| Corn shuck | 5 | 125 and 175 | 18 hrs |
| Corn shuck | 5 | 200 and 100 | 12 hrs |
| Corn leaf | 3 | 180 and 220 | 24 hrs |
| Half-split chopsticks | about 12.6 | 250 and 0 | 7 days |
| Commercial paper | | | |
| Recycled paper (acid-free paper) | | | |
| "Minogami" (A kind of Japanese paper, which made from mulberry) | | | |
| Unbleached mulberry paper | | | |

### 2.4. The sensory test of sample papers

The sensory test was carried out by SD-method in January-February 2014. The questionnaire consisted of 14 impression items: uniformity/ softness/ whiteness/ weight/ yellowness/ warmness/ porosity/ roughness/ degree of shiny/ weakness/ degree of ventilation/ thickness/ newness/ artificial level. Subjects judged each paper's impression by visual and tactile, and selected corresponding grid from 7 grids between symmetric adjectives. Subjects were 29 college students, 19 female and 10 male. Average age was 21 years old. The papers applied in the test are shown in Table 2. Recycled paper has 70 % mixing rate of used paper, 72 % degree of whiteness and 65 g/m$^2$ basis weight. At first, every subjects evaluated the recycled paper with unaware that it was the recycled. Subjects evaluated other papers by random turn. Finally, subjects considered all the questionnaire items. If subjects felt the item as to be unsuitable for evaluation of paper, subjects reported. Validity of questionnaire items was inspected.

## 3. RESULTS AND DISCUSSION

### 3.1. Relations of the bleaching agent quantity and the yield

Pulp was not generated, if corn leaves of 5 g were soaked for 24 hours in the mixed solution including the bleaching agent of 100 ml or 120 ml. Long vegetable fibers like string were produced. Since pulp was not generated even if soaked in the mixture including the agent of 120 ml for 5 days, it turned out that the leaf of 5 g couldn't be decomposed by the agent of 120 ml or less. On the other hand, the leaf of 3 g was pulpified with the mixture including the bleaching agent of 100 ml or 120 ml. Corn shuck of 5 g was pulpified even with the mixture including the agent of 100 ml.

For the shuck of 5 g, the yield increased with increase of the agent at the range of 100 ~ 150 ml and became the maximum at 150 ml (Figure 1(a)). The reason of the increase is unknown, further research is necessary. The result of decrease of yield with the increase in the agent can be explained. If using much the agent, fiber will be decomposed much and will become short. Then short fiber will flow out on the occasion of filtration. For the leaves of 3 g, in the range of 100 ~ 180 ml, the yield decreased with increase of the agent. (Figure 1(b)). In the range of 150 ~ 180 ml, both the leaves of 3 g and 5 g became pulp, and the leaves of 5 g had slightly higher yield.

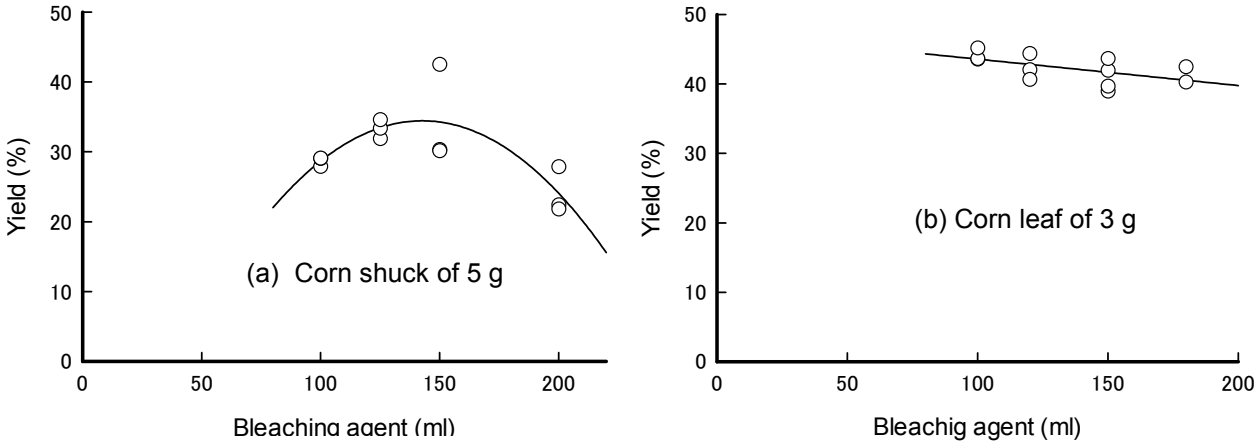


**Figure 1:** Relationships between the bleaching agent quantity and the yield

Notes: (a) $y = -0.0284x^2 + 2.71x - 29.9$, $R^2 = 0.612$
(b) $y = -0.152x + 47.4$, $R^2 = 0.317$

Half-split chopsticks were soaked in the bleaching agent for 7 days. The case of 250 ml bleaching agent, the chopsticks had become soft, and surface fiber could be peeled by hand. Even in this case, after crushing by mixer, wood chips were observed only a few. Also in the case of 200 ml, the chopsticks became also soft, but not as much as the case of 250 ml. After removal of wood chips from the pulp, oven-dry weight was measured and the yield was determined. The yield was increased with increase in the agent. The yield when raw material was soaked in the agent of 150 ml, 200 ml and 250 ml were 34.3 %, 39.8 % and 44.3 % respectively. This is because central portion of the chopsticks was not decomposed under few agents. The result will change if the chopsticks are peeled thinly before soaking. Since more than 70 % of wood is cellulose and hemicellulose, yield will rise by changing experimental conditions. The experiment of soaking in 250 ml agent for 23 days was also performed. The chopsticks of 23 days soaking became much softer than those of 7 days soaking, and was observed large bending by own weight. The yield was slightly increased, but was almost unchanged.

### 3.2. Relation between soaking time and the yield

For the shuck, the effect of soaking time was examined. In the range of 12 ~ 24 hours, the yield was decreased with increase of soaking time at the both condition of 125 ml and 200 ml（Figure 2）. Such a result might be caused by progress of fiber shortening.

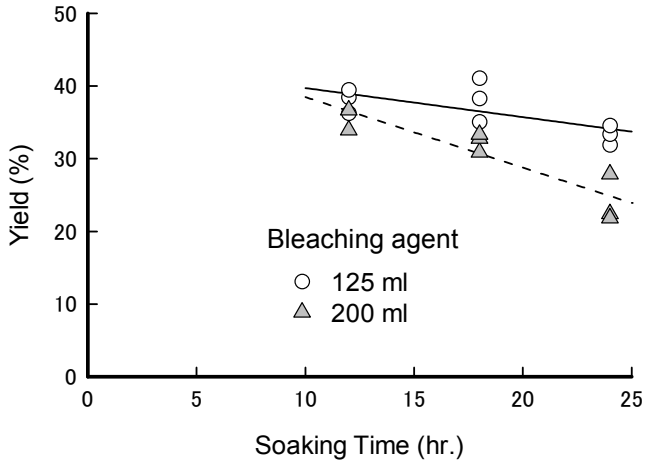


**Figure 2:** Relationships between the soaking time and the yield

Notes: Raw material is corn shuck of 5 g.
125ml: $y = -0.399x + 43.7$, $R^2 = 0.465$
200ml: $y = -0.972x + 48.2$, $R^2 = 0.797$

### 3.3. Physical properties of papers

The results of air resistance and compression properties are shown in Table 3 and Table 4 respectively. For created papers, average values were calculated according to raw material and shown in those tables. Three kinds of "Atsuyohsi" (thick Japanese paper) were measured and compared for their properties. Since differences among these papers were whether straw or hemp was included or not, differences were comparatively small. Therefore, averages of three "Atsuyohsi" were evaluated. The air resistance of various Japanese papers was too large to measure in this experiment. Apparent specific gravity and air resistance of created papers were smaller than those of commercial papers. The initial thickness $T_0$ of created papers were larger than those of commercial papers. In this research, after making paper, layer of pulp was not

compressed. Therefore, by compressing with appropriate level, these papers might be possible to approach properties of Japanese paper on market.

The difference of compression properties might be caused by fiber length. In most papers, the thickness at the maximum stress became 60 ~ 70 % of the thickness $T_0$. The value of Ganpi paper became 34 %. The reasons may be that the Ganpi paper is very thin and Ganpi fiber is shorter than that of hemp and mulberry. The papers which were made from the shuck (56 %) were compressed larger than the papers which were made from the leaf (72 %) and half-split chopsticks (67 %). The shuck's fiber was shorter than the leaf's fiber, which was evident from observation of fiber at edge of papers. The size of papers created from the shuck was changed readily by change in surrounding environment. Those reasons would also be same.

**Table 3**: Apparent specific gravity and air resistance

| Created paper | Apparent specific gravity ($\times 10^{-2}$) | Air resistance (kPa·s/m) |
|---|---|---|
| Corn shuck | 6.62 | 475 |
| Corn leaf | 6.12 | 366 |
| Half-split chopsticks | 8.95 | 700 |
| Commercial Japanese paper | | |
| Hemp paper | 38.4 | — |
| "Atsuyohsi" | 30.3 | — |
| "Minogami" | 15.0 | — |
| Mulberry paper | 21.9 | 739 |
| Unbleached mulberry paper | 23.9 | — |
| Handmade mulberry paper 1 | 22.1 | — |
| Handmade mulberry paper 2 | 15.1 | 1523 |
| Ganpi paper | 27.8 | — |

**Table 4**: Thickness and compression properties

| | $T_0$ (mm) | $T_{50}$ (mm) | $H$ (%) | $W$ (gf·cm/cm$^2$) |
|---|---|---|---|---|
| Created paper | | | | |
| Corn shuck | 1.16 | 0.651 | 34.9 | 0.558 |
| Corn leaf | 1.20 | 0.866 | 26.1 | 0.469 |
| Half-split chopsticks | 0.912 | 0.609 | 29.6 | 0.413 |
| Commercial Japanese paper | | | | |
| Hemp paper | 0.120 | 0.0807 | 20.8 | 0.0267 |
| "Atsuyohsi" | 0.313 | 0.221 | 41.8 | 0.0612 |
| "Minogami" | 0.364 | 0.204 | 31.9 | 0.120 |
| Mulberry paper | 0.116 | 0.0710 | — | 0.0407 |
| Unbleached mulberry paper | 0.168 | 0.0997 | — | 0.0460 |
| Handmade mulberry paper 1 | 0.214 | 0.123 | 32.0 | 0.0643 |
| Handmade mulberry paper 2 | 0.141 | 0.0690 | 21.8 | 0.0403 |
| Ganpi paper | 0.0383 | 0.0130 | — | 0.0217 |

When the shuck was soaked in 125 ml or 150 ml of the agent, created paper had less absorption at short-wavelength light, and had yellowish. Papers of 125 ml had stronger yellow tint than the papers of 150 ml. The papers of 200 ml had slightly less absorption at 460 ~ 480 nm light, compared with light of other wavelength. Papers had slightly orange tint.

The difference of papers of 18 hours soaking and 24 hours soaking was small, but papers of 12 hours soaking had less absorption at low wave-length. It means that these papers had yellowish.

Difference in reflectance of the both sides was small.

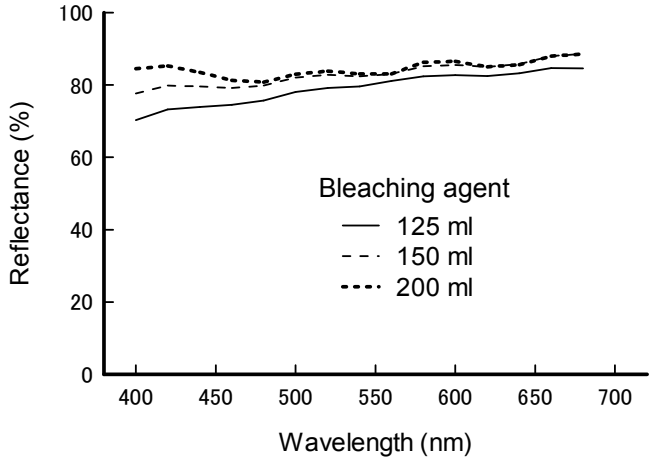


**Figure 3**: Reflectance of papers

Note: Raw material is corn shuck of 5 g. Soaking time is 24 hrs.

### 3.4. The sensory test of sample papers

High ventilation can say as a feature of Japanese paper, which suits to Japan's hot and humid climate. Slightly bright parts and slightly gloomy parts will be made if light passes through Japanese paper. Both of these was resulted from structural characteristics of Japanese paper as heterogeneous and porous, which are caused by Japanese paper fiber being long. Difference in brightness intensity induces fluctuation of light and brings comfort. In order to obtain fundamental data in examining such characteristics of Japanese paper, impression factors related to impression of heterogeneity/ high ventilation/ warmth were examined. Coefficients of correlation between each rating impression points were calculated.

If the paper was judged to be heterogeneous or rough by subjects, subjects felt the paper had high ventilation (Figure 4(a)). When a paper was evaluated as having few pores, it was estimated as low ventilation. Impression point of ventilation and air resistance measured was independent.

The impression point of heterogeneity was associated with many other impression points. If subjects felt a paper as porosity or rough surface, the paper was evaluated to be heterogeneous. If subjects felt a paper as heterogeneity, the paper was evaluated as natural. Thick papers were evaluated to be heterogeneous.

Surface smoothness has effect on ease of writing and visual evaluation of paper. It is important to examine what bring impression of smoothness. Smoothness impression was proportional to impression of homogeneity, and inversely proportional to impression of porosity, ventilation and thickness (Figure 4(b)).

If subjects estimated a paper had high homogeneity or high degree of artificial, the paper was estimated as low degree of warmth (Figure 4(c)).

Impression point of artificial level was proportional to the point of newness. If subjects felt a paper was white or less yellowish, subjects felt the papers was new (Figure 4(d)).

For the impression points of not-warmth and artificial level, created papers was evaluated to be similar to unbleached mulberry paper. For these points, seven papers shown in Table 2 had large difference, and could be divided into two groups. The one was consisted of “Minogami” and recycled paper, and these averages were 5.1 (not-warmth) and 5.3 (artificial level). Average points of other one were 3.2 and 3.0 respectively.

Ventilation degree and rough points had much difference among papers. The average ventilation points were 2.7 to 5.2, and these average was 4.0. The average rough points were 2.8 to 6.6 (the mean was 5.0). The difference in not-heavy impression point was very small (average points were 4.8 to 5.2), though the apparent specific gravities were much different. It was because subjects compared paper with other materials and made absolute evaluation.

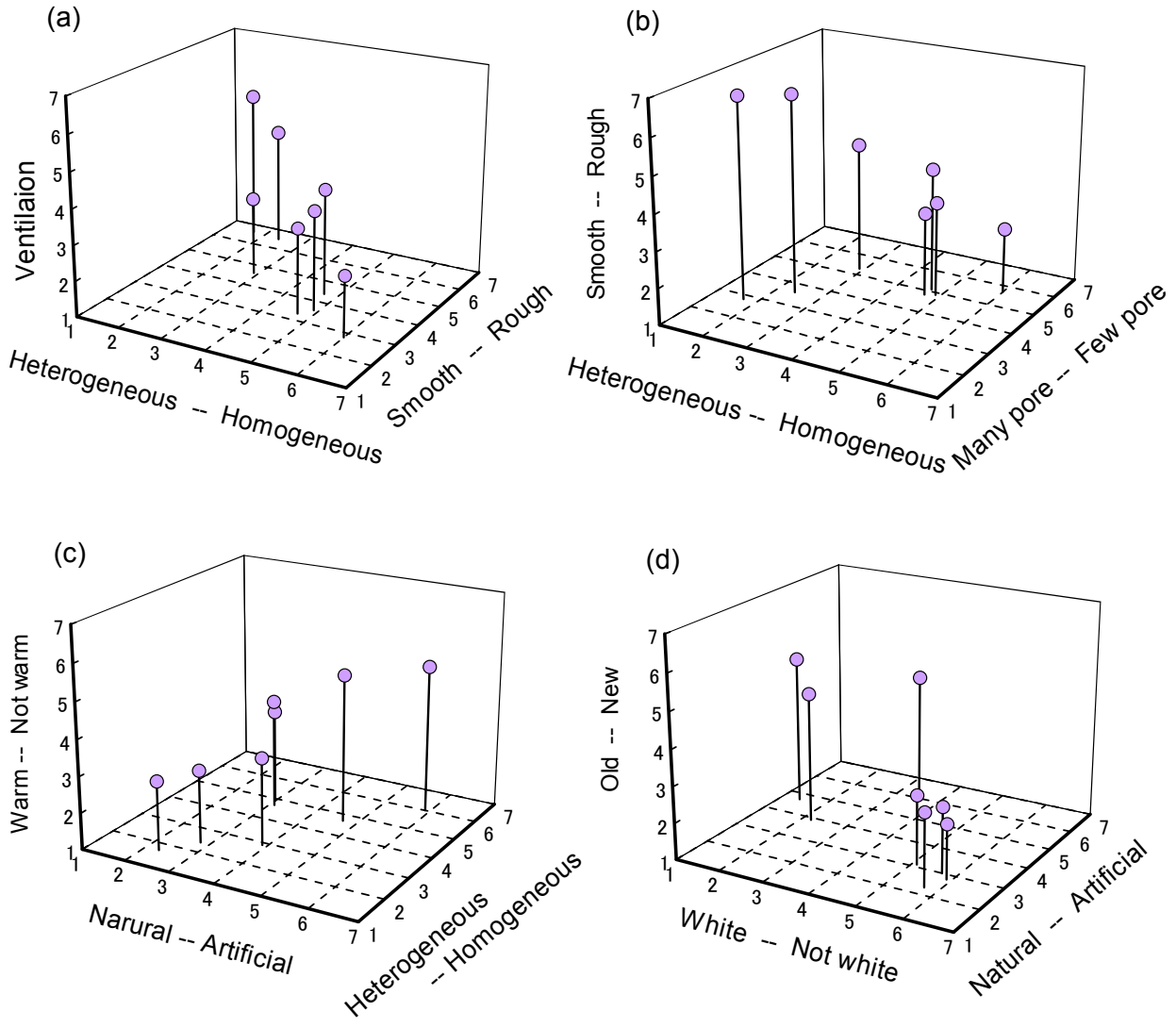


**Figure 4**: Relations of each impression point

To examine difference in the impression points among the subjects, standard deviation of the impression points was noted. Individual differences of the points related with visual and tactile were generally small in this study. For yellowness, individual difference was especially small (Figure 5). For 4 papers of 7 papers, the standard deviations were 1.0 or less. The average value of the 7 standard deviations was 1.0. The differences of whiteness, uniformity and roughness points were also small, the averages of the 7 standard deviations were 1.2, 1.2 and 1.3 respectively. But individual difference of judgment criteria of shiny degree was larger than those of color, uniformity and roughness. The average of the 7 standard deviations was 1.7, which was relatively large.

Impression points of items, which subjects might feel those as physical properties, had larger individual differences than those which might be evaluated by visual and tactile. Many of the standard deviations about judgment of warmness, softness and weakness were 1.6 or more.

For color, uniformity and roughness, individual difference had large variation among the papers, compared with other impression items. The standard deviation of the individual difference of 7 papers was noted. For color, uniformity and roughness, the standard deviations were 0.35 to 0.56. For other items, those were 0.084 to 0.31. The individual difference for certain paper was not always large or small.

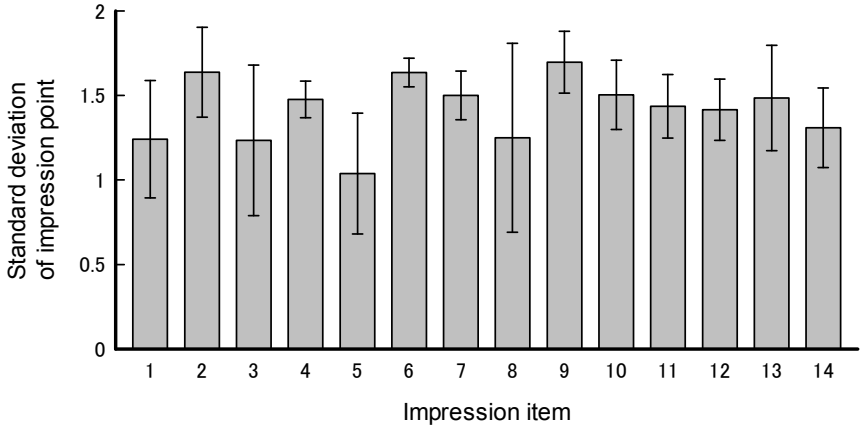


**Figure 5**: Individual difference of impression point

Legend: 1: uniformity, 2: softness, 3: whiteness, 4: weight, 5: yellowness, 6: warmness, 7: porosity, 8: roughness, 9: degree of shiny, 10: weakness, 11: degree of ventilation, 12: thickness, 13: newness, 14: artificial level

Note: Vertical bars and horizontal lines indicate averages of 7 standard deviations (individual difference) and standard deviation of those respectively.

## 4. CONCLUSION

By the simple method which raw material was soaked in commercial bleaching agent, shuck of corn/ leaf of corn/ half-split chopsticks was pulped. The quantity of the agent and soaking time were changed. The influences of these to pulp yield or paper properties were investigated. Moreover, the sensory test was conducted using both of created papers and commercial papers. The factors which gave the impression of high ventilation/ smoothness/ warmth/ etc. were examined. Results as follows:

The shuck was easy to become pulp than the leaf, but papers made from the shuck had insufficiently dimensional stability. For pulping the leaf of 5 g, the agent of 150 ml was needed at

least. On the other hand, the shuck of 5 g was pulped by the agent of 100 ml. In general, with increase of the agent or soaking time, the yield decreased. However, using less the agent, pulp generated had yellowish tint.

Impression of homogeneity was related to many other impressions. If homogeneity of paper was felt high by subjects, artificial impression was brought to subjects. Artificial impression indisputably brought not-warmth impression. Homogeneous paper gave rise to smooth impression. The impression of ventilation was influenced by surface appearance impression. Few pore/ smooth feeling resulted in low ventilation feeling.

Individual differences of impression points relevant to visual and tactile were apparently smaller than those which might be regarded as physical properties, in general.

These results will be big contribution, for the production of paper in environmental education. Outcome of this study shall contribute much to the environmental use of plant waste. By enhancing this study further, and finding the factors that determine the impression of paper, important data will be provided to create human-friendly paper.

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## BIOGRAPHY

Dr. Satoru Ohya has belonged to Hokkaido University of Education (1996- at present), and has been associate professor since 2001. His research areas in scientific achievement are a) wood slicing, b) strength of wood jointing and c) development of school education using wood. The number of his published original papers and articles is around 30.

Dr. Takako Fujimoto belonged to 1) Niigata University (1977-1981), and then has belonged to 2) Hokkaido University of Education (1981- at present) and has been professor since 1992. She has been i) Head, Japan Section of The Textile Institute of UK (2005- ), ii) a member of council of TI, iii) a council member of Japan Research Association for Textile End-uses, iv) a visiting professor, University of New South Wales, Australia (1996.9-1997.3). Her major research areas in scientific achievement are a) theory of heat transfer of fibrous materials, b) mechanical properties and handle estimation of clothing fabrics, c) objective evaluation of clothing materials and d) durability of clothing materials. The number of her published original books is 5, of her published original papers and articles is around 130. She received three prizes and awards related to the above scientific achievements.

Kento Tokumitsu is senior student of Hokkaido University of Education, and has been studying to become a science teacher, and has made study of making of paper from last year.