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Active Carbon from Residue on Wood Hydrolysis

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木材加水分解残渣からの活性炭

氏 家 雅 男**

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Introduction

It is obvious that the residue consisting mainly of lignin on wood hydrolysis should be effectively utilized for an economic success in this industry. Various studies have long been made on the trial uses of the lignin obtained in several types for adhesives,^{31),34)} soil mulch,³⁴⁾ a carrier of fertilizer,³⁴⁾ substitutes of tan³⁴⁾ or asphalt⁴⁰⁾, and on the testing gains of available chemicals by decomposition^{3)~11),18),21)} or grafting²⁵⁾ of the lignin. Some books concerning the chemistry of lignin containing its utilization have also been published.^{2),11),12),26),28),27),36),38),39)} On the other hand, the studies on preparation of active carbon from wood or decayed wood with some concentrated sulfuric acid or zinc chloride have been

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reported^{13)~16),22),23),30),43)} The author attempted to make the active carbon with concentrated sulfuric acid from the waste residue of wood hydrolysis which consists mainly of lignin. It is known that the vegetable materials are carbonized by dehydrating agents, such as sulfuric or phosphoric acid at a relative low temperature, and that this hydrated active carbon is obtained in a high yield and is effective for decolorization in moist state.^{1),13)~16),42)}

Since Japanese Industrial Standard (JIS) for the test method of the active carbon seems complicated and inaccurate¹⁹⁾, the author wanted to modify it by the application of a spectrophotometry¹⁷⁾³³⁾ and obtained the better results easily and accurately. TODA⁴²⁾ had also tried the similar experiments to this study.

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Material

A raw material used was a white birch (*Betula platyphylla var. japonica*) brought from Tomakomai, Hokkaido. The wood was barked and converted into the meals by a WILEY mill and they were sieved. The 20 to 40 mesh fraction of them was used for the preparation of a residue on hydrolysis, while the 60 to 100 mesh fraction was also used for wood analysis by standard procedure. Only the 40 to 60 mesh fraction was furnished for holocellulose determination by WISE's method⁴⁴⁾. The result of wood analysis is shown in **Table 1**.

Experimental

I. Wood Hydrolyses

The conditions of wood hydrolysis selected in this study were almost due to the proposal of Hokkaido Forest Products Research Institute.²⁴⁾

i) Pre-hydrolysis..... The 20 to 40 mesh fraction of the birchwood was treated with 1.3% sulfuric acid at 145°C for 30min to eliminate a main part of hemicelluloses. The liquor to wood ratio was maintained at 5. After the hydrolyzate obtained was removed, the residue was washed, dried and furnished for a main-hydrolysis.

ii) Main-hydrolysis.....The residue was again hydrolyzed with 80% sulfuric acid at a room temperature for 1.5 hr. The weight ratio of the chemical to the residue was 0.8 when the former was calculated as 100%.

iii) Post-hydrolysis.....The treated mixture was diluted to 50% concentration of the acid with water, and maintained with a frequent stirring at 100°C for 10min. To the resultant residue on the post-hydrolysis, a large quantity of water was added and the mixture was filtered, then the residue obtained was washed and dried.

iv) Estimation of carbohydrates in the two hydrolyzates.....An aliquot of the pre-and post-hydrolyzates was furnished for the estimation of carbohydrates by BERTRAND'S method and the reducing sugar was expressed as glucose. The rests of the hydrolyzates, on the other hand, were neutralized with barium carbonate to pH 6 and the filtrates were evaporated and paper-chromatographed using the solvent of n-butanol, benzene, pyridine and water (10:2:5:5)⁽⁴¹⁾ by a multiple ascending method⁽²⁰⁾. As a developer, an aniline hydrogen phthalate solution⁽³⁵⁾ was used for the detection of the carbohydrates.

v) Analysis of the residues on pre-and post-hydrolyses.....Both these residues were subjected to chemical determinations on ash, alcohol-benzene and 1% sodium hydroxide extracts and lignin. The content of pentosan and holocellulose was also determined in the residue on the pre-hydrolysis, and furthermore, the reducing sugar was estimated on the filtrate obtained from the lignin determination in the residues on the post-hydrolysis.

II. Preparation of Active Carbon

The specimens used were the residue on the post-hydrolysis. To 2 g of it, 10 g of 70 or 98 % sulfuric acid were added. The mixture was first heated in a water-bath at 80°C for 1 hr, then treated at a room temperature or in an electric oven with a rotating plate under the conditions as shown in Table 3. The products were ground in a mortar, filtered and washed perfectly with boiling distilled water to remove completely the sulfuric acid. The water content was determined by a usual method using a portion of this product, and the yields were calculated on the basis of the oven-dried residue.

III. Test Procedures for Adsorption Power of the Carbon

i) Methylene blue adsorption test..... According to JIS,⁽¹⁹⁾ 1.2 g of oven-dried methylene blue (G. R. Kanto Chemical Co.) was accurately weighed and dissolved with some distilled water and then diluted to 1,000 ml. An aliquot of the solution

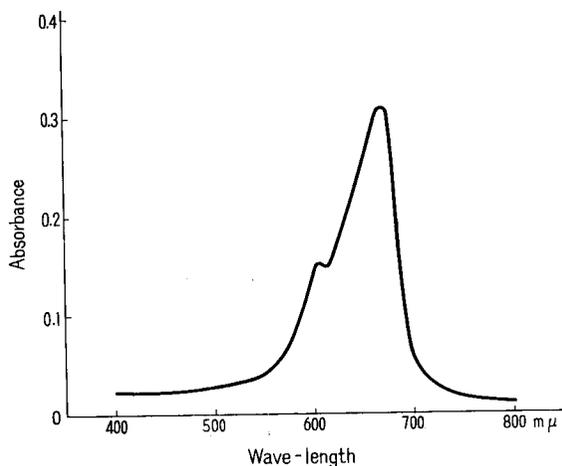


Fig. 1. Absorption curve of methylene blue solution.

was diluted to 1/1,000 concentration with water. An absorption curve of this solution was determined from 400 $m\mu$ to 800 $m\mu$ of the wave-length with a BECKMAN type spectrophotometer (Hitachi, EPU-2A),³³⁾ as shown in Fig. 1.

Absorbances were obtained at 609 $m\mu$ and 668 $m\mu$, of the wave-length the latter of which was the maximum one. This was therefore used for the preparation of a calibration curve, which is shown in Fig. 2.

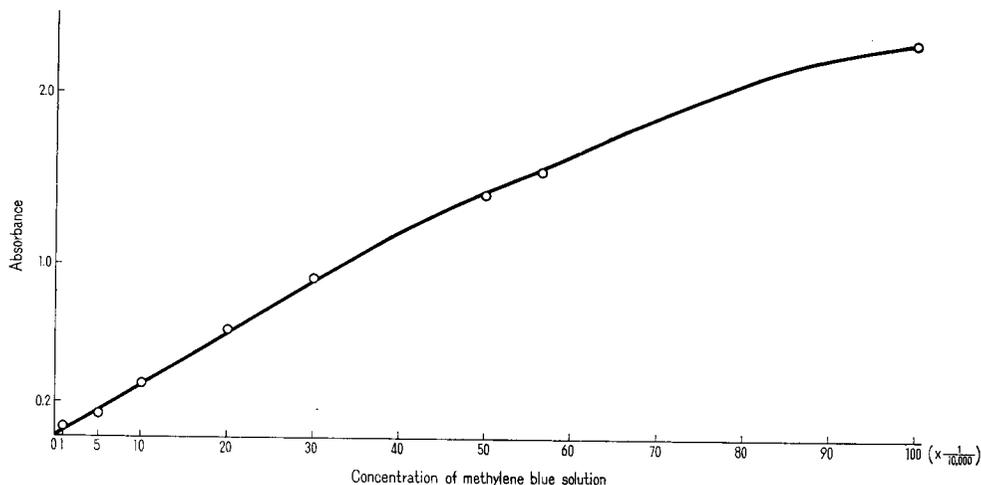


Fig. 2. Calibration curve of methylene blue solution at 668 $m\mu$.

In a 50-ml glass stoppered weighing bottle was placed 0.1 g equivalent weight based on oven-dry of each active carbon (because these hydrated active carbons are available for moist state, it is impossible to be directly weighed), to which 15 ml of 0.12% methylene blue solution and one drop of dilute hydrochloric acid (1:10) were added. Then this mixture was shaken by hand for 5 min at a room temperature, and filtered without suction. An aliquot of the filtrate decolorized was taken in a 1-cm thickness glass cell, and the concentration or the adsorption power of it was measured by the spectrophotometric method and determined with reference to the calibration curve. For comparison purposes, that of four commercial active carbons and the residue on the post-hydrolysis was similarly determined.

ii) Caramel adsorption test.....According to JIS, 12 g of oven-dried saccharose (E. P. Kanto Chemical Co.) were accurately weighed and dissolved with 48 ml of distilled water and 5 ml of dilute sulfuric acid (1:4). The solution was heated in a water-bath at 80 to 90°C for 30 min. While it was still hot, 2 g of sodium hydroxide were added to the solution, which was boiled on a wire gauze for 5 min. After cooling, it was neutralized and diluted to 120 ml with distilled water. This was again diluted to 1,500 ml on using for the adsorption test. An absorption curve of the dilute caramel solution was similarly determined

from 400 $m\mu$ to 800 $m\mu$ of the wave-length with the spectrophotometer and a maximum absorbance was obtained at 400 $m\mu$ within these ranges. The calibration curve obtained using this wave-length is shown in Fig. 3.

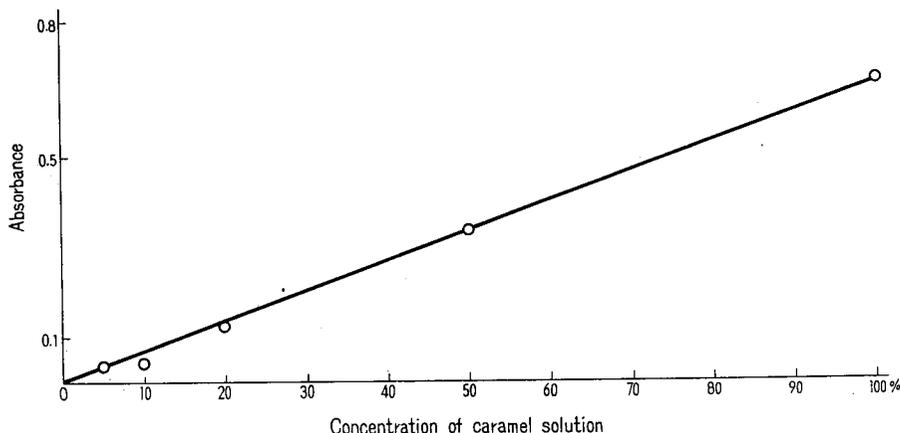


Fig. 3. Calibration curve of caramel solution at 400 $m\mu$.

Nine kinds of the active carbons used for the caramel adsorption test were selected from those of the methylene blue one. In a 100-ml glass stoppered weighing bottle was placed 0.1 g equivalent weight based on oven-dry of these active carbons, to which 40 ml of this caramel solution were added. The mixture was shaken for 15 min by a shaking machine, and filtered without suction. An aliquot of the filtrate decolorized was measured and determined by the same way as above described. For comparison purposes, that of two commercial active carbons was also similarly determined.

Results and Discussion

I. Chemical Properties of the Residues on Hydrolyses

The yield and chemical composition of the residues on the hydrolyses are shown in Table 1, together with that of the birchwood. Concerning to yield of the residue on the pre-hydrolysis, over one third of the material was dissolved. About 90% of pentosan and 50% of holocellulose were hydrolyzed. It was, therefore, calculated that about 18% of CROSS-BEVAN cellulose was also dissolved on the pre-hydrolysis. On the other hand, 77% of lignin was left in the residue. However, it seemed that the rest of lignin (23%) was partially decomposed and been extractable with alcohol-benzene solution. Solubility in 1% sodium hydroxide solution from the residue on the pre-hydrolysis was almost the same as that from the raw material, but the composition of it would have to be considerably different.

Concerning to the yield of the residue on the post-hydrolysis, over two third

Table 1. Yield and chemical composition of white birch-wood and the residues. (%)

	Yield	Ash	Solubility in			1% Sodium hydroxide	CROSS & BEVAN cellulose	Pen-tosan	Lignin	Holo-cellulose	Red-ucing sugar
			Alco-hol-ben-zene	Cold water	Hot water						
White birchwood	100	0.44	1.2	0.9	1.9	17.9	60.0	26.2	20.2	82.7	—
Residue on pre-hydrolysis	63.8	0.31 (0.20)	8.1 (5.2)	—	—	31.9 (20.4)	—	3.6 (2.3)	24.3 (15.5)	64.2 (41.0)	—
Residue on post-hydrolysis	32.7	0.42 (0.14)	19.7 (6.4)	—	—	61.4 (20.8)	—	—	57.2 (18.7)	—	15.0 (4.9)

Note, Values of the parentheses were based on the wood

of the raw material was dissolved. About 20% was extracted with alcohol-benzene solution, corresponding to 6.4% on basis of the material, which might contain a part of lignin decomposed. Furthermore, 1% sodium hydroxide solution extracted 61.4% of the residue, in which a considerable lignin might also be contained. The content of KLASON lignin was not so large, but the sum total of alcohol-benzene extract and lignin content amounted to 77%. The value of reducing sugar in the filtrate obtained on the lignin determination was 15% and this corresponded to only 5% on the basis of the material. These, on the whole, indicate the post-hydrolysis was almost perfectly carried out.

II. Carbohydrates in the Hydrolyzates

The results of reducing sugar and components of the hydrolyzates are shown in **Table 2**. The value of reducing sugar in the pre-hydrolyzate was 22.3%.

Table 2. Reducing sugar and components of the hydrolyzates.

	Reducing sugar (%)	Components				
		Rhamnose	Xylose	Mannose and Arabinose	Glucose	Galactose
Pre-hydrolyzate	22.3	+	+++++	+	+	+
Post-hydrolyzate	28.0	—	+	trace	+++++	—

It was shown that the components of it were mainly xylose derived from pentosan by paper-chromatography, and that the pre-treatment hydrolyzed hemicellulose, especially pentosan in the wood. Besides a large amount of xylose, smaller amounts of glucose, galactose, rhamnose and arabinose were detected together with some oligosaccharides which were the products caused by a partial hydrolysis.

On the other hand, the post-hydrolyzate contained 28% of reducing sugar on the basis of the residue on the pre-hydrolysis. The post-hydrolyzate consisted mainly of a large amount of glucose with some xylose, trace of mannose and oligosaccharides. It was shown that the post-treatment mainly hydrolyzed

cellulose in the residue on the pre-hydrolysis.

III. Yield of Active Carbon

Within the ranges of this study, the yield of the active carbons is shown in Table 3. The maximum and minimum yields were 78.8% and 65.4%, respectively. It decreased with the prolongation of reaction time and with

Table 3. Yield of active carbons. (%)

Conc. of H ₂ SO ₄ (%)	Temperature (°C)	Time (hr.)						
		2	4	8	14	24	48	72
70	Room-temp.	—	—	—	—	75.3	75.2	74.4
	120	—	—	75.8	75.9	72.5	—	—
	150	—	72.5	70.8	68.8	65.6	—	—
	170	72.3	70.1	68.4	65.4	—	—	—
98	120	—	—	77.4	78.8	75.1	—	—
	150	—	76.4	74.2	70.7	—	—	—

the elevation of the temperature. The yields of the carbon obtained with 98% sulfuric acid were rather higher than that with 70% one. Furthermore, active carbons prepared from another white birch under the same conditions show lower yields (53~63%)^{(13)~(16), (30)} than those from the residue, while the yield of an ordinary active carbon made from an oak (*Quercus mongolica var. grosseserrata*) with zinc chloride^{(22), (43)} is also as low as 48 to 16%. It is natural that the yields of the hydrated active carbons obtained from the residue on the post-hydrolysis were very high. These carbons are promising and characteristic in higher yields.

IV. Adsorption Power

i) Methylene blue solution.....The results of the adsorption power test with the methylene blue solution done by the spectrophotometric method is shown in Table 4, together with the conditions of preparations. Figures shown in this table are the value of relative concentrations obtained by the calibration curve (Fig. 2), when that of 0.12% methylene blue solution and water was expressed as 10,000 and 0, respectively. In case of a too high concentration of a solution filtered after the adsorption, it was diluted with distilled water to a proper intensity. Within these ranges of the active carbons tested, the highest adsorption power or the lowest concentration of the filtrate was given from the one obtained under the condition of 98% sulfuric acid at 120°C for 24hr. Table 4 also shows that the carbons of comparative high adsorption were prepared under such conditions as 70% sulfuric acid at 120°C for 14 and 24hr, at 150°C for 8, 14 and 24hr and at 170°C for 4 and 8 hr, while the carbons obtained by treating at the room temperature showed lower adsorption powers. From these results, it is inferred that the methylene blue adsorption power of

Table 4. Results of adsorption power test with methylene blue solution.

Conditions prepared		Time (hr.)						
Conc. of H ₂ SO ₄ (%)	Temperature (°C)	2	4	8	14	24	48	72
70	Room temp.	—	—	—	—	5010	6470	4590
	120	—	—	430	9	3	—	—
	150	—	38	7	4	5	—	—
	170	15	5	5	56	—	—	—
98	120	—	—	100	140	1	—	—
	150	—	118	58	48	—	—	—

Note, These figures are the relative values when the concentration of 0.12% methylene blue solution and water was expressed as 10,000 and 0, respectively.

the carbons was high when the residue was treated with 70% sulfuric acid at 120°C to 150°C of the temperature for a longer time, and also at higher temperature for a shorter time. When the treatment of 70% sulfuric acid was compared with that of 98% one, the former generally gave better results except some examples. The adsorption power of the residue itself on the post-hydrolysis and four commercial active carbons done for a comparison purpose is shown as follows:

		Adsorption power
The residue		7,140
Commercial active carbon	A	6
"	B	1
"	C	22
"	D	17

The residue itself naturally showed the lowest adsorption power. The data above mentioned describe that the better carbons prepared in this study were almost the same as the commercial active carbons.

ii) Caramel solution.....The results of the adsorption power test with the caramel solution done by the spectrophotometric method is shown in **Table 5**, together with the preparing condition selected and their adsorption powers of the methylene blue solution. Figures of concentrations of filtrates shown in this table are the values of relative concentration (absorbance), obtained by the calibration curve (**Fig. 3.**) when the concentration (absorbance) of the caramel solution used and water was expressed as 100% (0.666) and 0% (0), respectively. The highest adsorption power of the carbon, or the lowest concentration of the filtrate was given from a commercial active carbon. Within these ranges of the active carbons selected, all the carbons showed a considerably lower adsorption power.

The carbon obtained by the treatment of 98% sulfuric acid at 150°C for 4 hr

Table 5. Results of adsorption power test with caramel solution.

Conditions prepared			Concentration of filtrate in methylene blue test	Caramel adsorption test	
Conc. of H ₂ SO ₄ (%)	Temperature (°C)	Time (hr.)		Concentration of filtrate (%)	Absorbance
{Caramel solution (JIS)}				(100)	(0.666)
70	Room temp.	48	6,470	87.8	0.587
"	"	72	4,590	86.2	0.583
"	120	24	3	41.5	0.276
"	150	14	4	37.6	0.257
"	170	4	5	36.8	0.245
98	120	8	100	39.3	0.260
"	"	14	140	37.0	0.247
"	"	24	1	37.3	0.249
"	150	4	118	35.0	0.235
Commercial active carbon		B	1	7.2	0.049
"		D	17	9.5	0.064

showed the highest of all prepared. There were, however, generally little differences among the active carbons except the one prepared at the room temperature. The adsorption power order of the carbons by the caramel solution test, furthermore, did not always coincide with that by the methylene blue solution test. The reason is not understood at present why the caramel adsorption power of the carbons prepared is extremely inferior to the methylene blue one, but it might depend on the different sizes between the two molecules of methylene blue and caramel or the different mechanism in the adsorption process.

V. Comparisons of the Method of these tests with that of JIS

i) Comparison of the methylene blue test.....According to JIS⁽¹⁹⁾, an adequate volume of 0.12% methylene blue solution is added into a test tube with 0.1g of an active carbon, and the mixture was shaken for 5 min and filtered. The filtrate is compared, as widely known, with the standard solution by naked eye. In case of excess or deficiency, this test should be repeated to an adequate concentration. When the color of the filtrate coincides with that of the standard, the total volume is expressed as adsorbed volume of the solution. JIS defines that an active carbon of first class adsorbs over 12 ml of the methylene blue solution; second class, over 10 ml; and third class, over 8 ml, respectively. The relation between expressions of the spectrophotometric method and JIS method is shown in Fig. 4. If a concentration of a filtrate is given below 2,000 with the spectrophotometry, the adsorbed volume of the carbon belongs to first class of JIS method, and if over 4,650, it does not stand JIS. It is clear that all the hydrated active carbons prepared from the residue with concentrated sulfuric acids by heating belong to first class of JIS.

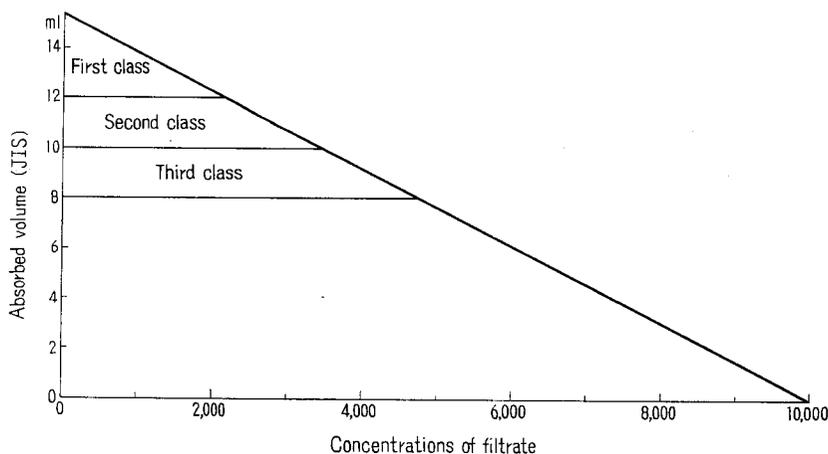


Fig. 4. Relation between expressions of the spectrophotometric method and JIS method in methylene blue solution test.

ii) Comparison of the caramel solution test.....According to JIS¹⁹⁾, percent of decolorization of the caramel solution is shown by measuring the filtrate with a DUBOSCQ colorimeter, after 40 ml of the solution were shaken with 0.1 g of an active carbon for 15 min. JIS defines that an active carbon of first class amounts to over 94% of decolorization, namely 6% of concentration of the filtrate; second class, over 90%; and third class, over 85%, respectively. The relation between expressions of the spectrophotometric method and JIS method

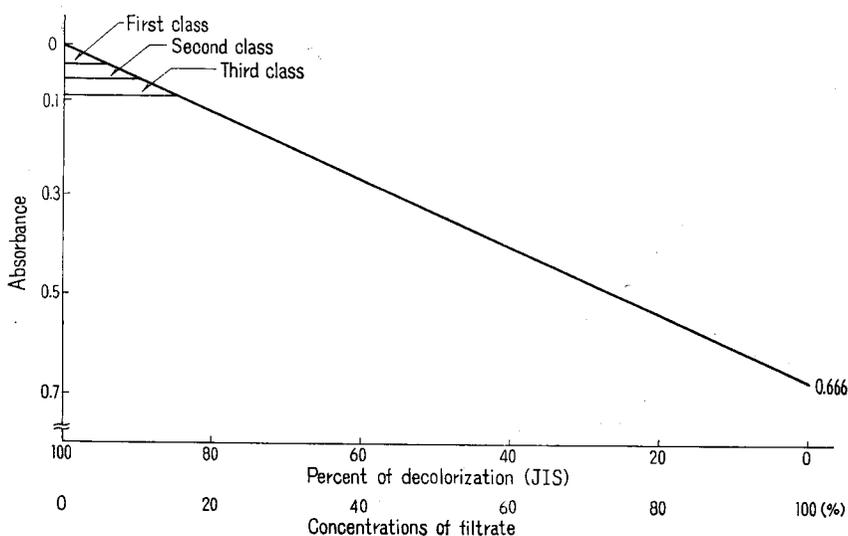


Fig. 5. Relation between expressions of the spectrophotometric method and JIS method in caramel solution test.

is shown in Fig. 5. If an absorbance of a filtrate with spectrophotometry is below 0.040, the carbon belongs to first class of JIS method (over 94%), and if over 0.096, it does not stand JIS (85%). Since the filtrate of a commercial active carbon was given in 0.049 of the absorbance (Table 4), the percent of decolorization of it belongs to second class of JIS. However, no active carbons prepared in this study amounted to these classes of JIS.

Conclusions

Hydrated active carbons were prepared under various conditions with concentrated sulfuric acid methods from the residue on wood-hydrolysis, and tested on the adsorption power of the methylene blue and caramel solutions according to a new spectrophotometric method. It is concluded that the active carbons prepared are promising and characteristic in higher yields than those obtained from wood directly with sulfuric acid or a different chemical, and that the carbons have a higher adsorption power on the methylene blue test, but greatly lower power on the caramel test. The carbon obtained under conditions of 98% sulfuric acid, at 120°C and for 24 hr is the most excellent adsorption power with the methylene blue solution. Furthermore, most kinds of the carbons could show the first class in the methylene blue test of JIS. However, the reason why the carbons showed an extremely lower adsorption power with the caramel solution is not understood and should be inquired in future. It might also seem at present that the differences in the adsorption powers between the two solutions depend on the different size of the two molecules or the different mechanism in the adsorption process. From the comparison of the spectrophotometric method with JIS method, it is shown that the new method using the spectrophotometer was very easy and accurate.

Summary

Hydrated active carbons were prepared with concentrated sulfuric acid from a residue on hydrolysis of a white birchwood grown in Hokkaido. To obtain the residue, pre, main and post-hydrolysis were done according to the condition investigated at Hokkaido Forest Products Res. Inst. From the residue, various active carbons were prepared in higher yields using conditions of 70 or 98% sulfuric acid, at a room temperature, 120, 150 and 170°C, for periods from 2 to 72 hr at each concentration and temperature levels. The adsorption power test with methylene blue and caramel solutions was carried out using a new spectrophotometric method instead of JIS method.

The active carbons obtained showed an excellent adsorption power in case of the methylene blue but not in case of the caramel solution. On the test procedure, it was shown that the new method with the spectrophotometer was easy and accurate.

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要 約

北海道産シラカンバ (*Betula platyphylla var. japonica*) を原料とし、加水分解をおこなった後えられた残渣から、濃硫酸を用いて水和活性炭を製造した。加水分解残渣をうるため、まず木粉を北海道林産試験場の条件に従い、前、主、および後加水分解した。その残渣から 70 あるいは 98% 硫酸を用い、常温、120、150 および 170 °C で 2~72 時間処理して種々の活性炭を調製した。この性能試験として吸着力を分光光電比色法によって測定した結果、メチレン・ブルー試験では極めて優良な結果が示されたが、カラメル試験ではすぐれた結果はまったくえられなかった。またその際用いた分光光電比色法は JIS 法にくらべて容易で、正確にその結果を示した。なお本研究をおこなうに当り、北海道大学教授、理学博士半沢道郎氏と同助教授、林学博士里中聖一氏には種々御指導と御援助をいただいた。またこの研究をすすめるに当り北海道から助成金が交付されている。ここに深甚の謝意を表す。