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NOVEL ROUTES OF ADVANCED MATERIALS PROCESSING AND APPLICATIONS

Hydrothermal liquefaction of plantation biomass with two kinds of wastewater from paper industry

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Abstract The effects of two kinds of wastewater (black liquor and paper regeneration wastewater) from paper industry as solvent on the hydrothermal liquefaction of plantation biomass were discussed in this study. For the solvent of eucalyptus, black liquor was not suitable because the oil yield was very low and the yield of water-soluble constituent was very high. There was little difference in the product yields between the liquefaction of eucalyptus with the paper regeneration wastewater (pH 9.5) and that with NaOH aqueous solution (pH 9.5). In comparison with the liquefaction with water, the decrease of residue yield and the increase of oil yield were observed on the liquefaction with the wastewater. During the liquefaction with water, it was anticipated that dehydration and polymerization between carboxylic and phenolic hydroxyl groups in the molecules of the eucalyptus occurred. On the other hand, carboxylic acid formed during the liquefaction of eucalyptus was considered to be neutralized with Na^+ in NaOH aqueous solution or cations in the wastewater because the formation of carboxylate was observed in the water-soluble constituents derived from these liquefactions. Therefore, the residue yield decreased owing to the inhibition of the condensation reaction. Accordingly, possibility for the reduction of the amount of greenhouse gas (CO_2) and paper regeneration wastewater, and the production of oil from eucalyptus was clarified on the liquefaction of plantation biomass (eucalyptus) with paper regeneration wastewater.

Introduction

On the pulp cooking process, a large amount of black liquor is produced every year. Since the organic substance is contained in 20% yield, black liquor is effectively utilized thermally on the pulp industry. Although the gasification of black liquor is widely reported [1–7], investigation about the utilization of black liquor as solvent is limited for coal liquefaction [8]. Therefore, the investigation of an alternative route for the effective utilization of black liquor is necessary. On the other hand, the effective utilization of paper regeneration wastewater is required since the amount of the wastewater is increasing with the growth of the waste paper regeneration process. However, the wastewater is difficult to use thermally because the content of organic substance in the wastewater is very low. As for the investigation about utilization of the wastewater, application of nanofiltration technology is reported to recycle the wastewater [9].

The plantation of eucalyptus is carried out due to the nature of rapidly growth, material for paper, fixation of CO_2 in air. The plantation of eucalyptus is also considered as the bright energy source [10]. Considering the steep rise in crude oil prices in recent years, establishment of liquefaction technology of biomass and waste resources is required.

One of the liquefaction methods of woody biomass, hydrothermal liquefaction with alkaline and alkaline earth metal salt [11–15] is well known. Although the liquefaction of hydrolytic eucalyptus lignin was investigated [16, 17], the liquefaction of woody eucalyptus has not been reported. Both the effective utilization of paper regeneration wastewater and alternative utilization of black liquor are necessary, as mentioned above. Therefore, hydrothermal liquefaction of plantation biomass (eucalyptus) with either

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black liquor or the wastewater as solvent was carried out in this study. These results were compared with the liquefaction of eucalyptus in either water or NaOH aqueous solution at each pH value. Both the utilization of waste liquids, such as paper regeneration wastewater and black liquor, and the liquefaction of plantation biomass to produce fuel oil effectively are the aim of this study.

Experimental

Samples

An eucalyptus sample was pulverized to pass through a 42 mesh screen, and dried for 3 h under vacuum at 110 °C before use. Black liquor and paper regeneration wastewater were obtained from the pulp digesting and paper regeneration processes, respectively.

Chemical

As the solvent on the liquefaction of eucalyptus, sodium hydroxide (CAS No. 1310-73-2) of special grade (Japanese Industrial Standards) was used as aqueous solution without further purification. The purity of sodium hydroxide was over 95%, and it was commercially purchased from Wako Pure Chemical Industries, Ltd.

Hydrothermal liquefaction

The experimental scheme for hydrothermal liquefaction of eucalyptus with either black liquor or paper regeneration wastewater is summarized in Fig. 1. An amount of 5 g of eucalyptus and 30 g of liquid (black liquor or the wastewater) were placed in a 200 cm^3 autoclave under an initial nitrogen pressure of 2.0 MPa. The reactor was heated at an average rate of 7 °C/min. to a desired temperature (150–350 °C) and maintained at the temperature for 0 min. After cooling, the gaseous products (gas) were analyzed by using GC-TCD. The product remaining in the autoclave was filtered and rinsed with water, followed by the separation into the filtrate and residue. The water-soluble material (WS) was prepared by evaporating the filtrate. After the residue dried under vacuum was extracted with acetone, acetone soluble (oil), and acetone insoluble (residue) materials were prepared. The liquefaction of eucalyptus with either water or the desired concentration of NaOH aqueous solution was carried out in a similar manner. On the liquefaction of eucalyptus with any one of the wastewater, water or NaOH aqueous solution, the yield of water was calculated as the difference between the weight of the feed eucalyptus and that of the recovered constituents (gas + WS + oil + residue) on a d.a.f. basis.

Analysis

The gas chromatographic analyses of gaseous components (methane, ethane, propane, butane, CO, and CO_2) evolved in the liquefaction were performed on a Shimadzu GC-9A instrument equipped with a thermal conductivity detector and a dual-column molecular sieve (7 m, Shimadzu) and Porapak N (2 m, Waters). The FT-IR spectra of WS, oil and residue constituents obtained from the liquefactions of eucalyptus were measured with a JASCO FT/IR-420 spectrometer.

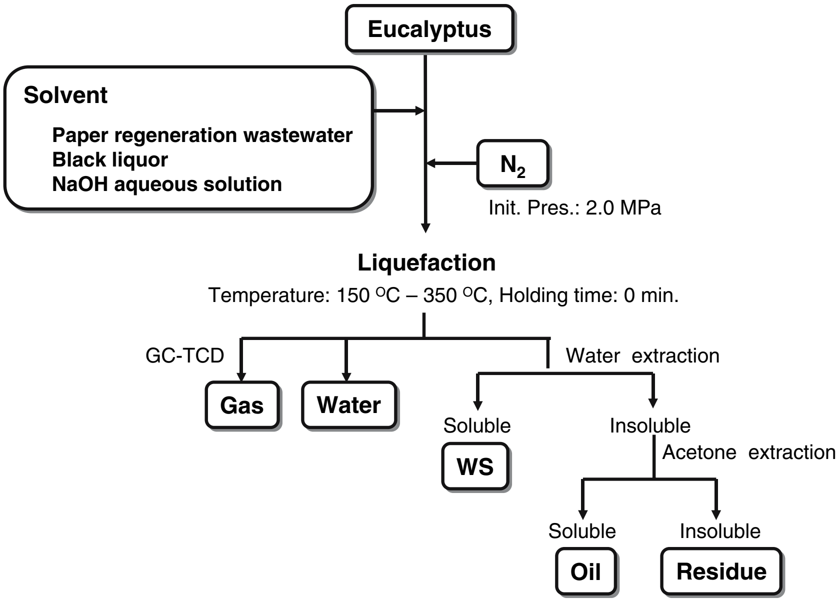


Fig. 1 Experimental scheme for hydrothermal liquefactions of eucalyptus

Table 1 Contents of water, organic substance and ash constituents in black liquor and paper regeneration wastewater (wt%)

	Water	Organic	Ash
Black liquor	82.9	10.7	6.4
Paper regeneration wastewater	99.2	0.4	0.4

Results and discussion

Analytical data of samples

The contents of water, organic substance and ash constituents in black liquor and paper regeneration wastewater are shown in Table 1. The utilization of wastewater as thermal energy was not suitable since the content of organic substance in the wastewater was very low. The analytical data and the calorific values of eucalyptus and solid constituents in black liquor and the wastewater are given in Table 2. The content of nitrogen was not detected, however, the high concentration of oxygen was observed in all samples. Therefore, the calorific values of these samples were low when these samples were used directly as thermal energy. The pH values of black liquor and the wastewater were 13.0 and 9.5, respectively.

Effect of pH on the hydrothermal liquefaction of eucalyptus with NaOH aqueous solution

Prior to the additive effect of black liquor or the wastewater on the hydrothermal liquefaction of eucalyptus, the effect of pH on the liquefaction of eucalyptus with NaOH aqueous solution is discussed. The product yields after the liquefaction at 300 °C are shown in Fig. 2. The yield of residue was over 40% on the reaction with water only (pH 5.8). The decrease of residue yield and the increase of WS yield were observed with the increase of pH value on the liquefaction with NaOH aqueous solution. However, the yield of oil was almost constant irrespective of the pH value in the solution. Therefore, the presence of alkaline solution was expected to be necessary for the liquefaction of eucalyptus, however, the yield of residue decreased on the liquefaction with even a dilute alkaline solution. On the other hand, liquefaction with a concentrated alkaline solution caused the increase of the yield of WS, which was difficult to use as fuel.

Hydrothermal liquefaction of eucalyptus with black liquor

The product yields after the hydrothermal liquefaction of eucalyptus with black liquor are shown in Fig. 3. The yields after the liquefaction of black liquor alone are also given in Fig. 3. At any reaction temperature, over 90% of gas constituent was CO_2. On the liquefaction of black liquor alone, the decrease of WS yield and the increases of the yields of gas, residue and oil were observed with the increase of reaction temperature. Although the pH value of black liquor was 13.0, the value decreased to 9.9 after the liquefaction of black liquor alone. Therefore, the alkaline constituents in black liquor were anticipated to be neutralized by the organic acids in WS constituent.

The yields after the liquefaction of eucalyptus with black liquor are compared with those in NaOH aqueous solution (pH 13.0) in Fig. 4. The yields with black liquor in Fig. 4 are based on the feed weight of organic substance in eucalyptus by subtracting the yields after the liquefaction of black liquor alone from the yields after the liquefaction of eucalyptus with black liquor in Fig. 3. The ultimate analyses and calorific values of residue and oil constituents after the liquefaction are given in Table 3. The atomic ratios of O/C in residue and oil constituents in Table 3 were lower than those in eucalyptus (Table 2). Therefore, pyrolysis of eucalyptus was accelerated by the increase of reaction temperature, which resulted the deoxygenation of eucalyptus. In Fig. 4, on the liquefaction of eucalyptus with NaOH aqueous solution at 350 °C, the decrease of oil yield and the increases of the yields of residue and gas were observed in comparison with that at 300 °C. Therefore, it was considered that pyrolysis and polymerization reactions were occurred simultaneously with the increase of reaction temperature on the liquefaction of eucalyptus with NaOH aqueous solution. On the other hand, the yield of oil was very low and the WS yield was very high after the liquefaction of eucalyptus with black liquor. The increase of WS yield and the decreases of the yields of oil and residue were

Table 2 Analytical data and the calorific values of eucalyptus and solid constituents in black liquor and paper regeneration wastewater (wt% d.a.f. basis)

	C	H	N	O[a]	Ash[b]	H/C	O/C	Calorific value[c]
Eucalyptus	46.2	5.9	0.0	47.9	0.4	1.52	0.78	15.3
Solid in black liquor	30.1	3.2	0.0	66.7	6.5	1.28	1.67	2.5
Solid in wastewater	41.5	5.2	0.0	53.3	49.3	1.48	0.96	–

[a] By difference; [b] Dry basis; [c] kJ/g

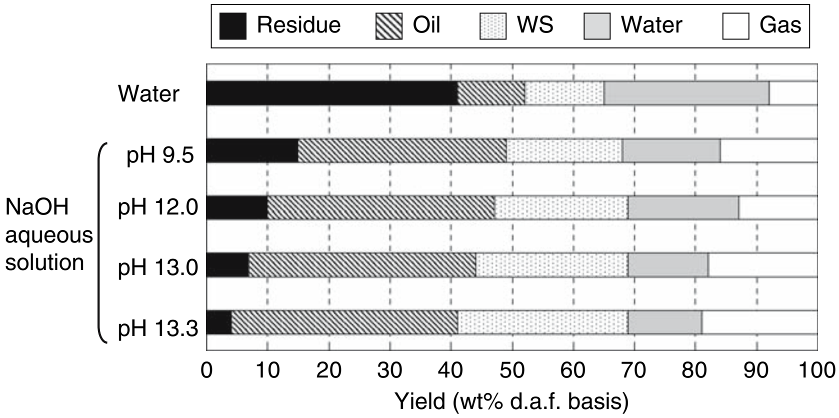


Fig. 2 Product yields after the liquefaction of eucalyptus with NaOH aqueous solution at 300 °C

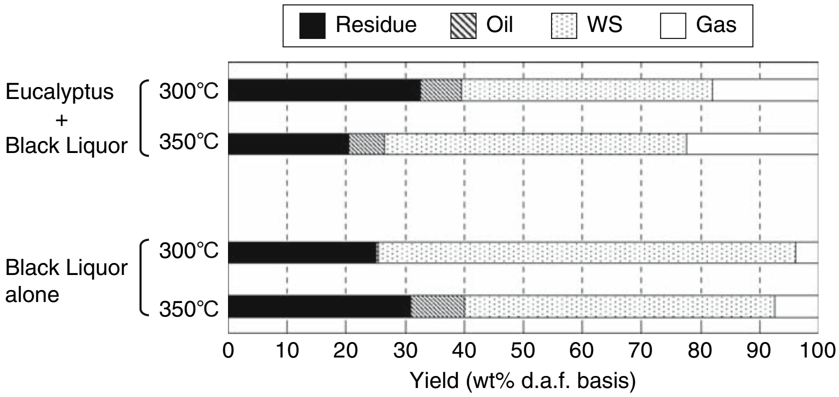


Fig. 3 Product yields after the liquefaction of black liquor alone and eucalyptus with black liquor

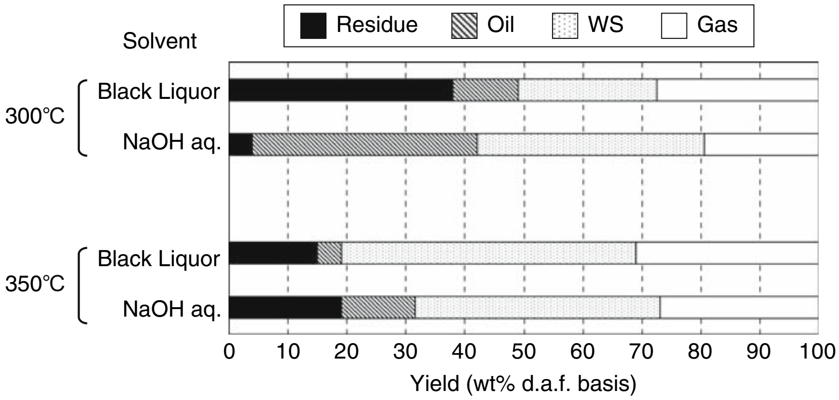


Fig. 4 Product yields after the liquefaction of eucalyptus with either black liquor or NaOH aqueous solution (pH 13.0)

observed with the increase of reaction temperature on the liquefaction of eucalyptus with black liquor. It was expected that not only pyrolysis but also some interaction between eucalyptus and black liquor took place on the liquefaction of eucalyptus with black liquor.

Compared with the liquefaction of eucalyptus with NaOH aqueous solution at 300 °C, the increases of the yields of gas and residue, and the decreases of the yields of oil and WS were observed on that with black liquor at 300 °C. Therefore, it was anticipated that the hydrolysis reaction was suppressed by the condensation between radicals derived from eucalyptus and those from black liquor on the liquefaction at 300 °C. On the other hand, in comparison with the liquefaction of eucalyptus with NaOH aqueous solution at 350 °C, the increase of WS yield and the decreases of yields of residue and oil were observed on that with black liquor at 350 °C. Therefore, on the liquefaction at 350 °C, it was considered that hydrolysis reaction to yield WS constituent was enhanced because the suppression of hydrolysis at 300 °C was decreased by the

Table 3 Ultimate analyses and calorific values of residue and oil constituents after the liquefaction of eucalyptus, black liquor and the mixture (wt% d.a.f. basis)

Liquefaction		Product	C	H	N	O[a]	H/C	O/C
Black liquor	300 °C	Residue	72.6	4.8	0.0	22.6	0.78	0.23
		Oil	75.8	7.0	0.0	17.2	1.10	0.17
	350 °C	Residue	81.3	5.2	0.0	13.5	0.76	0.12
		Oil	80.9	7.0	0.0	12.1	1.03	0.11
Eucalyptus	300 °C	Residue	70.1	5.1	0.0	24.8	0.86	0.27
		Oil	70.5	6.3	0.0	23.2	1.06	0.25
	350 °C	Residue	83.4	5.2	0.0	11.4	0.74	0.10
		Oil	79.2	7.0	0.0	13.8	1.05	0.13
Eucalyptus + Black liquor	300 °C	Residue	73.8	5.2	0.0	21.0	0.84	0.21
		Oil	74.0	6.5	0.0	19.5	1.05	0.20
	350 °C	Residue	81.9	5.3	0.0	12.8	0.77	0.12
		Oil	79.8	7.0	0.0	13.2	1.04	0.12

[a] By difference

enhancement of pyrolysis of molecules in black liquor. Accordingly, black liquor was not suitable for the solvent of eucalyptus because the oil yield was very low and the WS yield was very high after the liquefaction of eucalyptus with black liquor.

Hydrothermal liquefaction of eucalyptus with paper regeneration wastewater

The product yields after the hydrothermal liquefaction of eucalyptus with paper regeneration wastewater are shown in Fig. 5. On the liquefaction at 150 °C, the residue yield was very high, and it seemed that liquefaction of eucalyptus hardly occurred from the appearance of the product. With the increase of the reaction temperature from 150 to 300 °C, the oil yield increased and the residue yield decreased. In particular, the yield of oil increased significantly with the increase of the reaction temperature from 200 to 300 °C. On the other hand, in comparison with the liquefaction at 300 °C, the decrease of oil yield and the increases of yields of gas and WS were observed on that at 350 °C. As well as the liquefaction of eucalyptus with black liquor, over 90% of gas constituent was CO_2 at any reaction temperature. It was considered that the excess cracking reaction of oil constituent to yield gas occurred on the liquefaction at 350 °C. Although the pH value of paper regeneration wastewater was 9.5, the value decreased to 3–4 after the liquefaction with eucalyptus. Therefore, the alkaline constituents in the wastewater were neutralized completely by the organic acids in WS constituent because the pH value after the liquefaction indicated acidic.

The atomic ratios of O/C and H/C of residue and oil constituents after the liquefaction of eucalyptus with the wastewater are given in Table 4. With the increase of the reaction temperature from 200 to 300 °C, the yields of gas

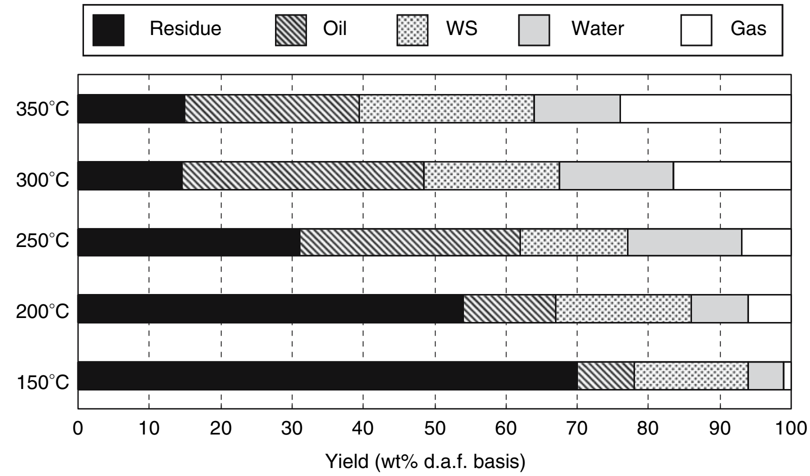


Fig. 5 Product yields after the liquefaction of eucalyptus with paper regeneration wastewater

Table 4 Atomic ratios of O/C and H/C of residue and oil constituents after the liquefaction of eucalyptus with paper regeneration wastewater

	150 °C		200 °C		250 °C		300 °C		350 °C	
	H/C	O/C	H/C	O/C	H/C	O/C	H/C	O/C	H/C	O/C
Residue	1.48	0.73	1.37	0.70	1.27	0.59	0.89	0.29	0.88	0.25
Oil	1.15	0.35	0.98	0.33	1.01	0.30	1.00	0.25	1.06	0.18

and oil increased and the atomic ratios of O/C and H/C in residue decreased significantly. Accordingly, it was anticipated that the upgrading reaction and deoxygenation of eucalyptus to yield oil constituent occurred significantly from 200–300 °C.

The FT-IR spectra of residue and WS constituents after the liquefaction of eucalyptus with the wastewater are shown in Figs. 6 and 7, respectively. From Fig. 6, the FT-IR spectrum of residue derived from the liquefaction at 150 °C was similar to that of eucalyptus. The strength of absorption bands ascribed to hydroxyl group (3,400 cm^{-1}) and aliphatic ether (1,050 cm^{-1}) decreased with the increase of reaction temperature above 200 °C. In particular, during the liquefaction from 250 to 300 °C, pyrolysis of eucalyptus enhanced because those absorption bands decreased significantly. On the liquefaction at 350 °C, the breakage of hydroxyl group and the cleavage of aliphatic ether were expected to be completed since those absorption bands almost disappeared. On the FT-IR spectra of WS in Fig. 7, the strength of absorption band ascribed to carboxylic salt (1,600 and 1,450 cm^{-1}) increased with the increase of reaction temperature. It was clarified that Na^+ and Ca^{2+} were contained in the wastewater. Therefore, it was considered that carboxylic acids were formed as WS constituent during the liquefaction of eucalyptus above 150 °C, and the proton in the carboxylic acid was exchanged with cation in the wastewater.

Hydrothermal liquefaction of eucalyptus with water

The product yields after the hydrothermal liquefaction of eucalyptus with water are given in Fig. 8. As well as the liquefaction with the paper regeneration wastewater, the residue yield was very high on the liquefaction at 150 °C. With the increase of the reaction temperature from 150 to 250 °C, the oil yield increased and the residue yield decreased. However, the opposite tendency, such as the decrease of oil yield and the increase of residue yield, appeared with the increase of the reaction temperature from 250 to 350 °C. The yield of gas increased with the increase of the reaction temperature. At any reaction temperature, over 90% of gas constituent was CO_2 as well as the liquefaction with the other solvent. Therefore, it was expected that the excess cracking reaction of oil constituent to yield gas and polymerization reaction of oil constituent to yield residue occurred simultaneously on the liquefaction above 250 °C.

The FT-IR spectra of residue and WS constituents after the liquefaction of eucalyptus with water are shown in Figs. 9 and 10, respectively. On the FT-IR spectra of residue in Fig. 9, the strength of absorption bands ascribed to hydroxyl group and aliphatic ether decreased with the increase of reaction temperature above 200 °C, as well as the liquefaction with the wastewater. On the other hand, on the FT-IR spectra of WS in Fig. 10, the strength of

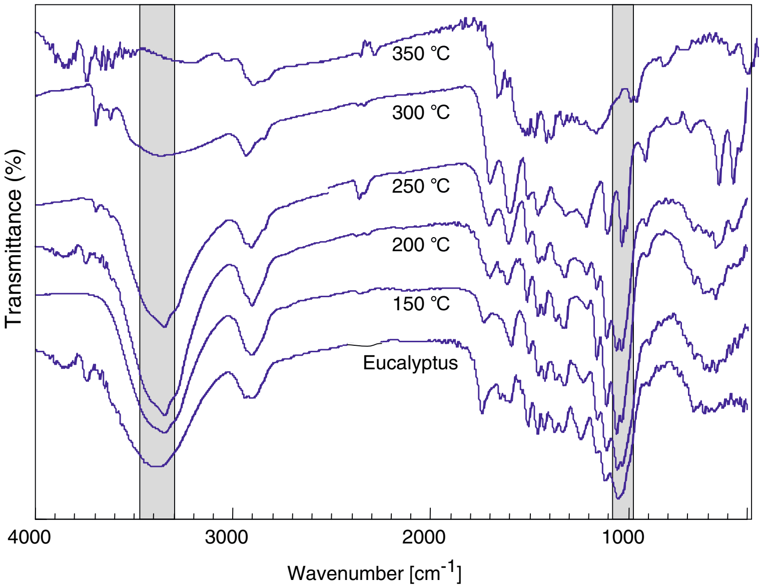


Fig. 6 FT-IR spectra of residue constituent after the liquefaction of eucalyptus with the wastewater

Fig. 7 FT-IR spectra of WS constituent after the liquefaction of eucalyptus with the wastewater

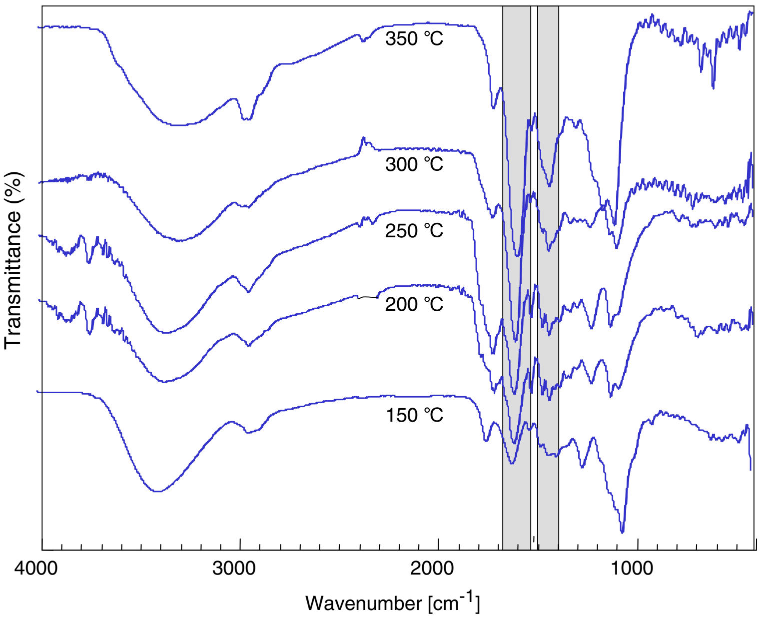


Fig. 8 Product yields after the liquefaction of eucalyptus with water

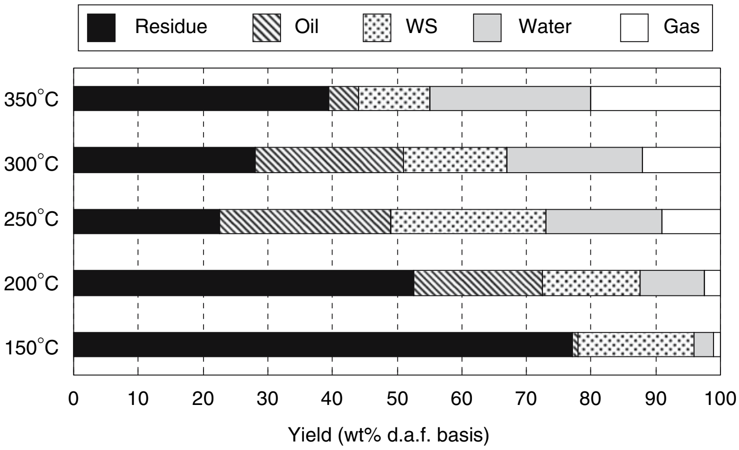
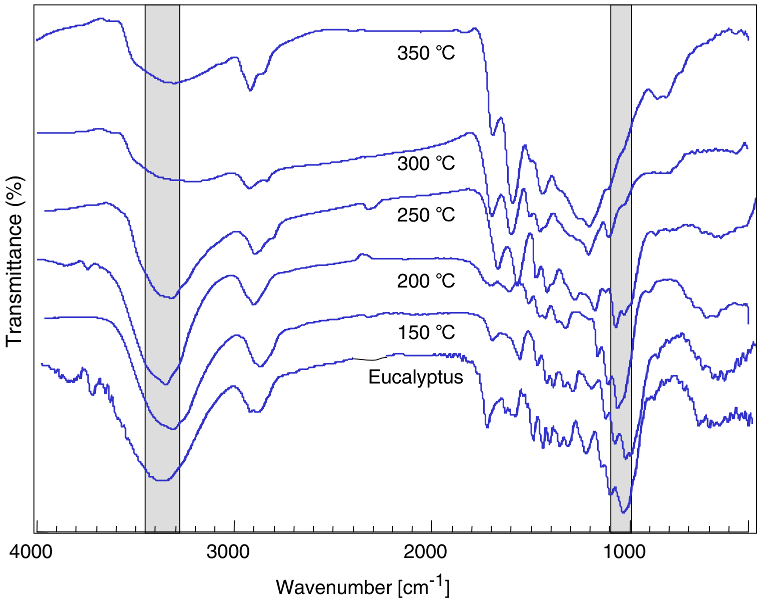


Fig. 9 FT-IR spectra of residue constituent after the liquefaction of eucalyptus with water



absorption bands ascribed to carboxyl group (1,700 cm^{-1}) and aliphatic ester (1,100 cm^{-1}) increased with the increase of reaction temperature. On the liquefaction of eucalyptus with paper regeneration wastewater, carboxylic acids formed during the liquefaction were neutralized with cations in the wastewater as discussed in 3.4. However, the neutralization did not occur due to the absence of cation on the liquefaction with water, and the aliphatic ester bonds were formed by the dehydrolysis between the hydroxyl and carboxyl groups in the molecules derived from eucalyptus. Therefore, on the liquefaction of eucalyptus with water above 250 °C, it was considered that the residue yield increased because the dehydration and polymerization, such as the formation of aliphatic ester bonds between the hydroxyl groups attached to the molecules in residue and the carboxyl groups attached to the molecules in WS, occurred.

Hydrothermal liquefaction of eucalyptus with NaOH aqueous solution

The product yields after the hydrothermal liquefaction of eucalyptus with NaOH aqueous solution are given in Fig. 11. As well as the liquefaction with the paper regeneration wastewater, the residue yield was very high on the liquefaction at 150 °C. With the increase of the reaction temperature from 150 to 300 °C, the oil yield increased and the residue yield decreased. However, the opposite tendency, such as the decrease of oil yield and the increase of

Fig. 10 FT-IR spectra of WS constituent after the liquefaction of eucalyptus with water

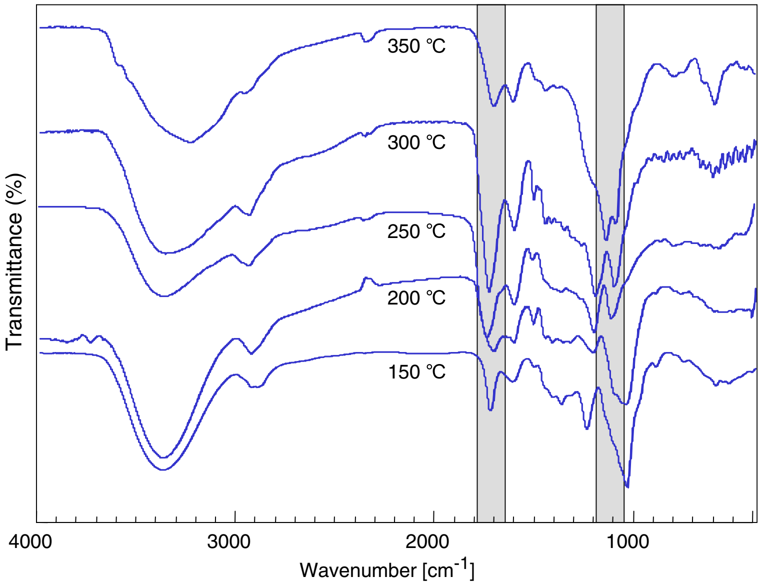
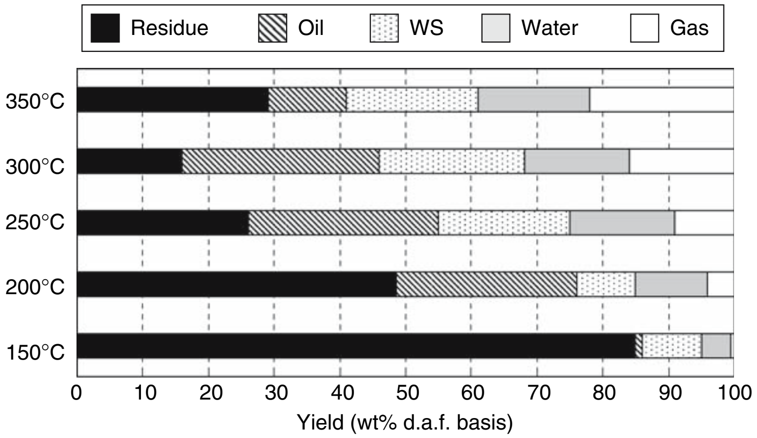


Fig. 11 Product yields after the liquefaction of eucalyptus with NaOH aqueous solution (pH 9.5)



residue yield, appeared with the increase of the reaction temperature from 300 to 350 °C. The yield of gas increased with the increase of the reaction temperature, and over 90% of gas constituent was CO_2 at any reaction temperature.

The FT-IR spectra of residue and WS constituents after the liquefaction of eucalyptus with NaOH aqueous solution are shown in Figs. 12 and 13, respectively. As well as the tendency in FT-IR spectra of constituents after the liquefaction with paper regeneration wastewater, the strength of absorption bands in residue ascribed to hydroxyl group and aliphatic ether decreased significantly with the increase of reaction temperature above 200 °C, as shown in Fig. 12. In Fig. 13, the strength of absorption band in WS constituent ascribed to carboxylic salt increased with the increase of reaction temperature. Therefore, as well as the discussion for the liquefaction with paper regeneration wastewater, it was considered that carboxylic acids were formed during the liquefaction of eucalyptus, and the proton in carboxylic acids were exchanged with Na^+ in NaOH aqueous solution.

Effect of solvent on the hydrothermal liquefaction of eucalyptus

The product yields after the hydrothermal liquefaction of eucalyptus at 300 °C with several kinds of solvents are

Fig. 12 FT-IR spectra of residue constituent after the liquefaction of eucalyptus with NaOH aqueous solution

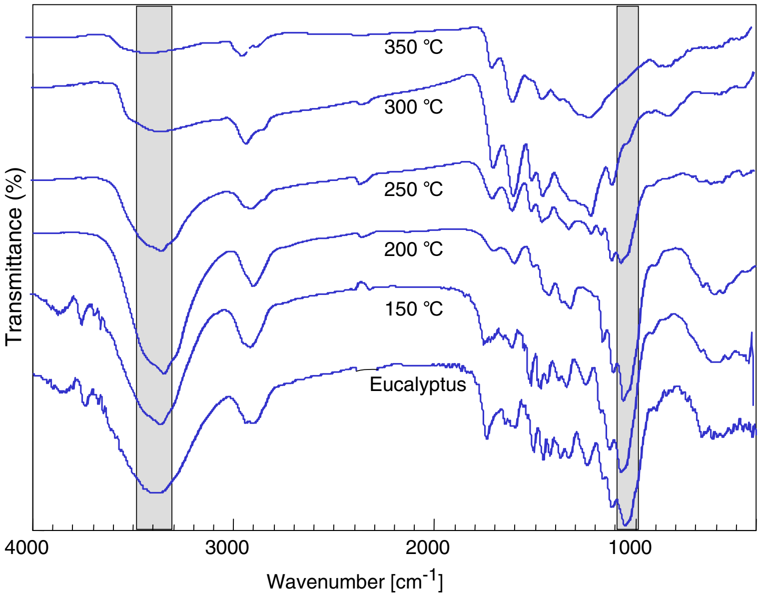
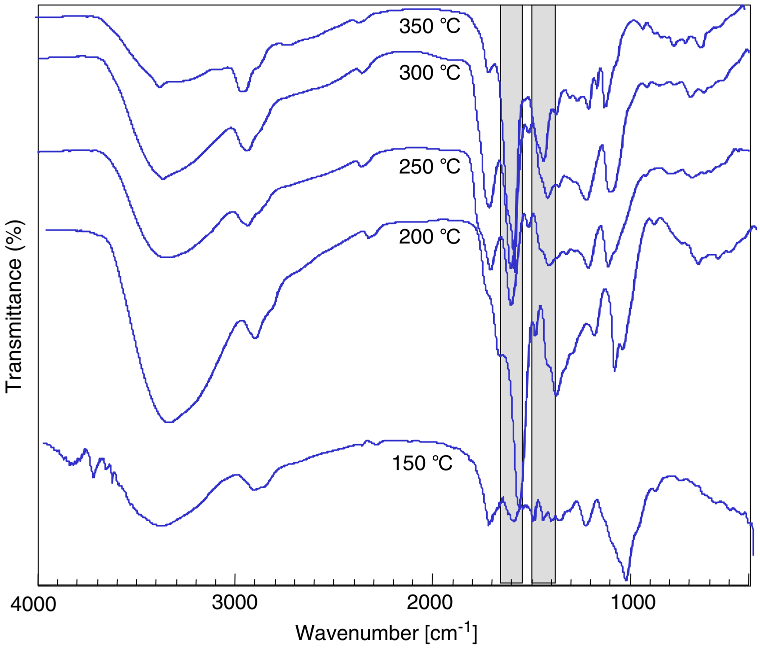


Fig. 13 FT-IR spectra of WS constituent after the liquefaction of eucalyptus with NaOH aqueous solution



compared in Fig. 14. The pH value of NaOH aqueous solution was adjusted to that of paper regeneration wastewater (pH 9.5). The FT-IR spectrum of oil constituent after each liquefaction is shown in Fig. 15. The atomic ratios of O/C and H/C of residue and oil constituents after each liquefaction are given in Table 5. The calorific value of oil constituent is shown in Table 6. There was little difference on the product yields, FT-IR spectra, the atomic ratios and the calorific value of oil constituent between the liquefaction with the wastewater and that with NaOH aqueous solution. On the liquefaction with the wastewater, the residue yield was lower, and the yield and the calorific value of oil constituent were higher than those with water. As mentioned above, on the liquefaction with water, the polymerization, such as the formation of aliphatic ester bonds between the hydroxyl groups attached to the molecules in residue and the carboxyl groups attached to the molecules in WS, occurred. However, on the liquefaction of eucalyptus with paper regeneration wastewater, carboxylic acids formed during the liquefaction were neutralized with cations in the wastewater. Therefore, it was clarified that cation exchangeable to the proton of carboxyl group in WS derived from eucalyptus was necessary in the liquefaction solvent of eucalyptus.

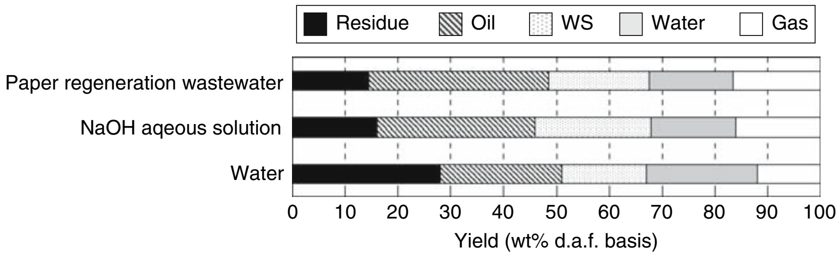


Fig. 14 Product yields after the liquefaction of eucalyptus at 300 °C with several kinds of solvents

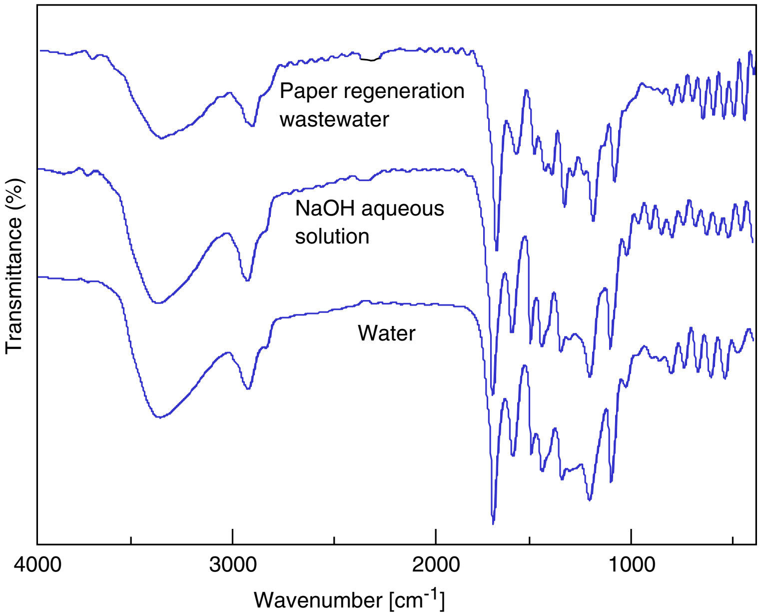


Fig. 15 FT-IR spectra of oil constituent after the liquefaction of eucalyptus at 300 °C with several kinds of solvents

Table 5 Atomic ratios of O/C and H/C of residue and oil constituent after the liquefaction with any one of the paper regeneration wastewater, NaOH aqueous solution or water

	Wastewater		NaOH aq.		Water	
	H/C	O/C	H/C	O/C	H/C	O/C
Residue	0.89	0.29	0.84	0.26	0.85	0.26
Oil	1.00	0.25	1.00	0.25	0.98	0.27

Table 6 Calorific value (kJ/g) of oil constituent after the liquefaction with any one of the paper regeneration wastewater, NaOH aqueous solution or water

	Wastewater	NaOH aq.	Water
Calorific value	28.0	28.1	27.0

Conclusions

1. For the solvent of eucalyptus, black liquor was not suitable because the yield of oil constituent was very low and the yield of water-soluble constituent was very high.
2. On the liquefaction of eucalyptus with water, the yield of residue increased because the dehydration and polymerization, such as the formation of aliphatic ester bonds, occurred.
3. On the liquefaction of eucalyptus with paper regeneration wastewater, the residue yield was lower, and the yield and the calorific value of oil constituent were higher than those with water. Therefore, the condensation reaction observed on the liquefaction with water was inhibited because carboxylic acids formed during the liquefaction of eucalyptus were neutralized with cations in the wastewater.
4. The yield of oil on the liquefaction of eucalyptus with paper regeneration wastewater at 300 °C was highest among those with other temperature (150–350 °C).

Accordingly, possibility for the reduction of the amount of greenhouse gas (CO_2) and paper regeneration wastewater, and the production of oil from eucalyptus was clarified on the liquefaction of plantation biomass (eucalyptus) with paper regeneration wastewater.

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