A New Pulverized Biomass Utilization Technology

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INTRODUCTION

Of recent years, the utilization of unused wood biomass was attracted attention as the new environmental clean energy for the development of resource cycling society in Japan, because the wood biomass was carbon-neutral and renewable resource. The unused or unwanted wood biomass, such as thinned wood, pruning wood and architectural waste, was miscellaneous. Generally, the wood biomass was widely distributed, and it was difficult to collect large amount from wide area and it was difficult to utilize at last. Therefore, the wood biomass was usually disposed in a small incinerator or dumped to a landfill, it has not been considered as the useful resource by now.

There have been lots of studies and developments regarding the new utilization process of wood biomass, but the technological or economical issues were still remained for the commercialization. For effective miscellaneous and small amount wood biomass utilization, the biomass had to be available as the value-added material. Adding new value to the miscellaneous wood biomass, an efficient utilization process, such as power generation by a gasification or material recycling by liquefaction have been considered. However, the efficiency of the process was not enough for a practical application. To improve the efficiency of process or the yield of products, a pretreatment of wood biomass was very important before those processes were conducted practically. The pulverization was thought to be the one of the solutions to improve the efficiency of the process or the wood biomass quality as raw material. In other word, the pulverization of wood biomass was very important factor to improve the combustibility, reactivity, feeding ability and handling ability, and it can lead the high efficiency of the utilization process.

In this paper, the study on the efficient utilization of pulverized wood biomass powder was discussed. New pulverization technique by a vibration mill was introduced firstly, and the application process with this pulverized powder was considered secondary. The pulverized wood power was employed for the gasification process and the liquefaction process as the starting raw material. In the gasification process, this wood powder was used as the gasification fuel in a direct injection high temperature gasification. In the liquefaction process, the wood powder was used as the raw material of the useful wood components extraction. In both process, the high performance of the process was promised by using the wood powder. Finally, total system of the wood biomass utilization has considered for the commercialization of the biomass utilization process. The total system was the combination between gasification, liquefaction and the other application process.

On this study, the pulverization technique for the pretreatment, the gasification and liquefaction process as the application process of wood powder was discussed, and total wood biomass utilization system were proposed.

1. New pulverization technology for wood biomass

For the effective wood biomass utilization, the pulverization was very important. The quality and quantity of pulverization wood biomass as the products was also so important that the effective pulverization of wood biomass was absolutely imperative. The mass wood powder production at low cost was the key technology to create new biomass utilization applications and new markets.

There were several conventional pulverization techniques such as the cutting method, mechanical shock method and grinding method. However, wood biomass, which was consisted of cellulose, hemi cellulose and lignin, was too strong to pulverize micron order particle in large quantity at low costs by those conventional pulverization techniques. New pulverization techniques such as a freezing pulverization or burst pulverization were developed in these days, but economical and practical issues were still remained for the commercialization.

In this paper, the pulverization of wood biomass has been conducted by using a vibration mill equipped rod or ball with the aim of effective pulverization of wood biomass. One of the results obtained from pulverization experiment was introduced.

Figure 1 shows schematic diagram of a vibration mill for the pulverization of wood biomass. This mill consisted of two pulverization jacketed vessels and vibration motor.

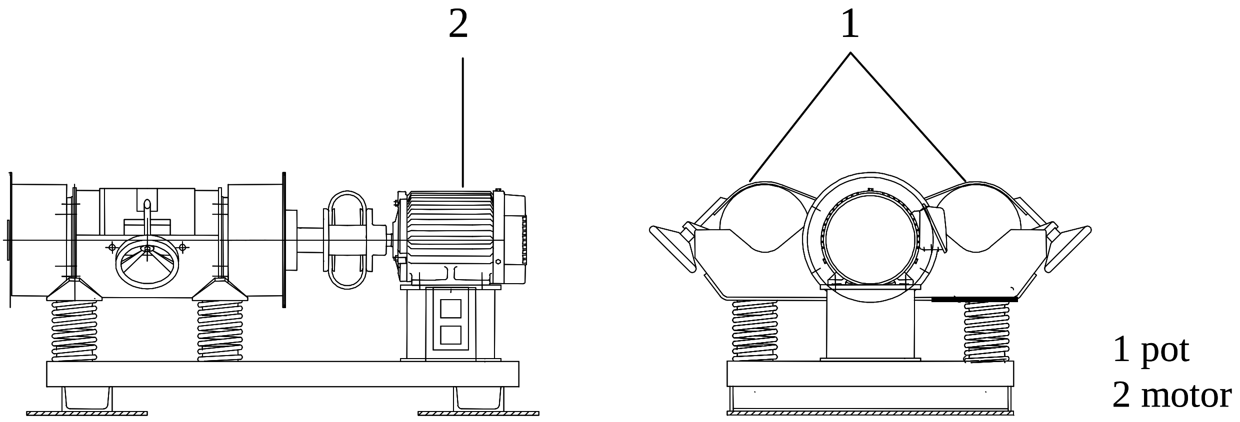

Figure 1 schematic diagram of the vibration mill

The volumetric capacity of vessel was 3.6 l, and the inside of vessel was reinforced with the alumina lining. To keep the temperature constant of the pulverized material in the vessel around 300 K, the generated heat by the pulverization process was taken by a water cooling system. The rotation frequency and vibration amplitude of mill was controlled by the vibration motor, which were 1200 rpm and 9 mm respectively. As the pulverization media, both alumina ball and alumina rod was employed. The ball size was 20 mm and the weight was around 18 g / each. The rod was 30 mm in a diameter and the weight was around 1200 kg / each. The impact power generated by the mill was calculated from centrifugal effect, which was the relationship between rotation frequency and vibration amplitude, as following equation;

$$K = {r\omega^2}/{g}$$

Where, *K* [G] is centrifugal effect, *r* [cm] is vibration amplitude, ω [radian / s] is angular rate, and *g* [cm / s^2] is gravity acceleration. In this equipment, the impact power was around 6.5 G. The pulverization strength was calculated by multiplying the impact power and the pulverization media weight. Since the rod weight was around 65 times heavier than that of ball, the pulverization strength of rod was bigger than that of the ball. But, the number of impact by the ball was much larger than that of the rot. Although the vibration mill was one of mechanical shock pulverization method, biomass could be pulverized not only by the impact power of mechanical shock, but also by the shear stress of friction. The biomass could be broken completely by multiplying the impact power and shear stress.

Norway spruce tree shaving dust was (a few millimeters in the thickness and a few centimeters in the length) used as a pulverization material in this experiment. Before the pulverization, the materials were dried in the sun for several days or in an oven for 24 hours at the temperature of 483 K. The moisture content was around 10 % or 3 % respectively after the drying. 200 g of dried material was filled in the vessel and pulverized. As the basic pulverization sequence, firstly the rods were filled 60 % of the vessel (11 rods) and pulverized. After the pulverization by the rods, the rods were replaced the balls and pulverized again. The balls were filled 80 % of the vessel (410 balls). The pulverization conditions were summarized in **Table 1**.

Table 1 Pulverized Condition

Run number	*Pulverized material*	*Moisture content*	*Pulverization media*	*Pulverization time*
1	Norway spruce	3 %	Rod	30 min
2				60 min
3				120 min
4			Rod + Ball	30 min+30 min
5		10 %		
6			Cutter	60 min

The characteristic of pulverized particle was measured and evaluated by the particle diameter, particle shape and aspect ratio. The particle diameter was measured by a laser diffraction /dispersion particle diameter distribution analyzer (Shimazu Co Ltd.). For the particle shape observation, a SEM (Scanning electron microscope) (JASCO Co. Ltd) was used. The photographic image method by a CCD camera and microscope was employed for the measurement of aspect ratio. The characteristic of wood powder, which was pulverized by a cutter mill, was also evaluated for the comparison. The cutter mill (Ikemoto Rika Co. Ltd) was wiley type equipped 4 cutters. The pulverized conditions of cutter mill were also summarized in Table 1.

1.1 The particle diameter and specific surface area of pulverized powder

Figure 2 shows cumulative particle diameter distribution of pulverized biomass by the vibration mill. Both rod and ball were used for the pulverization. After the 30 min of rod pulverization, shaving dust (moisture content 3 %) was broken into small particle and the 50 % particle diameter was around 30 µm. However, the 50 % particle diameter of 60 min or 120 min of rod pulverization was increased to around 35 µm or 45 µm, respectively, by the aggregation effect. On the other hand, the 50 % diameter pulverized by ball after 30 min rod pulverization was progressed in 24 µm. Although the impact strength of ball was not strong, the particle dispersion was better than that of rod. Therefore, the pulverization progressed without the aggregation. Considering the optimum combination of rod and ball was very important for the effective pulverization in the relationship of impact strength, aggregation and particle dispersion.

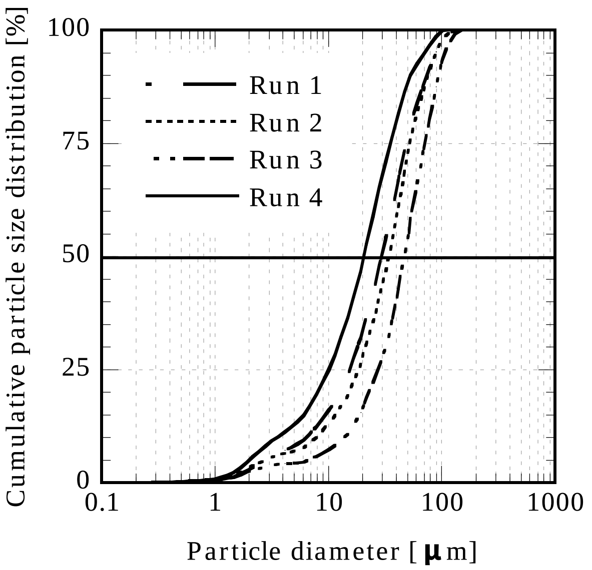

Figure 2 Cumulative particle diameter distribution of pulverized biomass

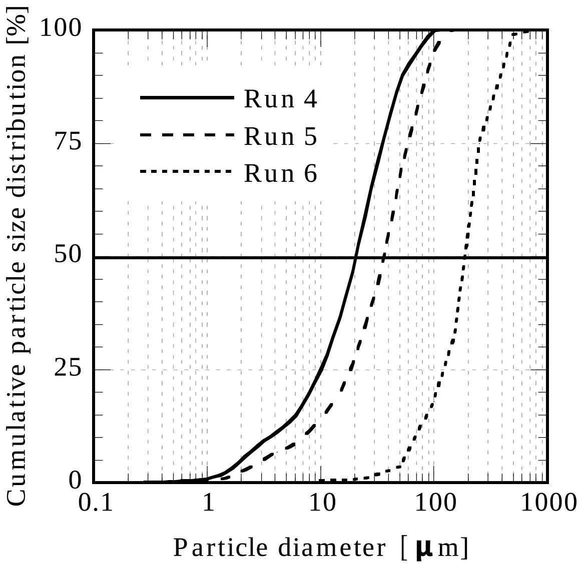

Figure 3 Cumulative particle diameter distribution of pulverized biomass

The moisture content was also a big factor for the biomass pulverization. The particle diameter distribution of pulverized biomass by the vibration mill and cutter mill was shown in **Figure 3**. The 10 % and 3 % moisture content biomass was used for the pulverization material. The 50 % diameter of 10 % moisture content biomass was 38 µm. The particle dispersion was forbidden, but the aggregation was encouraged by the moisture. In contrast, the cutter mill can not be pulverized as small particle as the vibration mill. The 50 % diameter was around 200 µm, and the particle distribution was very wide.

By the combination of ball and rod, the wood biomass could be pulverized into fine powder easily. Since the size of pulverized powder could be controlled by changing the pulverization media or moisture content, the combustibility and reactivity of pulverized powder could be control.

1.2 Particle Shape and Aspect ratio of pulverized powder

The particle shape and aspect ratio of pulverized particle was very import characteristics for the evaluation of feeding ability and handling ability. If the particle shape was irregular and the aspect ratio was big, the feeding ability and handling ability was low. In a gasification process, smooth feeding without blockages and high reactivity was the most important aspect to be considered. In the liquefaction process, highly dispersion in slurry and high extractability was also important factor for the high performance.

The SEM pictures of typical pulverized particle by the vibration mill and cutter mill are shown in **Figures 4** and **5**, respectively. The shape of pulverization particle by the vibration mill was almost round, and the surface of particle was smooth. It was also highly uniform in each shape. The biomass fiber was totally broken by the strong mechanical impact of the rod or ball, and the edge of particle was round off. On the other hand, the particle shape pulverized by the cutter mill was rectangle. The shape of the particle was irregular, and the fibers could be still observed. The both end of particle had well-regulated rectangle shape. Since the wood fiber was strong along the fiber, it was very difficult to pulverize in uniform shape by the cutter mill. For the comparison, **Figure 6** shows the particle shape pulverized by a grinder. The shape was totally irregular and the wood fiber was branch off at the end of each particle.

The aspect ratio distribution of pulverized particle was shown in **Figure 7**. Aspect ratio was defined as the ratio of long and short length of pulverized particle. The aspect ratio of 80 % of the pulverized particle by the vibration mill was less than 2.0. It indicated that the shape of the particle was close to sphere shape as in figure 4. In contrast, the aspect ratio of pulverized particle by the cutter mill was distributed very wide and 20 % of them were more than 5.0. The pulverized particle by the vibration mill was almost round shape, therefore the feeding ability and handling ability was though to be much higher than that by the conventional pulverization equipment.

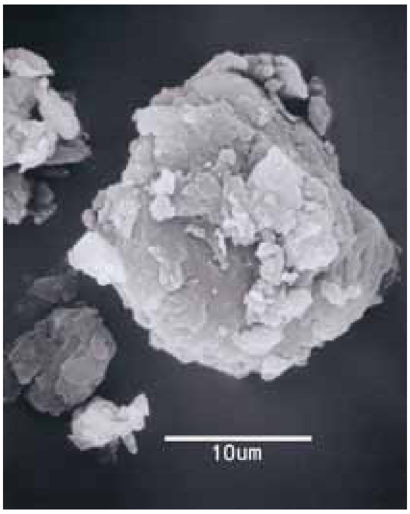

Figure 4 SEM Picture of Run 4 (Vibration mill)

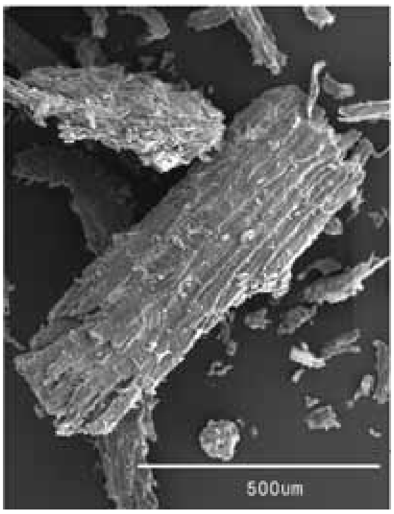

Figure 5 SEM Picture of Run 6 (Cutter mill)

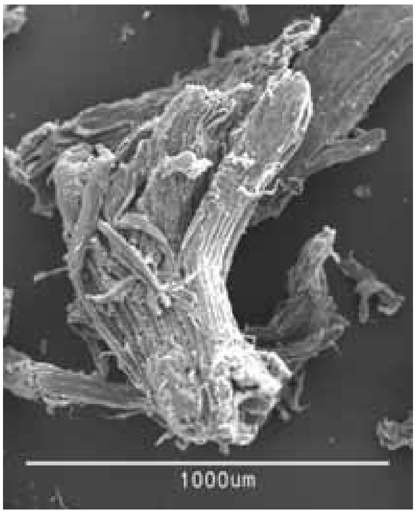

Figure 6 SEM Picture (Grinder mill)

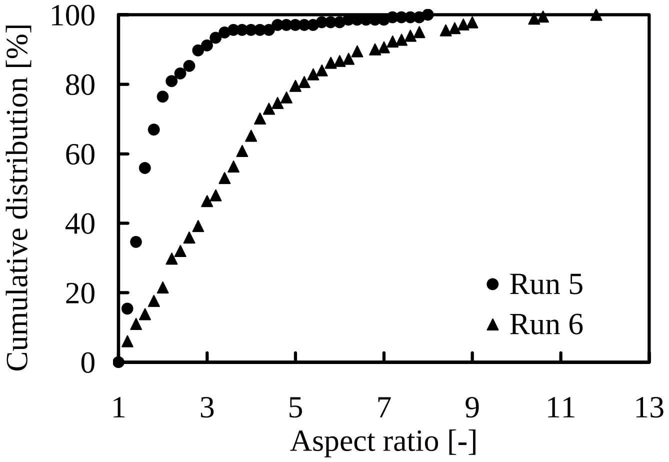

Figure 7 Aspect ratio distribution of pulverized particle

2. High temperature gasification with pulverized wood biomass

The wood biomass gasification was one of the thermal recycle processes, and this process was also one of the promising technologies for the efficient wood biomass utilization. There was several gasification processes proposed, which were equipped a fluidized bed gasifier or rotary kiln gasifier. However, these conventional gasification processes have not been used as a practical application by now, because of a crucial technological issue. The technological issue was tar components problem, which might be caused of choking or erosion of the gasification system. To clear up the tar trouble, new gasification technique for wood biomass was introduced. The new system was the high temperature gasification process in entrained down flow furnace, which was able to control the tar components problem. This process was the gasification used the high reactivity micron-order wood powder as fuel to create the high temperature (over 1300K) in the very simple down flow furnace. Creating this high temperature reaction zone, the wood biomass was almost completely decomposed into gaseous components, such as the hydrogen, carbon monoxide, carbon dioxide and a small amount of ash without yield of tar components. It was very clear that the technological issues regarding tar components were all fixed by the high temperature gasification process.

In this paper, one of the gasification experiments with the pulverized wood biomass was introduced and the gasification performance was evaluated.

Figure 8 and **9** show the outline of the experimental setup and the structure of the gasification furnace, respectively. The setup consists of the sections of sample feed, the down flow gasification furnace, cooler, cyclone, bag filter, adsorption of contaminants and incinerator of gas product. The reaction room in the gasification furnace is 800 mm in diameter and 2000 mm in height. A main burner for sample fuel and two methane burners for heating up and ignition are assembled on the top of the furnace.

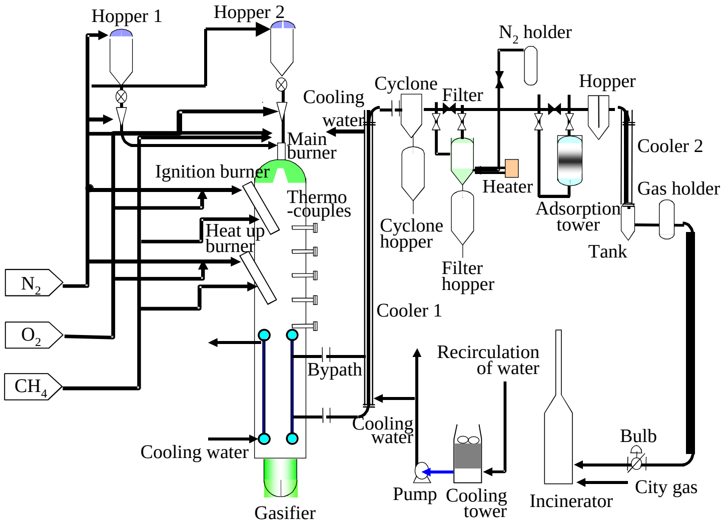

Fig.8 Organic waste materials gasification test unit

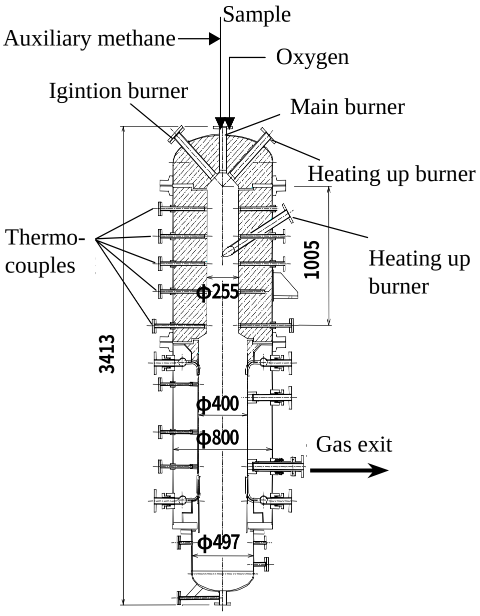

Figure9 Down flow furnace of gasification

The feed of sample started after the furnace was heated up to 1300 K by the methane combustion. The properties of the samples are listed in **Table 2**. The sample was carried pneumatically with nitrogen to the main burner through a tube of 4 mm. Oxygen or air was used for high temperature gasification of the sample by partial combustion. When heating by auxiliary combustion was added in the gasifier, methane was supplied with the sample to the main burner. Three components, CO, O_2 and CH_4 included in the gas products were monitored continuously by infrared gas analyzers. The composition of 10 different components, H_2, CO, CO_2, N_2, CH_4 and the other hydrocarbons in the gas was analyzed by the auto gas chromatography in every 6 minutes.

In the gasification examination, the supplying rate of auxiliary methane gas was controlled1.8 to 2.7 m^3/h, and the heating value of auxiliary methane was equivalent to about 1/10 to 1/7 of the sample fed. The experimental condition is summarized in **Table 3**. When the Air was used as the gasifying agent, the total amount of nitrogen was increased in 4 times, and then the temperature of the furnace was not able to keep over 1300 K. Therefore, the supplying rate of auxiliary methane was increased to maintain the high temperature of the furnace. Increased input heating value by increasing the supply of auxiliary methane was as same as the exhaust sensible heat of increased nitrogen. The oxygen ratio (the O/C ratio) $\alpha_{\mathrm{O/C}}$ was defined by the molar ratio of total O supplied as the oxygen gas and included in the sample to C content in the sample as:

$$\alpha_{\mathrm{O/C}} = \frac{V_{\mathrm{O_2}}/22.4 \times 2 + C_{\mathrm{O}} W_R/16}{C_{\mathrm{C}} W_R/12 + V_{\mathrm{CH_4}}/22.4}$$

where $V_{\mathrm{O_2}}$ and $V_{\mathrm{CH_4}}$ denote the rates of O_2 and CH_4, C_{O} and C_{C} ultimate analyses of C and O in the dry sample and W_R the feed rate of the dry sample. The O/C ratio was kept constant at 1.7 in this gasification examination, because it was found that O/C = 1.7 was shown the best gasification performance in previous experiment.

Table 3 Properties of wood.

Sample	Wood
Proximate analysis [wt%]	
Moisture	4.60
Volatile matters	82.77
Fixed carbon	16.60
Ash	0.63
Ultimate analysis [wt%]	
Carbon	48.7
Hydrogen	6.3
Nitrogen	0.12
Oxygen	44.18
Heating value [MJ/kg]	18.2
50 % mean size [m]	80

Table4 Experimental condition of gasification examination

Gasifying agent	Wood powder feeding rate [kg/hr]	$\alpha_{O/C}$ [-]	Supplying rate of auxiliary methane [Nm^3/h]
Oxygen	17.0	1.7	1.8
Air	17.5	1.7	2.7

Figure 10 shows the temperature profiles of the gasification furnace, when the wood biomass was gasified with oxygen and air at the O/C ration 1.7. The maximum temperature was over 1650 K at 200 mm from the top, when the oxygen was used. On the other hand, the maximum temperature was less than 1450 K at 1000 mm from the top, when the air was used. The maximum temperature was dropped around 200 K by the sensible heat of nitrogen, and the position of the maximum temperature measured was shifted downward of the furnace in the case of using air. However, the exit temperature of the gas was higher than the case of using the oxygen. The high temperature zone (Over 1300 K), the reaction zone, was extended by changing the gasifying agent. This phenomenon was strongly related with the superficial velocity of gas and the sensible heat of nitrogen.

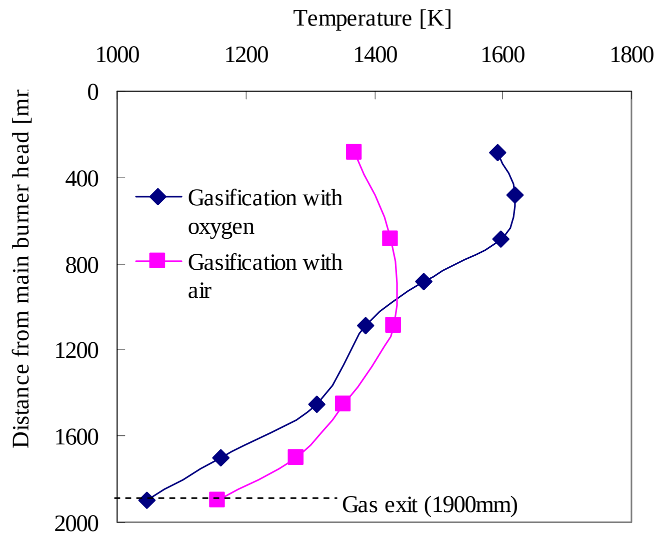

Figure 10 Temperature profile of the gasification furnace

Figure 11 shows the compositions of the gas products excluding nitrogen. The yield of hydrogen and carbon monoxide was decrease and carbon dioxide was increased by changing the gasifying agent. When the oxygen was used as the gasifying agent, O/C = 1.7 was the best operation condition. However, when the air was used, the amount of carbon dioxide was increased. When the air was used as the gasifying agent, O/C should have been controlled less than 1.7 to improve the gasification performance.

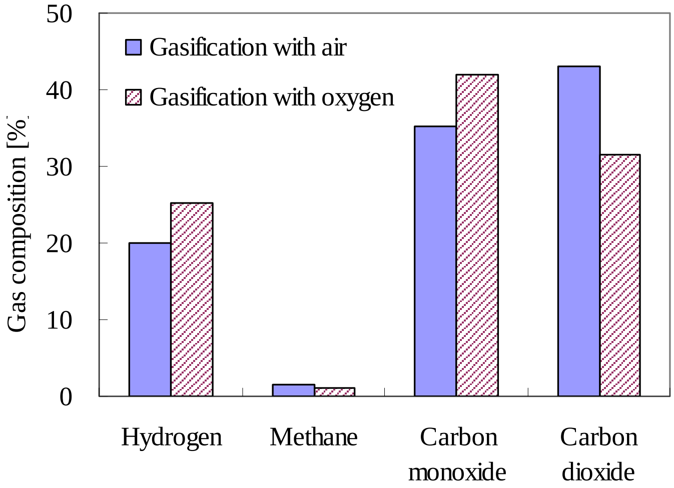

Figure 11 Compositions of gas products excluding steam and nitrogen

Figure 12 shows the carbon to gas conversion and cold gas efficiency in the both experimental conditions. The carbon to gas conversion,η_c and cold gas efficiency,η_d was expressed in the following equations as;

$$\eta_C = \frac{([CO]+[CO_2]+[CH_4]) \times Q/22.4}{C_C \cdot W_R/12.01 + V_{CH_4}/22.4} \times 100$$

Where [*i*] denotes the volume fraction of an *i*-component in yield gas at the outlet of the furnace and *Q* the gas volume produced per unit time. The temperature changed depending upon the O/C ratio usually.

$$\eta_d = \frac{H_{H2} \times V_{H2} \div 22.4 + H_{CO} \times V_{CO} \div 22.4 + H_{CH4} \times V_{CH4} \div 22.4}{H_{W,i} \times W_R + H_{CH4} \times W_{CH4,i} \div 16} \times 100$$

Where $H_{[i]}$ denotes the heating value of *i*-componets. The carbon conversion ratio was increased and the cold gas efficiency was decreased significantly, when the air was used as the gasifying agent, because the amount of carbon dioxide was increased instead of hydrogen and carbon monoxide.

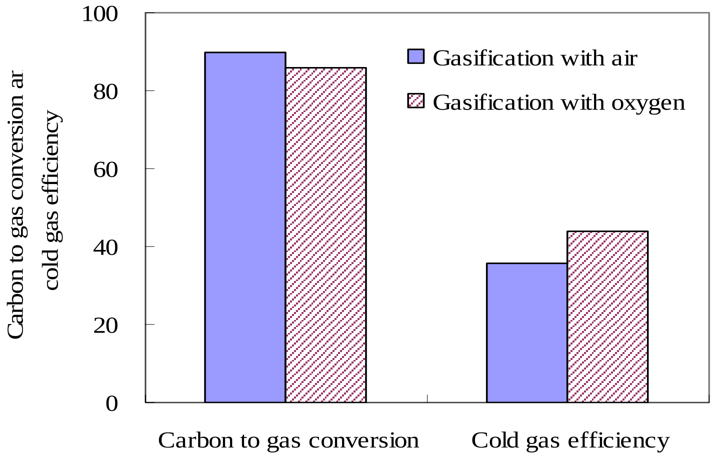

Figure 12 Carbon to gas conversion and cold gas efficiency

3. Hydrothermal treatment with pulverized wood biomass

The liquefaction was one of methods to produce chemicals from wood biomass and this method was one of the component fractionation techniques of wood constituent called hydrothermal process. There were a lot of liquefaction methods, such as steam explosion method and supercritical water method, but the liquefaction reaction in hot compressed water was progressed in mild reaction condition and simple operation condition rather than the other liquefaction method. Therefore, it was possible to simplify the total system and cut down energy consumption. For such reasons, liquefaction by hot compressed water was also considered as the effective method for the efficient wood biomass utilization.

For the effective liquefaction by hot compressed water, pretreatment such as pulverization could be effective on operations and reactions. The liquefaction technique used pulverized fine powder as materials started developing, because reactivity of the wood components may enhance. However, there were few papers considering the effect of the particle size on the liquefaction efficiency.

In this paper, in order to develop the efficient liquefaction technique of wood biomass, the wood biomass pulverized into micron order particle by a vibration mill was liquefied in hot compressed water and the one of results was introduced.

Schematic diagram of liquefaction system was shown in **Figure 13**. This was the batch type reactor (autoclave). The apparatus was consists of a pressure vessel, heater, pressure gauge, agitator for mixing the biomass slurry, thermocouple, temperature controller and rpm controller. The pressure vessel was made of stainless and the volume was 120 cc. The pressure vessel was sealed by an insulator to reduce the heat release. The temperature biomass slurry inside of the pressure vessel was measured by the inserted thermocouple and was controlled by the PID temperature controller. The biomass slurry was agitated by the agitator during the reaction to prevent the deposition of biomass power. The number of rotation was set 500 rpm. The pressure of the vessel was the same as the water vapor pressure of that temperature. The wood biomass slurry was prepared by using the pulverized power. The wood powder was mixed with deionized water and it was agitated for several minutes by shaker utile the pulverized power was dispersed uniformly. In the experiment, the biomass slurry of 80 g was made by mixing 8 g wood powder and 72 g deionized water together. Well-mixed biomass slurry of 80 g was fed in the pressure vessel. Nitrogen gas was purged for 15 minutes with agitating the biomass slurry to create an inert condition. After replacing the gas, the vessel was pressurized 2 MPa. Then the vessel was heated up at the temperature of 423 K to 573 K. After the temperature of the slurry reached the target temperature, the heater was turned off and the temperature was kept for 1 minute. One minute was the reaction time at that temperature, but it took 15 to 20 minutes for heating up the target temperature. Then, the vessel was quenched to 298 K by water immediately to prevent the secondary reaction. The reaction product was taken from the pressure vessel. It was separated solid residue from liquefied product by a suction filtration. Solid residue was dried at 378 K for 24 hours in an oven and was measured the weight.

In this study, water soluble product was defined as the liquid that the solid residue was removed from the whole liquefied product. Water soluble product and dried solid residue were designated as WS and S, respectively. Yield of the water soluble product was calculated by the following equation;

Yield of WS = (1 – amount of S / amount of charged dry biomass powder) × 100 [wt%]

Although the gas or oil soluble content was generated by the liquefaction process, the amount of those was so low that it was neglected.

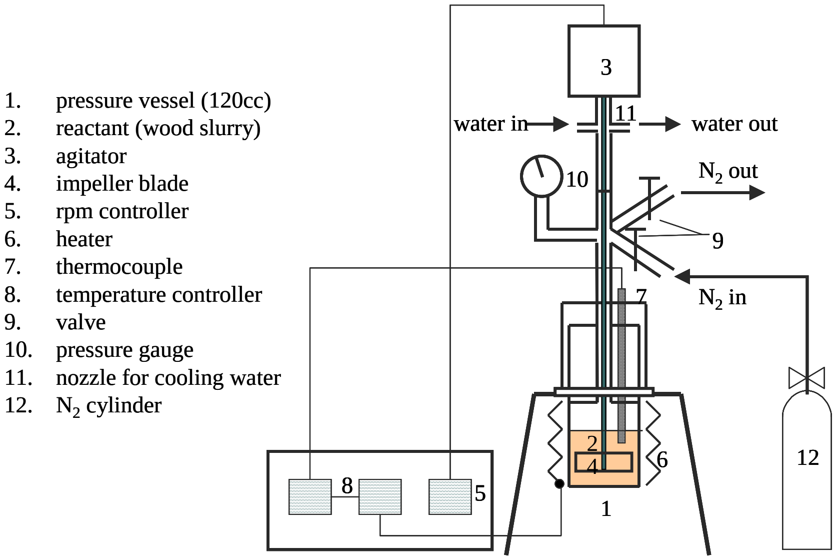

Figure 13 Schematic diagram of batch reactor for liquefaction

3.1 Water Soluble Products

The effect of the reaction temperature on the yield of WS was measured. The pulverized powder that was sieved between 53 μm and 100 μm were used. The reaction temperature was set 423 K to 573 K by each 50 K. The yield of WS in the different reaction temperature was shown in **Figure 14**. As the reaction temperature rise, the yield of WS also increased gradually. The yield of WS was around 60 % at the temperature more than 523 K. The soluble water product was decomposed components that came from cellulose and hemi-cellulose. Those decomposed included disaccharide, monosaccharide, or phenolic compounds etc. According to the results of analysis, the component of the water soluble product was considered to be different from the product formed in the different temperature.

Wood powder pulverized by the vibration mill, cutter mill and grinder was used as liquefaction materials. The wood powder was sieved between 212 µm and 500 µm to minimize particle size effect. The reaction temperature was set 523 K. **Figure 15** showed the experimental result of liquefaction. The left axis indicates the yield of WS and the right axis indicates the specific surface area of the wood particles. The yield of wood particles pulverized by the vibration mill was higher than that of the cutter mill and grinder. The specific surface area of the wood powder pulverized by the vibration mill was bigger even in the same particle size range. The contact area of the reaction enlarged and it improved reactivity of the wood powder. However, the specific surface area of the wood particles pulverized by the grinder was lower than that of the cutter mill. The yield of both materials was almost same. It indicated that the shape of the wood powder also might affect the water soluble yield.

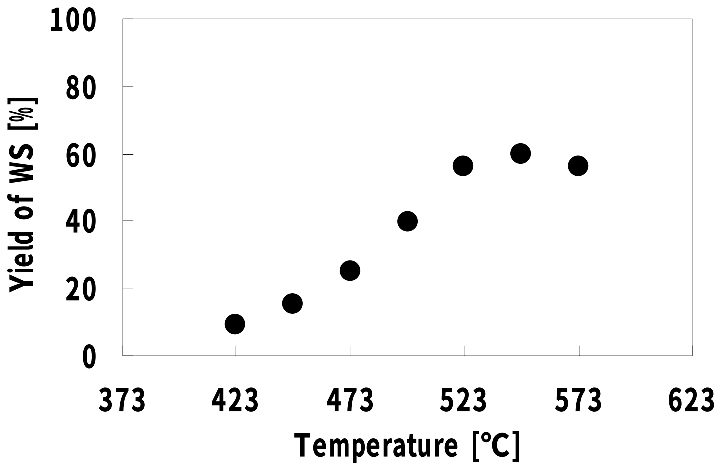

Figure 14 Effect of the reaction temperature on the yield of WS

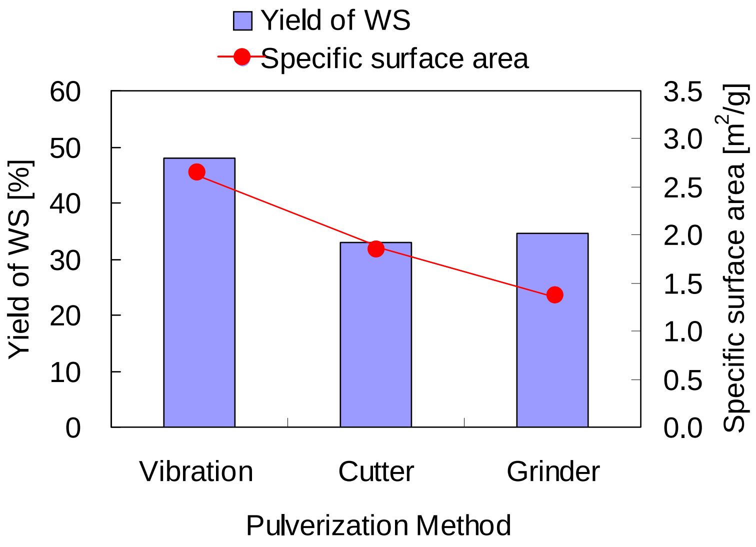

Figure 15 Effect of the particles pulverized in different method on the yield of WS

3.2 Solid products

In the liquefaction process, solid product was also obtained. This solid product was the residual of lignin. Proximate analysis and ultimate analysis of solid product were summarized in **Table 4**. This solid product had very high heating value as same as brown coal, but the volatile matter was very high and nitrogen and sulfur content were very low. **Figure 16** shows the O/C, H/C atomic ratio of solid product.

Table 4 Proximate analysis and ultimate analysis of solid product

Sample	Wood
Proximate analysis [wt%]	
Volatile matters	72.5
Fixed carbon	27.3
Ash	0.25
Ultimate analysis [wt%]	
Carbon	58.0
Hydrogen	5.6
Nitrogen	0.03
Oxygen	36.1
Sulfur	0.0
Heating value [MJ/kg]	23.3

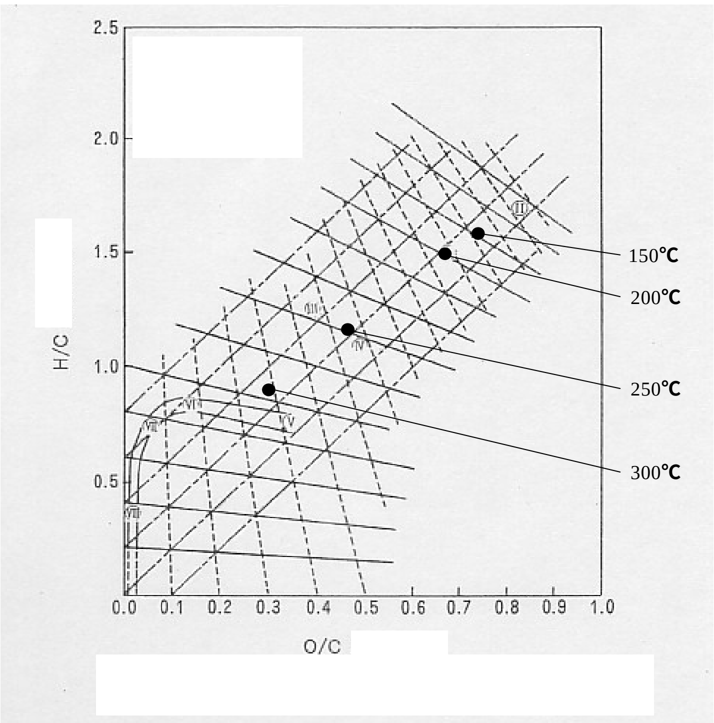

Figure 16 O/C, H/C atomic ratio of solid product

Heating temperature in the liquefaction process was increased with decreasing the O/C and H/C atomic ratio of the solid product. This was the dehydrate-reaction of wood biomass. At the temperature of 300 C, the solid product reached the same composition as the brown coal. From these results, this solid product might have used as the good solid fuel instead of coal, because reformed wood biomass (solid product) had high heating value and very clean to combustion.

4. Total wood biomass utilization system based on pulverized wood powder

Figure 17 shows total wood biomass utilization system based on pulverized wood powder. The pulverized wood powder was used as the base raw material of this system. This new utilization process was the combination of gasification and liquefaction process. The wood powder was consumed by starling engine to produce heat and electricity. The wood powder was also used for the liquefaction raw material to produce the wood recycling material. The produced electricity was consumed by the pulverization of wood biomass. On the other hand, produced heat was used for the liquefaction process or for the distillation process of liquefaction products. Generated electricity and heat was used up in this utilization system. Finally, plastic products, ethanol or solid fuel was produced as the final value-added products.

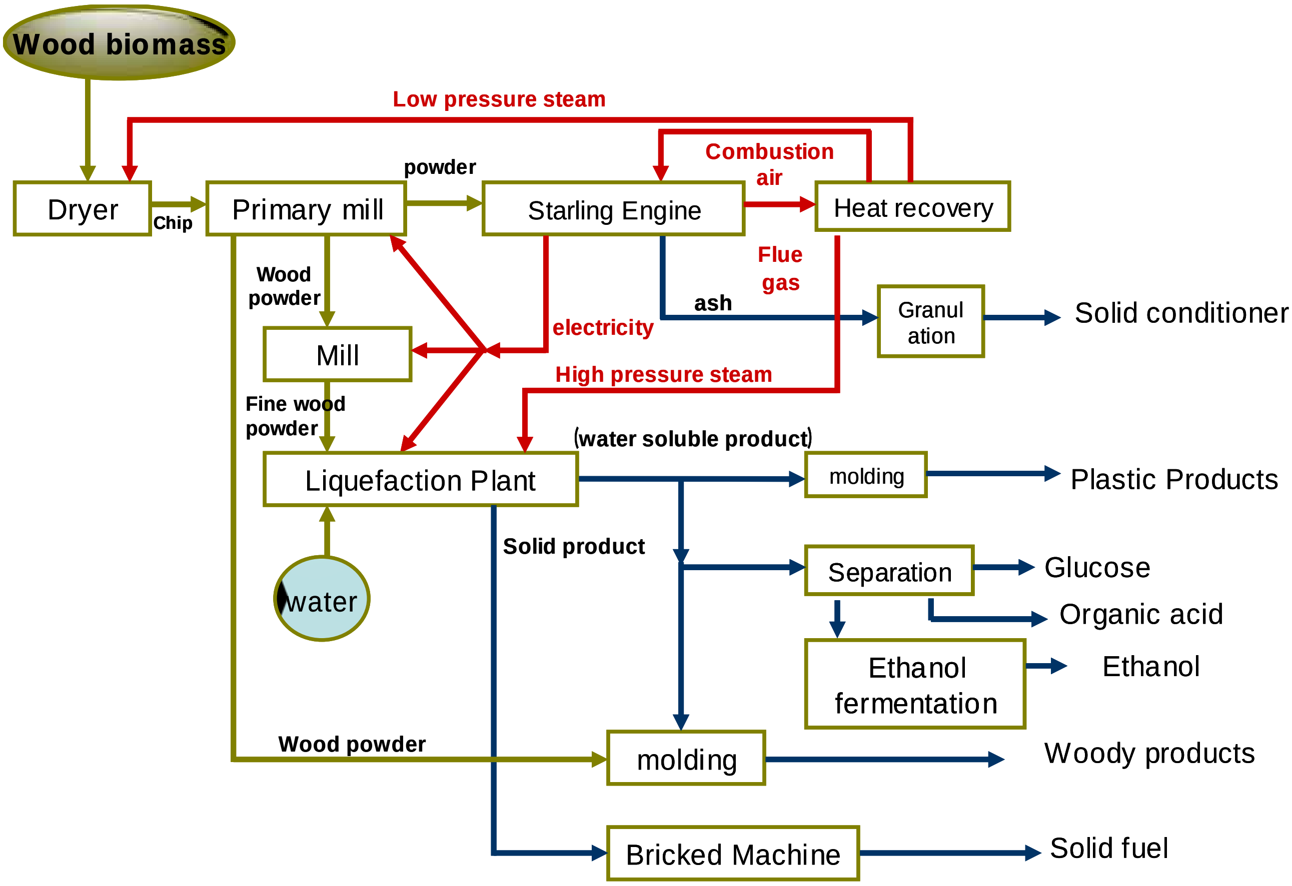

Figure 17 Total wood biomass utilization system

Conclusion

In this paper, new biomass utilization applications were discussed. Pulverization technique for the wood biomass was developed firstly, and this technique was the key technology for the development of the biomass utilization system. Various applications with the pulverized wood powder, such as gasification process and liquefaction process, were developed successfully. In the pulverization process, the wood biomass was pulverized into the micron-order particle easily by using the vibration mill. In the gasification process, high temperature gasification without yielding tar was archived. In the liquefaction process, high yield of water soluble product and solid product was confirmed. But, the establishment of total biomass utilization system by using those applications was the most important to develop resource cycling society in Japan.

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