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Preparation of Activated Carbon with Phosphoric Acid from Waste Liquors, Waste Sludges and Waste from Deinking*

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パルプ廃液，パルプ粕，脱墨廃物からリン酸賦活
による活性炭の製造

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1. Introduction

For the improvement of environmental problems, activated carbon is expected largely. On the occasion of the purification of pulping waste liquor, the final treatment may rely on the application of the activated carbon.^{2,12)}

In case of the production of pulp and paper, pretty much wastes are produced. If the wastes are used as the raw material of activated carbon, and the pulping waste liquor is purified with the activated carbon, it may be said that "One stone hits two birds".

Now, there are two methods³⁾ for the industrial production of activated carbon. One is made from charcoal or coal at about 900°C activated with over heated steam. The other is made from organic matters such as sawdusts at about 650°C activated with zinc chloride.

The first method is the treatment at high temperature, generally it consumes much calories, so the heat management is important. As there is not environmental pollution problem, the share is increasing.^{4,5)}

In the second method, there are some problems such as the generation of white smoke derived from zinc chloride, and there is the appointment of zinc ion in waste liquor as a harmful substance, which should be controlled strictly.

In our laboratory, sawdusts etc. were dehydrated at 170°C by use of the sulfuric acid and high yield activated carbon was obtained.⁶⁾ But there is a weak point which is the generation of sulfur dioxide during the activation.

As the results of surveying the non-pollution dehydrant, phosphoric acid was adopted⁷⁾. In this case, harmful gas is not detected during the heating at 150° to 200°C. And 90% or more of phosphoric acid can be recovered by the washing.⁸⁾ Even if there is a leak, which will not give the enormous pollution.

In our laboratory, the activation of an oak (*Quercus mongolica* var. *grosseserrata*) with phosphoric acid at 190°C for 8 hours gave the high yield activated carbon such as 54%.⁹⁾

The adsorption ability of this activated carbon is similar to that of the commercial carbon. Then the yield is pretty higher than 8% of the carbon activated by steam method and 33% by zinc chloride method.⁹⁾

In this paper, various activated carbons were prepared from KP waste liquor, SP waste liquor, waste from deinking and waste sludges with phosphoric acid. Then the adsorptive characteristics were observed.

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2. Experimental

2.1 Materials

Pulping waste used for these activated carbons are KP waste liquor from a white birch (*Betula platyphylla* var. *japonica*), KP and SP waste liquor from a spruce (*Picea glehnii*), pulp waste sludge and waste of deinking from H Paper Co. in K city located in the eastern Hokkaido and pulp waste sludge from J Paper Co. in the same city.

2.2 Preparation of waste liquors

KP waste liquors were prepared by the digestion from white birch at liquor ratio 4, from the spruce at liquor ratio 6, with active alkali 16%, sulphidity 25%.⁹⁾

Chips of 500 grams (O. D. equivalent) were digested in a 4-liter autoclave at 170°C, rising time 60 minutes, holding time 90 minutes. As for the SP method, total SO₂ was 6%, combined SO₂ was 5%, free SO₂ was 1%, pretreatment by water digestion was 60 minutes, digested at 140°C, rising time was 60 minutes and holding time was 180 minutes.¹⁰⁾

After cooling, the waste liquors were separated from the pulps.

2.3 Analysis of waste liquor

Total solid material is determined as follows: Ten ml of waste liquor were taken in a crucible, and evaporated on a water bath for about 2 hours until the liquid did not flow out. Then, it was dried in an electric oven for 24 hours. After cooling it was weighed and total solid (g/l) was calculated.

Ash: Ammonium nitrate (25 w/v%) was added to the above-mentioned total solid until the soaking, the volatile matter was evaporated with a weak burner, then it was heated in an electric oven at 600±25°C for 30 minutes. After cooling it was weighed and the ash content was calculated.

COD: Chemical Oxygen Demand was determined with KMnO₄ at 100°C based on the JIS K 0102.¹⁰⁾

pH: This value was determined by H·HM 7 type pH meter.

2.4 Preparation of activated carbon

Water in the above-mentioned waste liquors, waste sludges and waste from deinking was evaporated on the water bath and various solids were prepared. Every solid of O. D. 2.0 g equivalent was placed in a 200-ml tall beaker, and 10 g of 78% conc. phosphoric acid were added and heated on a horizontal rotary disc in an electric oven under four conditions.

Carbonized substance was transferred in a mortar, ground well, washed with distilled water until pH 5 using a Buchner funnel.

The yield of activated carbon was calculated from the double experiments.

2.5 Adsorption for methylene blue

Each activated carbon of O. D. 0.1 g equivalent was taken in a 25 ml-sample bottle, 20 ml of 0.3% methylene blue solution was added and shaken for 30 minutes.

After natural filtration, the filtrate was diluted to 4000 times, the absorbance at 665 nm was observed by an UV and visible spectrophotometer. Based on the blank test, the adsorptive percent was obtained.

For comparison, two commercial activated carbons from K Co. and W Co. were also tested.

2.6 Adsorption for waste liquor

Experimental method was about the same as the above-mentioned one for methylene blue. The factors of dilution for each liquor were shown in Table 1.

Table 1. Factors of dilution in case of the adsorption for pulping waste liquor

Kind of waste liquor	Lignin	Color
KP waste liquor from <i>Betula platyphylla</i> var. <i>japonica</i>	20	50
KP waste liquor from <i>Picea glehnii</i>	20	50
SP waste liquor from <i>Picea glehnii</i>	50	10

2.7 Ultimate analysis

Carbon and hydrogen contents of raw materials and some activated carbons prepared were analyzed by the asking at the Instrumental Analysis Center, Hokkaido University.

3. Results and Discussion

3.1 Chemical properties of waste liquors

The results are shown in Table 2. A literature¹²⁾ indicates that COD of KP and SP are 160,000 and 100,000 and pH are 12.9 and 1.0 respectively. The results in this study show that the values of COD were pretty lower than the data described in the literature, values of pH were similar to those in the literature.

Table 2. Chemical properties of waste liquors

Kind of waste liquor	Total solid (g/ℓ)	Ash (g/ℓ)	COD (ppm)	pH
KP waste liquor from <i>Betula platyphylla</i> var. <i>japonica</i>	191.4	48.3	72030	11.3
KP waste liquor from <i>Picea glehnii</i>	152.6	53.7	71295	13.1
SP waste liquor from <i>Picea glehnii</i>	81.4	30.0	55370	1.4

3.2 Yield of activated carbon

The results are shown in Table 3. The maximum yield was 80% from the waste from deinking, then 73% from waste sludge of J Co. The yields of the activated carbons from other samples were in the order of waste sludge of H Co. >

Table 3. Yield of activated carbon (%)

Raw material	Condition of activation				Average	Rank
	170°C		190°C			
	1 hour	2 hours	1 hour	2 hours		
KP waste liquor — <i>Betula p.</i>	33 (37, 29)	32 (35, 29)	35 (40, 30)	36 (36, 35)	34	7
KP waste liquor — <i>Picea g.</i>	37 (38, 35)	36 (36, 36)	39 (41, 36)	40 (40, 39)	38	6
SP waste liquor — <i>Picea g.</i>	62 (62, 62)	63 (65, 61)	61 (65, 57)	61 (61, 60)	62	4
Waste from deinking	76 (76, 76)	84 (84, 83)	81 (84, 77)	80 (81, 79)	80	1
Waste sludge — H Co.	66 (70, 62)	67 (68, 66)	66 (66, 65)	67 (70, 63)	67	3
Waste sludge — J Co.	73 (75, 70)	74 (74, 73)	71 (72, 70)	74 (78, 69)	73	2
Oak sawdust — <i>Quercus m.</i>	56 (56, 56)	57 (59, 55)	61 (63, 59)	57 (60, 54)	58	5
Average	58	59	59	59	59	

SP waste liquor from the spruce > oak sawdust > KP waste liquor from the spruce > KP waste liquor from the white birch.

The minimum value of 34% is less than the half of the yield at the waste from deinking. Four conditions composed of temperature and time for activation did not give the effects on the yield of activated carbon.

All average value was 59%, which was similar to 58% from the oak sawdust.

3.3 Adsorption for methylene blue

The results are shown in Table 4. The maximum value was 63% of the activated carbon from W Co., then followed by 50% from the oak sawdust, 48% from the spruce-SP waste liquor, 42% from K Co. and 39% from H Co.-waste

Table 4. Adsorption for methylene blue (%)

Raw material	Condition of activation				Av.	Rank
	170°C		190°C			
	1 hr	2 hr	1 hr	2 hr		
KP waste liquor — <i>Betula p.</i>	26	10	12	10	15	7
KP waste liquor — <i>Picea g.</i>	0	6	1	0	2	9
SP waste liquor — <i>Picea g.</i>	33	54	54	51	48	3
Waste from deinking	11	10	4	4	7	8
Waste sludge — H Co.	46	33	31	38	39	5
Waste sludge — J Co.	26	24	14	27	23	6
Oak sawdust — <i>Quercus m.</i>	49	53	48	50	50	2
Commercial activated carbon — K Co.					42	4
Commercial activated carbon — W Co.					63	1
Average	27	27	23	26	53	
Rank	2	2	5	4	1	

Table 5. Adsorption for KP waste liquor from *Betula platyphylla* var. *japonica* (%)

Raw Material	170°C				190°C				Average		Rank			
	1 hour		2 hours		1 hour		2 hours		Lig. Col.		Lignin	Color		
	Lig.	Col.	Lig.	Col.	Lig.	Col.	Lig.	Col.	Lig. Col.					
KP waste liquor — <i>Betula p.</i>	-45	-105	-38	-102	-50	-71	-19	-49	-38	-82		9	9	
KP waste liquor — <i>Picea g.</i>	-47	-38	-21	-75	-35	-21	0	49	-26	-21		8	8	
SP waste liquor — <i>Picea g.</i>	10	78	22	64	22	76	28	72	21	73		3	2	
Waste from deinking	5	9	5	21	5	16	7	36	6	21		7	7	
Waste sludge — H Co.	21	92	20	48	15	55	27	67	21	66		3	3	
Waste sludge — J Co.	9	22	12	40	8	42	18	63	12	42		6	5	
Oak sawdust — <i>Quercus m.</i>	25	81	32	76	26	73	34	76	29	77		2	1	
Commercial activated carbon — K Co.											20	24	5	6
Commercial activated carbon — J Co.											39	50	1	4
Average	-3	20	5	10	-1	24	14	45			30	37		
Rank	5	(4)	3	(5)	4	(3)	2	(1)			1	(2)		

Table 6. Adsorption for SP waste liquor from *Picea glehnii* (%)

Raw Material	170°C				190°C				Average		Rank			
	1 hour		2 hours		1 hour		2 hours		Lig. Col.		Lignin	Color		
	Lig.	Col.	Lig.	Col.	Lig.	Col.	Lig.	Col.	Lig. Col.					
KP waste liquor — <i>Betula p.</i>	-7	-50	-6	-32	0	-2	-12	-121	-6	-51		9	6	
KP waste liquor — <i>Picea g.</i>	-11	-146	0	-12	-1	-45	0	-31	-3	-59		8	7	
SP waste liquor — <i>Picea g.</i>	-5	-20	6	0	6	-6	13	7	5	-5		4	5	
Waste from deinking	0	-2	0	-1	0	4	2	0	1	0		7	4	
Waste sludge — H Co.	3	1	8	10	5	4	4	6	5	5		4	2	
Waste sludge — J Co.	-3	-1	6	7	3	4	8	6	4	4		6	3	
Oak sawdust — <i>Quercus m.</i>	5	16	10	15	10	14	19	8	11	13		3	1	
Commercial activated carbon — K Co.											12	-230	2	9
Commercial activated carbon — W Co.											28	-82	1	8
Average	-2	-29	3	-2	3	-4	5	-18			20	-106		
Rank	5	(4)	3	(1)	3	(2)	2	(3)			1	(5)		

sludge and so on. The minimum value was 2% of the one from the spruce-KP waste liquor.

3.4 Adsorption for KP waste liquor from a white birch

The results are shown in Table 5. As regards the adsorption for lignin, the maximum value was 39% of the activated carbon from W Co., next was 29% of the one from the oak sawdust, then SP waste liquor from the spruce, waste sludge from H Co. and the one from K Co.. The carbons from KP waste liquor of white birch and spruce showed the minus values. This means the incomplete formation of activated carbon, because the alkaline fraction neutralized the acidic property of phosphoric acid.

As for the decolorizing power, an activated carbon from the oak sawdust occupied the first position showing 77%. The carbon from the spruce SP waste liquor was the second showing 73%. These values suppressed the abilities of commercial activated carbons.

3.5 Adsorption for SP waste liquor from a spruce

The results are shown in Table 6. In case of the adsorption of lignin, the activated carbon from W Co. occupied the first position indicating 28%, next the activated carbon from K Co., then the one from the oak sawdust. The others were pretty low abilities.

In case of the adsorption of color, the activated carbon from oak sawdust occupied the first position, next the one from the waste sludge of H Co. then the carbon also from the waste sludge from J Co..

3.6 Adsorptive characteristics

The adsorptive rank of various activated carbons prepared under various conditions is shown in Table 7. For the adsorption of methylene blue, the condition at 170°C seems superior to that at 190°C. For the adsorption of pulping waste

Table 7. The adsorptive rank of various activated carbons prepared under various conditions

Adsorbate	170°C		190°C		Commercial activated carbons
	1 hour	2 hours	1 hour	2 hours	
Methylene blue	2	2	5	4	1
KP Lignin	5	3	4	2	1
KP Color	4	5	3	1	2
SP Lignin	5	3	3	2	1
SP Color	4	1	2	3	5
Total of KP & SP	18	12	12	8	9
Total of KP, SP & Mb	20	14	17	12	10
Rank	5	3	4	2	1

liquor, on the contrary the condition at 190°C seems superior to that at 170°C. Especially, the condition of 190°C-2 hours is best, next 190°C-1 hour and 170°C-2 hours, and 170°C-1 hour is worst.

Total consideration on both the adsorption of methylene blue and that of pulping waste liquor indicates that the condition of 190°C-2 hours is best, next 170°C-2 hours, next 190°C-1 hour, and the worst is 170°C-1 hour.

All consideration including the commercial activated carbons shows that commercial ones are best.

But there are special adsorptive characteristics, namely, the most useful activated carbon for the color of SP waste liquor may be prepared under the condition of 170°C-2 hours. Then the most useful carbon for the color of KP waste liquor may be obtained under the condition of 190°C-2 hours.

The weak point of the commercial activated carbons is the adsorption for the color of SP waste liquor. The SP waste liquor seems to disperse the micro carbon particles and to cause the filtration through a hard filter paper such as No. 5 C.

3.7 Elementary composition of activated carbon

The results are shown in Table 8. Ash contents of raw material ranged from 0.8% (oak) to 65% (waste sludge from J Co.), and it varied by activation from 3% (KP waste liquor-*Betula*) to 68% (waste sludge from J Co.).

Table 8. Elementary composition of activated carbon and the raw material

Specimen	Original				Ash free		
	Ash	C	H	O etc.	C	H	O etc.
Activated carbon from KP (<i>Betula p.</i>) waste liquor	3.48	63.20	4.97	28.35	65.48	5.15	29.37
Raw material	48.25	39.08	4.06	8.61	75.52	7.85	16.63
Activated carbon from KP (<i>Picea g.</i>) waste liquor	8.96	62.61	5.18	23.25	68.77	5.69	25.54
Raw material	53.73	35.33	3.81	7.13	76.36	8.23	15.41
Activated carbon from SP (<i>Picea g.</i>) waste liquor	9.82	60.10	3.78	26.30	66.64	4.19	29.17
Raw material	29.99	45.29	4.74	19.98	64.69	6.77	28.54
Activated carbon from the waste of deinking	54.35	39.40	3.82	2.43	86.31	8.37	5.32
Raw material	51.42	34.54	3.87	10.17	71.10	7.97	20.93
Activated carbon from the waste sludge of H Co.	45.34	36.68	2.90	15.08	67.11	5.31	27.58
Raw material	40.82	30.37	3.94	24.87	51.32	6.66	42.02
Activated carbon from the waste sludge of J Co.	67.91	27.67	2.16	2.26	86.23	6.73	7.04
Raw material	65.33	16.68	2.40	15.59	48.11	6.92	44.97
Activated carbon from oak	8.58	64.20	3.59	23.63	70.23	3.93	25.84
Raw material	0.83	47.55	6.08	45.54	47.95	6.13	45.92

Carbon content of raw material ranged from 17% (Waste sludge from J Co.) to 48% (*Quercus m.*), and it varied by activation from 28% (Waste sludge from J Co.) to 64% (*Quercus m.*). The value of 64% is similar to the data in previous paper.⁹⁾

When these values are calculated on basis of ash free, the carbon content of raw material ranged from 48% (*Quercus m.*) to 76% (KP waste liquor from *Picea g.*), and the values varied by activation from 65% (KP waste liquor from *Betula p.*) to 86% (Waste from deinking). The carbon content of 86% contained in the activated from the waste from deinking is fairly high value, but the adsorptivity of that activated carbon is not excellent, meaning that the carbon in the waste from deinking is too rigid to be bored to get suitable pores with phosphoric acid under these weak conditions.

4. Conclusion

Excellent activated carbons could be obtained from the SP waste liquor of *Picea glehnii* and the waste sludge from H. Co. as well as the oak sawdust.

Activated carbon from the waste of deinking showed the high yield but the adsorptivity was low, meaning the hard structure of the carbon obtained by deinking.

Activated carbon from KP waste liquors indicated low yield and low adsorptivity. The results must be derived from the neutralization of phosphoric acid with the alkaline fraction of KP waste liquor. There may be an opportunity to get high grade activated carbon after the neutralization with an acid as pre-treatment.

Summary

1) Various activated carbons were prepared from two KP waste liquors, SP waste liquor, waste from deinking, two waste sludges from two paper mills and an oak (*Quercus mongolica* var. *grosseserrata*) with phosphoric acid under the conditions of 170°C to 190°C and 1 to 2 hours.

2) The yield ranged from 32% (KP waste liquor from a birch) to 84% (waste from deinking), and the average was 59%. These values are superior to these from ordinary methods such as steam or zinc chloride.

3) As regards the condition of preparation, 190°C-2 hours is the best, next 170°C-2 hours, then 190°C-1 hour and the lowest is 170°C-1 hour. Commerical activated carbons were superior to that prepared in our laboratory.

4) In case of the adsorption of methylene blue, the carbons prepared at 170°C are better than those prepared at 190°C.

5) On the contrary, in case of the adsorption for waste liquor, the carbons prepared at 190°C are better than those prepared at 170°C.

6) As for the adsorption of the color of KP waste liquor, the carbon prepared at 190°C-2 hours was best.

7) As for the adsorption of the color of SP waste liquor, the carbon prepared at 170°C-2 hours was best.

8) Generally, the commercial activated carbons are useful for the adsorption of methylene blue, lignin and color of KP waste liquor, but very weak for the color of SP waste liquor.

9) The activated carbons prepared from SP waste liquor of *Picea glehnii* and waste sludge from H Co. as well as *Quercus mongolica* var. *grosseserrata* indicated the excellent ability similar to that of the commercial activated carbons.

10) The carbon content of these activated carbons ranged from 28% (waste sludge from J Co.) to 64% (oak). On basis of ash free, these values ranged from 65% (KP waste liquor from white birch) to 86% (Waste from deinking).

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要 約

1) 紙パルプ産業からは、大量の廃棄物が排出される。これらから活性炭をつくり、その汚濁排水を浄化すれば、一石二鳥であろう。そこで、シラカンバとアカエゾマツを原料とするクラフトパルプ廃液、アカエゾマツを原料とする亜硫酸パルプ廃液を実験室で調製した。さらに、道東K市のH・J両製紙工場から排出されるパルプ粕、H工場の脱墨廃物、比較のためにミズナラ木粉を原料として、リン酸を用いて、170°Cまたは190°Cで1~2時間加熱することによって、活性炭を製造した。

2) 収率は32% (シラカンバを原料とするKP廃液を原料としたもの) から84% (脱墨廃物を原料としたもの) に分布し、平均59%であった。これは通常の水蒸気賦活法(8%)、塩化亜鉛法(33%)とくらべて、かなり高収率であった。

3) 活性化の条件として、一般的に 190°C—2 時間が最良で、ついで 170°C—2 時間、ついで 190°C—1 時間、最低は 170°C—1 時間であった。市販活性炭は、これらより優れていた。

4) メチレンブルーの吸着は、170°C の条件で製造した活性炭のほうが、190°C よりも良い結果を与えた。

5) パルプ廃液の吸着にたいしては、一般的に 190°C の条件で製造した活性炭のほうが、170°C よりも良い結果であった。

6) KP 廃液の色の吸着にたいしては、190°C—2 時間の条件で製造した活性炭が最良であった。

7) SP 廃液の色の吸着にたいしては、170°C—2 時間の条件で製造した活性炭が最良であった。

8) 市販活性炭は、メチレンブルー、リグニン、KP 廃液の色にたいして有用であったが、SP 廃液の色の吸着にたいしては弱かった。

9) アカエゾマツを原料とする SP 廃液、H 社からのパルプ粕、ならびにミズナラ木粉からの活性炭は、市販活性炭とほぼ同等のすぐれた能力を示した。

10) これら活性炭の炭素含有率は、28% (J 社からのパルプ粕) から 64% (ミズナラ木粉) に分布し、無灰物にたいする炭素含有率は 65% (シラカンバを原料とする KP 廃液) から 86% (脱墨廃物) までに分布していた。