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DISSOLUTION OF SOLVENT-EXTRACTED RICE STRAW IN KEROSENE BY CATALYTIC HYDROCRACKING

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ABSTRACT

In order to increase the solubility of solvent-extracted component from rice straw (RS Soluble) in kerosene, the RS Soluble was hydrocracked in an autoclave at 200 and 350°C in a cyclohexane solvent using 5 wt% platinum-, palladium- or rhodium-loaded Y zeolite catalysts (5PtY, 5PdY or 5RhY). A NH₃-TPD (temperature-programmed desorption) revealed that 5PtY had almost the same acid property (acidity and the amount of acid sites) as the HY zeolite. The kerosene-soluble yield for the untreated RS Soluble was only about 20 wt%. The yield of kerosene-soluble components produced by the hydrocracking of RS Soluble at 200°C increased to 34-45 wt% with or without catalyst. The maximum yield of kerosene-soluble components produced by the hydrocracking of RS Soluble at 350°C was 61 wt%, which was achieved using 5PtY.

INTRODUCTION

In order to prevent global warming, the development of a liquid fuel alternative to petroleum is an urgent issue. Biomass, a renewable energy, has a potential to replace petroleum. It is well known that pyrolysis of biomass at 450-550°C at a heating rate of 10³-10⁴ K / s produces bio-oil [1]. However, the calorific value of bio-oil is as low as 16-19 MJ / kg because the oxygen content in bio-oil is quite high (35-40 wt%) [2]. Therefore, deoxygenation is necessary to upgrade bio-oil [3]. Hydrodeoxygenation (HDO) and zeolite cracking are known as deoxygenation methods [4-8], but at present it cannot be said that a high-quality liquid fuel is obtained in high yield.

The authors focused on the solvent extraction method developed by Miura group [9-12]. This is a method of upgrading by successfully removing oxygen from low-rank coal and biomass. When low-rank coal or biomass is placed in an autoclave together with 1-methylnaphthalene (MN) solvent and the mixture is stirred at 350°C for 60 min in a nitrogen atmosphere, three products with different solubilities in the solvent are obtained: (1) MN-insoluble component at 350°C (Residue), (2) MN-insoluble component at room temperature (Deposit), and (3) MN-soluble component at room temperature (Soluble). The third component, Soluble, can be recovered as a solid by removing the MN solvent. For example, from a rice straw with a carbon content of 42.5 wt% (dry ash free) and a calorific value of 14.7 MJ / kg (daf), Soluble with a carbon content of 84.7 wt% (daf) and a calorific value of 37.6 MJ / kg (daf) was extracted with a yield of 20.7 wt% (daf) [12]. In this study, we attempt to dissolve Soluble extracted from rice straw (hereafter referred to as RS Soluble) in kerosene. Kerosene is a mixture of C₁₀-C₁₆ alkanes (molecular weight: approximately 140-230) and can dissolve not only alkanes but also benzene, naphthalene and their derivatives. On the other hand, previous studies have shown that there are polycyclic aromatic hydrocarbons containing three rings in the structure of RS Soluble and the molecular weight of RS Soluble is approximately 500 [11, 12]. That is, in order to dissolve the RS Soluble in kerosene, the RS Soluble must be hydrocracked.

Sano et al. reported that the hydrogenation conversion of benzene decreased significantly with increasing in the SiO₂ / Al₂O₃ ratio of HZSM-5 zeolite [13]. This was considered to be due to the decrease in the amount of Bronsted acid with the increase in the SiO₂ / Al₂O₃ ratio. Arora et al. used Beta zeolite to hydrogenate benzene and naphthalene [14]. As a result, they reported that benzene was converted to cyclohexene and methylcyclopentane, and naphthalene was converted to tetralin and 1-propenylbenzene. This result indicates that the zeolite has functions of cleaving covalent bonds as well as hydrogenating aromatic rings. Wang et al. reported that the hydrogenation conversion of benzene and selectivity of cyclohexane increased with increasing amount of Pt as a result of hydrogenation of benzene using Y zeolite catalysts with various Pt loadings [15]. Similarly, it has been reported that the use of bifunctional catalyst such as Pd-Pt / Y zeolite [16], Ru / Y zeolite [17], MoC / Y zeolite [18], Pt / Y zeolite [19], and Ni-W / Beta zeolite [20], which combines a zeolite (ability to cleave covalent bonds)



with metal (ability to hydrogenate aromatic hydrocarbons), promoted the hydrocracking of aromatic hydrocarbons. Therefore, the bifunctional catalysts consisting of Pt, Pd and Rh and Y zeolite with relatively large pores (around 9 Å) are expected to hydrocrack the RS Soluble to increase the yield of kerosene-soluble components.

In this study, the effect of the prepared Pt, Pd and Rh-loaded Y zeolite catalysts on the hydrogenation of polycyclic aromatic hydrocarbons such as anthracene, phenanthrene, pyrene and so on, which were selected as model compounds of RS Soluble, was investigated. Furthermore, the effect of these catalysts on the yield of kerosene-soluble components produced by the hydrocracking of RS Soluble was also investigated.

EXPERIMENTAL

Sample

The Soluble extracted from rice straw at 350°C (RS Soluble) was provided by Kobe Steel. This RS Soluble was crushed and sieved to less than 150 µm. Table 1 shows the ultimate and elemental analyses, and calorific value of RS Soluble.

Table 1. Ultimate analysis, proximate analysis and calorific value of RS Soluble.

Ultimate analysis (wt%, d.a.f.)				Proximate analysis (wt%, d.b.)			Calorific value (MJ/kg)
C	H	N	O	VM	FC	Ash	
81.9	7.3	0.6	10.3	84.3	14.7	1.0	36.1

In order to evaluate the hydrogenation ability of the catalyst, the polycyclic aromatic hydrocarbons such as anthracene (AN, Nacalai), phenanthrene (PH, Nacalai), pyrene (PY, Nacalai), 9-phenylanthracene (PA, Nacalai) and 9,10-diphenylanthracene (DPA, Nacalai) were used as model compounds of RS Soluble.

Catalyst preparation

A NH₄Y zeolite (Wako) (Si / Al ratio: 7, specific surface area: 700 m² / g, pore diameter: 9 Å) was calcined at 500°C for 2 h to obtain HY zeolite. Hexachloroplatinic acid hexahydrate (H₂PtCl₆·6H₂O, Wako), palladium chloride (PdCl₂, Wako) and rhodium chloride trihydrate (RhCl₃·3H₂O, Wako) were dissolved in 50 mL of distilled water so that the metal loading was 5 wt%. However, since palladium chloride was difficult to dissolve in distilled water, it was dissolved by adding a small amount of hydrochloric acid. The HY was added to the aqueous precursor solution, and the mixture was stirred with a hot stirrer at 80°C for 1 h, and then distilled water was removed with a rotary evaporator. The sample was dried at 60°C for 12 h and calcined at 500°C for 2 h. After crushing, the sample was sieved to less than 150 µm and kept in a desiccator until just before use. The prepared catalysts are referred to as 5PtY, 5PdY, and 5RhY.

Catalytic hydrocracking

The model compound (1 mmol) or RS Soluble (1 g), a catalyst (0.1 g) and cyclohexane solvent (50 mL) were put into an autoclave. The autoclave was replaced with hydrogen three times and then pressurized to 1 MPa with hydrogen. The mixture was heated up to 200 or 350°C with stirring at 200 rpm and reacted at the temperature for 2 h. After the reaction, the mixture was cooled to room temperature and filtered under vacuum with a membrane filter (a pore size of 10 µm). The filtrate obtained by hydrogenation of the model compound was analyzed by GC-MS (Shimadzu GCMS-QP2010SE).

Fig. 1 shows the flowchart of the hydrocracking of RS Soluble and the solvent fractionation using kerosene and tetrahydrofuran (THF). Cyclohexane-soluble component (CHS) was obtained by completely removing cyclohexane from the filtrate using a rotary evaporator. On the other hand, cyclohexane-insoluble component (CHI) was obtained by drying at 110°C until no weight loss of the filtration residue was observed. The yield of each component was calculated from the weight. The CHI yield was calculated by subtracting the weight of the charged catalyst from the weight of the filtration residue.

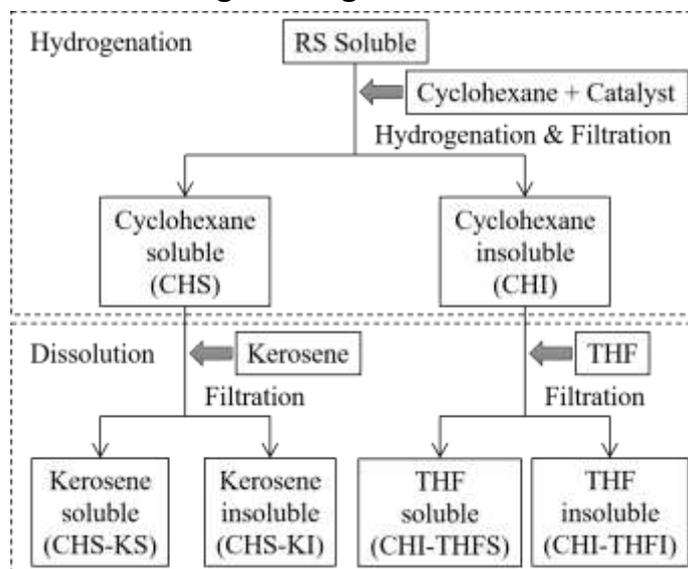


Fig. 1. Flowchart of hydrogenation and dissolution in kerosene and tetrahydrofuran (THF).

CHS (0.1 g) was added into 50 mL of kerosene and the mixture was stirred well. The mixture was filtered under vacuum through the membrane filter whose weight was measured in advance and the filtration residue was dried together with the filter at 110°C for more than 1 h. The filtration residue and the filter were weighed after cooling for 1 h in the desiccator. By repeating this drying and cooling cycle until the weight of filtration residue became constant, the yield of the kerosene-insoluble component (CHS-KI) was determined. The yield of kerosene-soluble component (CHS-KS) was calculated by subtracting the CHS-KI yield from the CHS yield.

All the recovered CHI was added into 50 mL of THF and the mixture was stirred well. By the same method as the determination of CHS-KI yield, the yield of THF-insoluble component (CHI-THFI) was calculated. The yield of THF-soluble component (CHI-THFS) was also calculated by subtracting the CHI-THFI yield from the CHI yield.

Characterization

The chemical form of catalyst was measured by X-ray diffraction (Rigaku Ultima IV). The XRD pattern was obtained using CuK α radiation at a divergence slit of 0.5°, a scan speed of 10° / min, 2 θ = 10-80°, and a sampling width of 0.02°. The acid properties (acidity and amount of acid sites) of catalyst were measured by a NH₃-TPD (temperature-programmed desorption) (MicrotracBEL BELCAT-B). Before the measurement, the catalyst was heated under He flow at 500°C for 1 h and cooled to 100°C. Then, NH₃ was adsorbed on the catalyst by flowing an NH₃ / He mixed gas for 30 min. The adsorbed NH₃ was desorbed up to 610°C at a rate of 10°C / min, and the desorbed amount was determined by TCD.

The calorific value measurement (Shimadzu CA-4PJ) and thermogravimetry (10°C / min up to 1000°C under N₂ flow, Shimadzu TGA-50) of the hydrocracked product were carried out.

RESULTS AND DISCUSSION

Characterization of catalyst

The XRD patterns for the catalysts after preparation and after use in hydrocracking at 350°C were the same as that for unused HY, indicating that the zeolite structure did not change and that the loaded metals remained finely dispersed.

Fig. 2 shows the NH₃-TPD profiles for the catalysts after preparation. In the case of HY, two peaks appeared at around 200°C (l-peak) and 400°C (h-peak). Here, the TPD profile was divided into two peaks, and the amount of each acid was calculated from the peak area (Table 2). The amounts of l-peak and h-peak of HY were calculated to be 1.6 mmol / g and 1.8 mmol / g, respectively. Generally, it is considered that the strong acid sites



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corresponding to h-peak contribute to the catalytic activity. The amount of strong acid (h-peak) for 5PtY was almost the same as that for HY. On the other hand, 5PdY and 5RhY had smaller amounts of strong acid than HY and 5PtY. In this study, the loading of the noble metal on HY zeolite was performed by the impregnation method. Because PdCl_2 and $\text{RhCl}_3 \cdot 3\text{H}_2\text{O}$ were used, Pd and Rh were present as cations (Pd^{2+} and Rh^{3+}) in the aqueous solution. The decrease in the amount of strong acid sites for 5PdY and 5RhY was probably due to the ion-exchange of Pd^{2+} and Rh^{3+} with H^+ on HY zeolite. Since Pt was present as an anion (PtCl_6^{2-}), it was considered that the amount of acid site did not decrease. From these results, it was concluded that the acidity for 5PdY and 5RhY was lower than that for HY and 5PtY.

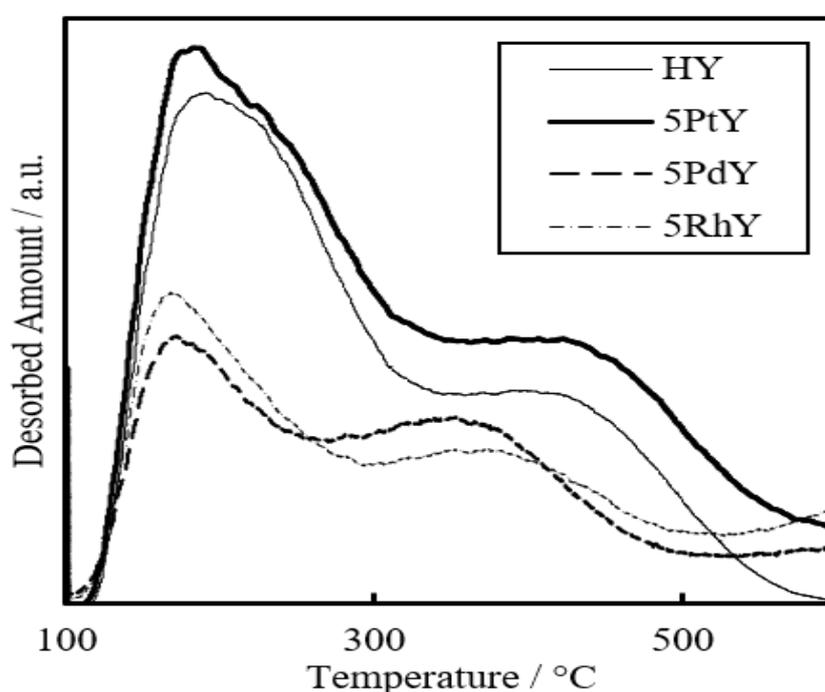


Fig. 2. NH_3 -TPD profiles of HY, 5PtY, 5PdY and 5RhY catalysts.

Table 2. Acid amounts (mmol/g) for HY, 5PtY, 5PdY and 5RhY catalysts.

	HY	5PtY	5PdY	5RhY
l-peak	1.6	1.5	0.4	0.6
h-peak	1.8	1.7	1.3	1.2

Hydrogenation of model compounds

Table 3 shows the hydrogenation conversion calculated from the peak area of GC-MS when the model compounds were hydrogenated using various catalysts. For anthracene (AN) and 9-phenylanthracene (PA), the hydrogenation conversion was 100 mol% for all catalysts. By contrast, there was a difference in the hydrogenation conversion of phenanthrene (PH), pyrene (PY), and 9,10-diphenylanthracene (DPA) depending on the catalyst species. In particular, in the cases of hydrogenation of PH and PY, the conversions for 5PdY were less than 13 mol%, while those for 5PtY were more than 88 mol%.

Table 3. *Hydrogenation conversion for model compounds.*

	Conversion (mol%)		
	5PtY	5PdY	5RhY
AN	100	100	100
PH	100	13	57
PY	88	5	61
PA	100	100	100
DPA	100	89	92

Moreover, Table 4 shows the number of hydrogen additions in the hydrogenation of the model compounds using various catalysts. In the case of hydrogenation of AN, the main product for 5PtY was tetradecahydroanthracene ($C_{14}H_{24}$) formed by the addition of seven hydrogen molecules to AN. On the other hand, the main product for 5PdY was 1,2,3,4,5,6,7,8-octahydroanthracene ($C_{14}H_{18}$) formed by the addition of four hydrogen molecules to AN. Also, in the hydrogenation of other model compounds, it was found that 5PtY produced compounds with a large addition number of hydrogen molecules, while 5PdY produced compounds with a small addition number of hydrogen molecules. From the above results, it became clear that 5PtY had the highest activity for hydrogenating polycyclic aromatic hydrocarbons. Therefore, 5PtY catalyst is expected to be effective for hydrogenation of the RS Soluble. However, no hydrocracked products were found at 200°C.

Table 4. *Number of hydrogen added in catalytic hydrogenation of aromatic hydrocarbons (●: Main component, △: Minor component).*

		Number of hydrogen added				
		+H ₂	+2H ₂	+3H ₂	+4H ₂	+7H ₂
AN	5PtY				△	●
	5PdY				●	△
	5RhY				△	●
PH	5PtY	△			●	△
	5PdY	△	△			
	5RhY	●	△		△	
PY	5PtY	●	●	△		
	5PdY	△				
	5RhY	●	△	△		
PA	5PtY		△		●	
	5PdY		●		△	
	5RhY		●		△	
DPA	5PtY		△		●	
	5PdY		●		△	
	5RhY		●		△	

**Hydrocracking of RS Soluble**

Fig. 3 shows the yield of each solvent-soluble component for the untreated RS Soluble and the products obtained by hydrogenation of RS Soluble at 200 or 350°C. In some cases, the total yield may be significantly smaller than 100 wt%, but the reason is unclear at this time (this may be because gas or low-boiling components could not be collected). The CHS-KS yield for the untreated RS Soluble was 18.5 wt%. Cyclohexane dissolves alkanes and alkenes up to C₃₀ and aromatic hydrocarbons such as benzene, naphthalene, phenanthrene, anthracene, and pyrene [21, 22]. In addition, among the aromatic hydrocarbons with side chain, it is reported that diethylxylene, methylnaphthalene, dimethylnaphthalene, methylanthracene are soluble in cyclohexane, but ethylnaphthalene, dimethylantracene are insoluble. Previously, the results of solvent extraction of low-rank coal revealed that the low-rank coal was composed of polycyclic aromatic hydrocarbons and aliphatic hydrocarbons associated by intermolecular interactions such as hydrogen bond, π - π interaction, and π -hydrogen bond interaction [21, 23, 24]. As described in the experimental section, the RS Soluble is a component dissolved in 1-MN at room temperature. Therefore, the RS Soluble does not contain a large amount of high molecular weight components but is considered to have a structure in which aliphatic hydrocarbons of short chain length and aromatic hydrocarbons up to four-rings are associated by the above-mentioned intermolecular interaction. Since the CHS-KS for the untreated RS Soluble is a component extracted at room temperature, this CHS-KS is considered to be a component that has been incorporated into the RS Soluble by the very weak intermolecular interaction. In addition, it was found that all the components insoluble in cyclohexane (CHI) were soluble in THF.

Hydrocracking of RS Soluble at 200°C resulted in 34-45 wt% of the CHS-KS yield, which was more than twice that for the untreated RS Soluble. In contrast, the CHI-THFS yield was greatly reduced from 81.5 wt% for the untreated RS Soluble to 48-56 wt% by reaction at 200°C. In Section 3.2, it was clarified that even if a catalyst was used, the hydrocracking of the model compound did not occur at 200°C. Therefore, the change in CHS-KS and CHI-THFS yields with increasing temperature is considered to be due to the complete or partial hydrogenation of aromatic hydrocarbons. However, in the hydrogenation at 200°C, the difference of the catalyst species had little effect on the CHS-KS yield.

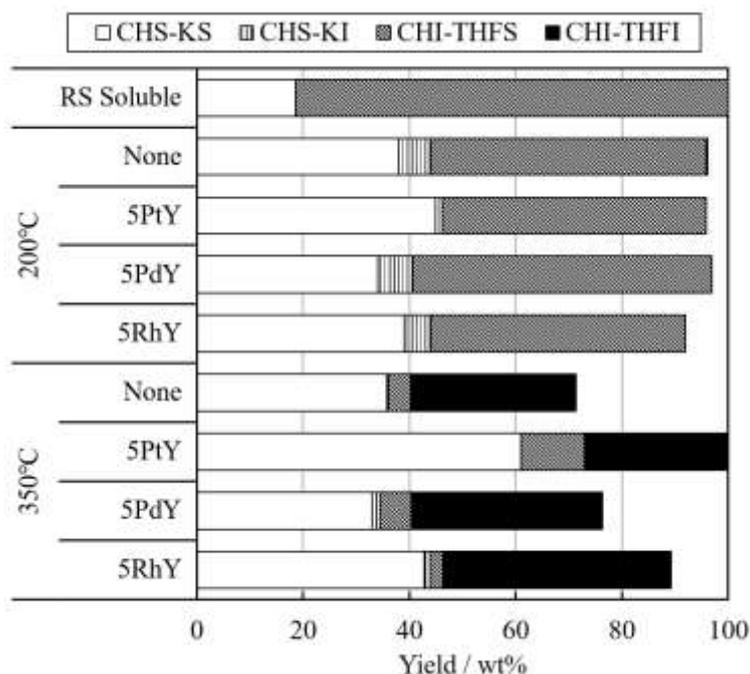


Fig. 3. Kerosene and THF solubility of RS Soluble and hydrocracked products.



When the RS Soluble was hydrocracked at 350°C, the CHS-KS yields varied greatly depending on the catalyst species. In the cases of no catalyst, 5PdY and 5RhY, the CHS-KS yield at 350°C was 33-43 wt%, which was almost the same as the CHS-KS yield at 200°C. On the other hand, in the case of 5PtY, the CHS-KS yield at 350°C increased significantly from that at 200°C, reaching 61 wt%. This indicates that 5PtY is effective in hydrocracking the RS Soluble at 350°C. Also, it was found that most of the CHI at 200°C was dissolved in THF, while the amount of THF-insoluble component increased at 350°C. This suggests that not only low molecular weight components but also high molecular weight components were produced by the hydrocracking of RS Soluble at 350°C. However, since the CHI-THFI yield was the lowest in the case of 5PtY, it was also found that the ability of 5PtY to produce the high molecular weight components was the lowest.

3.4. Properties of solvent extraction components

Fig. 4 shows the TG profiles of CHS and CHI-THFI obtained by hydrocracking using 5PtY, 5PdY and 5RhY at 350°C together with the TG profile of the untreated RS Soluble. The weight loss for CHS occurred at lower temperature than that for untreated RS Soluble, indicating that CHS obtained by hydrocracking at 350°C has lower molecular weight than the untreated RS Soluble. In particular, the weight loss for the CHS obtained by hydrocracking using 5PtY or 5RhY shifted to the lower temperature side by around 200°C than that for the untreated RS Soluble. This result also indicates that CHS obtained by hydrocracking with 5PtY or 5RhY is rich in small molecular weight components. Since the TG curves of CHI-THFI are almost the same, it is considered that similar components were produced regardless of the catalyst species. Comparing these TG curves with that for the untreated RS Soluble shows that decomposition occurs at a very high temperature. This also indicates that CHI-THFI is composed of high molecular weight components.

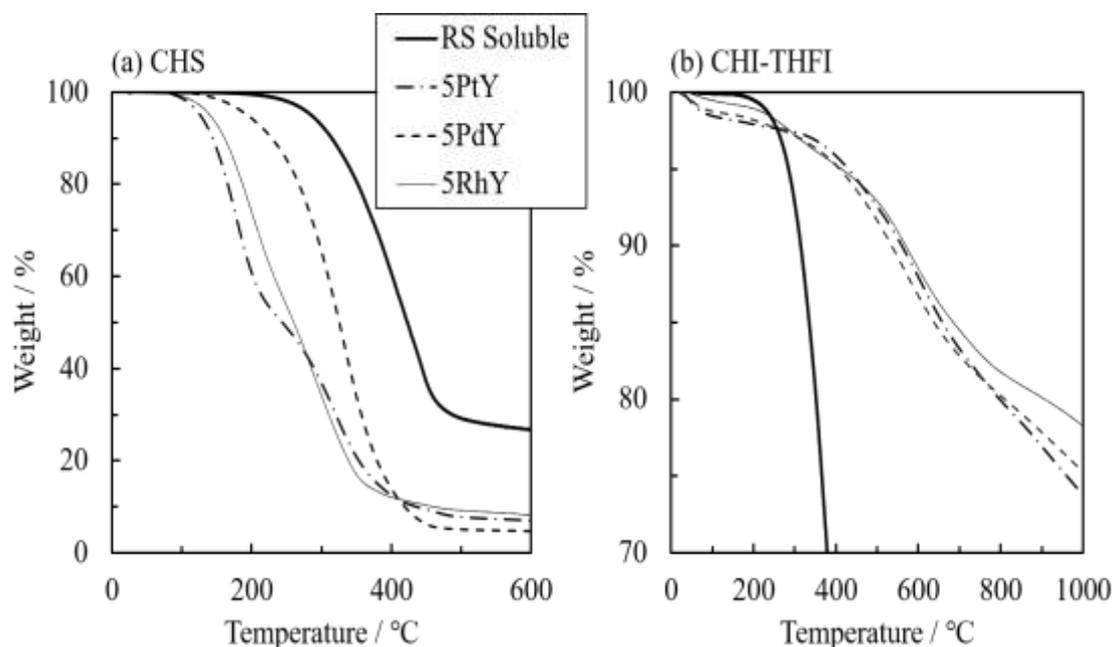


Fig. 4. TG curves of untreated RS Soluble and (a) CHS and (b) CHI-THFI obtained by hydrogenation at 350°C with 5PtY, 5PdY and 5RhY.

Table 5 shows the calorific values of CHS obtained by hydrocracking at 350°C using 5PtY, 5PdY and 5RhY. As shown in Table 1, the calorific value of untreated RS Soluble was 36.1 MJ / kg, while the calorific value of CHS was still high at 36-39 MJ / kg.



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Table 5. Calorific values of the CHS obtained by catalytic hydrocracking at 350°C.

	Calorific value (MJ/kg)
5PtY	39.4
5PdY	39.4
5RhY	36.4

CONCLUSION

Hydrocracking of RS Soluble was carried out using a noble metal-loaded Y zeolite catalyst. The CHS-KS yield for the untreated RS Soluble was only 18.5 wt%. When the RS Soluble was hydrocracked at 200°C, the CHS-KS yield was almost the same regardless of the catalyst species. The hydrocracking of the RS Soluble 350°C in the presence of 5PtY yielded the products containing a large amount of low molecular weight components, where the CHS-KS yield was 3.3 times larger than that of the untreated RS Soluble.

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