Studies on Evaluation and Utilization of Activated Carbon (Part 4)

Batch and Continuous Reactivation of Activated Carbon with a 100 mm ϕ Fluidized Bed Reactor

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High temperature reactivation of activated carbons used for treatments of phenolic wastewater effluents was investigated by using a 100 mm ϕ fluidized bed reactor equipped with a screw feeder. Reactivation could be carried out in both batch and continuous systems. Batch reactivation was carried out by using a reactor installed with one perforate plate (that is, one hearth), and continuous reactivation was performed by using a reactor installed with one additional perforate plate (that is, a co-current two-hearth fluidized bed reactor).

Specific surface area and methylene blue adsorption capacity of the spent carbon could be regenerated to 90% of the levels of the virgin carbon by simple heat treatment in nitrogen.

Improvement of such properties as specific surface area and methylene blue adsorption capacity through reactivation with steam was slightly less in the continuous system than in the batch system due to the apparent decrease in the reactivation yield caused by the burning of the carbon with the coexisting air and the loss of fine particles produced in the screw feeder. However, sufficiently effective regeneration could be achieved in both systems.

The decrease in strength of carbon by reactivation and reactivation losses were larger in the presence of air.

The carbon obtained with a yield of about 95~96% in either batch system or in continuous system could be used for the treatment of phenolic wastewater effluents.

1. Introduction

In one of the authors' previous reports¹⁾, batch reactivation of activated carbon used for treatment of phenolic wastewater effluents was investigated with a 50 mm ϕ fluidized bed reactor.

A fluidized bed reactor, one of the typical reactors for reactivation of spent activated carbon^{2),3)}, is simple in structure and maintenance, and is considered to be suitable for batch reactivation. There are only few reports on continuous reactivation of spent carbons utilizing a fluidized bed reactor due, perhaps, to the fact that solid particles of different sizes in a fluidized bed reactor generally are completely mixed, and to control their residence times accurately to achieve the same degree of reactivation of the particles would be difficult^{2),3)}.

Recently, Kawabata et al. showed that it was possible to carbonize coal continuously with a co-current two-hearth fluidized bed reactor⁴⁾.

In this report, the results of continuous reactivation

of spent activated carbons used for treatment of phenolic wastewaters in an industrial plant were compared with the results of batch reactivation.

The effects of different processes and raw materials for activated carbons on reactivation were also discussed.

2. Experimental

2.1 Activated Carbon

Activated carbon was a commercially available product manufactured from coal. About 1,000 m³ of phenolic wastewater (phenol and its derivatives: 100~150 ppm as phenol) from a fluid catalytic cracking plant (FCC) was treated with 1 t of the activated carbon in a commercial plant. The spent carbon was dried in the atmosphere of nitrogen prior to reactivation in a continuous fluidized bed reactor, similar to that shown in **Fig. 1** but without a second perforate plate. The carbon thus dried in particle size ranging from 8 to 32 mesh (Tyler) was used as the spent activated carbon for the reactivation runs.

The carbon was fed into the reactor, fluidized with nitrogen (velocity: 30 cm/sec at 400°C), and then heated with an electric furnace. When the

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Table 1 Properties of Virgin and Spent Activated Carbon

Properties	Virgin	Spent
Specific Surface Area (m ² /g)	1,105	884
Methylene Blue Adsorption Capacity (mg/g)	303	206
Bulk Density (g/ml)	0.393	0.497
Strength (%)	89	94
Ash (%)	1.7	—
Particle Size (mesh, Tyler)	8~32	8~32

temperature just above the perforate plate reached $400 \pm 20^\circ\text{C}$, a continuous feeding of the carbon was started. The temperature was controlled by changing the carbon feed rate. The average residence time, 15 min, was calculated from the carbon hold-up time in the reactor and its recovering rate.

Properties of both virgin and spent activated carbons are shown in Table 1.

2.2 Apparatus

A 100 mm ϕ continuous fluidized bed reactor^{4),5)} shown in Fig. 1, equipped with two perforate plates, was used. The second perforate plate (pore diameter: 5 mm, total pore area: 20% of the total area of the plate, i.e., the sectional area of the reactor) was placed at 150 mm above the first perforate plate (pore diameter: 2 mm, total pore area: 2% of the total area of the plate). An overflow line was located at 300 mm above the first perforate plate.

In the case of batch reactivation, the second perforate plate was removed from the reactor.

2.3 Procedure

2.3.1 Batch Reactivation

1.0 kg of spent activated carbon was put into the

RV-1 : Reactor, RV-2 : Receiver for product
RV-3 : Cyclon, SF : Screw feeder
HP-1 : Hopper for used carbon
HT-1~3 : Heater, M-1~2 : Manometer
PT-1~2 : Perforate plate

Fig. 1 Continuous Fluidized Bed Reactor

reactor. While the reactor was heated with the electric heaters, nitrogen was introduced as a fluidizing gas (velocity: 40 cm/sec at 800°C). The minimum fluidizing velocity was 11 cm/sec at 25°C . Temperature was measured just above the perforate plate and controlled by adjusting the electric current of the heaters. When it reached the desired temperature, the carbon was reactivated for a definite period of time with nitrogen (thermal regeneration) or with steam (velocity: 40 cm/sec at 800°C), and then left in the reactor for cooling in a stream of nitrogen. When the temperature attained below 200°C , the carbon was withdrawn from the reactor and weighed, and its properties were determined.

2.3.2 Continuous Reactivation

1.0 kg spent activated carbon was fed into the reactor with the screw feeder. Nitrogen gas was introduced at a linear velocity of 40 cm/sec (at 800°C), and then the reactor was heated with the electric heaters. When the temperature just above the second perforate plate reached the desired point, the spent carbon was fed into the reactor with the screw feeder, and the fluidizing gas was changed from nitrogen to steam or a mixture of steam and air. After 1 hr, the reactivated carbon was collected in the receiver through the overflow line for additional 1 hr. The fluidizing gas was changed back to nitrogen, feeding of the spent carbon was stopped and reactor was left alone for cooling. When the temperature reached below 200°C , the carbon remaining in the reactor and that recovered in the receiver were taken out for testing.

The temperature was controlled by adjustment of the electric current of the heaters, and the residence time of carbon was controlled by the feeding rate of the screw feeder. Average residence time was calculated from the combined weight of the carbon remaining in the reactor and the reactivated carbon recovered in one hour.

2.4 Determination of Properties of Activated Carbon

Determinations of specific surface area, methylene blue adsorption capacity, bulk density, strength, ash content of activated carbon and adsorption properties for phenols in the wastewater of the carbon were carried out by the methods described in the previous report¹⁾.

3. Results and Discussion

3.1 Thermal Analysis of Spent Activated Carbon

The weight decrease in the spent activated carbon

Fig. 2 T. G. Curve of Spent Activated Carbon

by heat treatment was measured by the method used in the previous report¹⁾. The results obtained are shown in **Fig. 2**. The carbon tested in this experiment was not dried in the continuous fluidized bed reactor.

The weight decrease occurring above 100°C proceeded continuously and gradually due to the vaporization and decomposition of the substances on the carbon. The tendency is similar to those about fundamental thermal analyses of the activated carbon adsorbing pure phenol⁶⁾ and substances in sewage⁷⁾, but considerably different from that obtained in the previous report¹⁾.

Methylene blue adsorption capacity (MB) of thus treated carbon was 274 mg/g, about 90% of that of the virgin carbon. This result indicated that most of the substances on the spent carbon were removed by simple heat treatment in nitrogen, differing from the results obtained in the previous report and in the fundamental experiments⁶⁾. This may be attributed to the fact that different carbons show different characteristics as catalysts for vaporization and decomposition of the adsorbed substances, and further investigations are necessary to clarify this point.

3.2 Batch Reactivation with Nitrogen (Thermal Regeneration)

Relationships between reactivation temperatures and yields as well as properties of reactivated carbons are shown in **Fig. 3**.

In the atmosphere of nitrogen, no reaction of the skeleton carbon can practically occur, that is, reaction of the spent carbon with nitrogen results only from the vaporization (sometimes accompanied by decomposition) of the adsorbates. This is obvious from **Fig. 3**. Reactivation yield does not decrease in the temperature range of 800~900°C. The vaporization concerned (with or without decomposition) is almost complete below 800°C. It is interesting, however, to note that specific surface area (S) and MB of the carbon thus treated are recovered as high as 90% of those of the virgin carbon.

Average yield in the thermal regeneration is 95.0%

Fig. 3 Effect of Reactivation Temperature on Reactivation Yield and Properties of Reactivated Carbon —by N₂, Batch—

indicating that the original activated carbon fraction in the spent carbon is 95.0%. From this result, reactivation yields in the following sections will be calculated as follows:

$$\text{Yield (\%)} = \frac{\text{Amount of reactivated carbon obtained (g)}}{\text{Amount of spent carbon fed (g)} \times 0.950} \times 100$$

This calculation process is the same as that used in the previous report¹⁾.

3.3 Batch Reactivation by Steam

Experimental conditions and results of reactivation are shown in **Table 2**.

Reactivation yield decreases with increasing temperature and with increasing reactivation period as shown in **Fig. 4**; however, the tendency is small, compared to that observed in a 50 mmφ fluidized bed reactor¹⁾. For example, reactivation at 900°C for 10 min is necessary in order to decrease the reactivation yield to 90%, while reactivation at 820°C for 10 min has given the same yield in a previous experiment. This indicates that, except for the difference in the types of reactors, reactivity of carbon with steam varies depending on the original carbonaceous material.

Fig. 5 shows the effect of reactivation temperature on the properties of reactivated carbon.

S and MB are regenerated more remarkably by raising the reactivation temperature, but the degree

Table 2 Conditions and Results of Reactivation—Batch—

Conditions			Yield (%)	Properties				
Ex. No.	Temp. (°C)	Period (min)		S ¹⁾	MB ²⁾	BD ³⁾	St ⁴⁾	Ash (%)
H-26	828	10	99.2	1,078	289	0.438	86.7	2.1
H-37	820	20	95.4	1,022	286	0.435	90.1	2.0
H-23	874	10	90.7	1,132	301	0.433	87.5	2.3
H-24	848	20	90.3	1,096	312	0.403	86.2	2.1
H-25	865	30	84.2	1,543	332	0.400	82.5	2.5
H-36	896	10	89.5	1,125	296	0.425	90.8	2.8
H-35	911	20	87.5	1,198	315	0.403	86.2	2.2
H-00 ⁵⁾	810	10	98.5	1,168	340	—	—	—

1) S : Specific Surface Area (m²/g) 2) MB : Methylene Blue Adsorption Capacity (mg/g)
3) BD : Bulk Density (g/ml) 4) St : Strength (%) 5) Further activation of virgin carbon

Fig. 4 Effect of Reactivation Temperature on Reactivation Yield —by Steam, Batch—

of increase is small. Reactivation at about 850°C is necessary for complete regeneration of S and reactivation at 840°C for 20 min or 870°C for 10 min for complete regeneration of MB.

Reactivation with nitrogen has recovered S and MB to their average values of 1,045 m²/g and 270 mg/g, respectively. The effects of regeneration with steam then appear as increases in their values. Reactivation conditions for complete regeneration of S and MB give the yield levels of 90~95%. Increases in S and MB that correspond to a 5~10% decrease in yield are only about 60 m²/g and 30 mg/g, respectively. Gasification of the virgin activated carbon with steam to about 98.5% yield increases S and MB to 65 m²/g and 37 mg/g respectively, as shown in **Table 2**. These results indicate that increases in S and MB for decreases in reactivation yields are considerably smaller compared with those in the further activation of the base carbon, probably because some species of substances found on the spent carbon may exert a retarding effect.

Fig. 5 Effect of Reactivation Temperature on Properties of Reactivated Carbon —by Steam, Batch—

Bulk density can be restored by reactivation at 860°C for 20 min. The strength of the carbon reactivated at 850°C, where complete regeneration is attained for S and MB, is decreased by about 3%. The ash component in the carbon that has increased by the adsorption process cannot be removed by reactivation.

From the above statements, it is clear that some difference in the yields of products would result even when the same reactivation conditions used in the previous experiments¹⁾ were used here, and that a lesser degree of improvement of S and MB as well as the lowering of strengths of the carbon was obtained. These results may partly due to

Table 3 Conditions and Results of Reactivation—Continuous—

Ex. No.	RG-203	RG-204	RG-205	RG-206	RG-207	RG-208	RG-209
Temp. (°C)	797	815	815	785	801	789	814
Fluidizing Gas							
Steam (cm/sec)	36.2	35.3	23.6	38.7	40.4	40.4	29.6
Steam+Air (cm/sec)	—	36.9	33.7	—	41.6	—	34.0
Residence Time (hr)	1.05	0.88	0.57	0.65	0.53	0.39	0.50
Yield (%)	80.0	81.6	87.6	97.9	98.2	96.5	83.7
Burning by O ₂ (%)	—	4.6	12.0	—	1.4	—	7.4
S ¹⁾	1,125	1,169	1,075	1,046	1,060	1,075	1,233
MB ²⁾	312	312	316	297	301	288	324
BD ³⁾	0.423	0.403	0.410	0.430	0.420	0.420	0.400
St ⁴⁾	79.1	74.8	75.1	87.9	87.7	86.8	79.2
Ash	1.9	2.0	2.1	2.1	2.2	1.9	2.2
Dust (% to CHG)	5.0	2.9	—	2.0	0.3	1.6	2.1

1) S : Specific Surface Area (m²/g) 2) MB : Methylene Blue Adsorption Capacity (mg/g)

3) BD : Bulk Density (g/ml) 4) St : Strength (%)

the difference in the reactivation systems used, but mainly due to the difference in the species of the activated carbons, i.e., the reactivity of activated carbon with steam varies depending on the characteristics of its raw carbonaceous material, and that reactivation conditions should be investigated individually depending on the species of the activated carbon used.

The ash content of activated carbon appears to increase with repeated usage and reactivation, which is in line with the results obtained in the previous experiments¹⁾.

3.4 Continuous Reactivation by Steam

Experimental conditions for continuous reactivation and the results obtained are shown in **Table 3**.

Combustion gas is usually utilized as a heat medium in industrial reactivation of spent activated carbon^{8),9)}, and consequently a small amount of oxygen is introduced into the reactor. Several experiments have been made under coexistence of air and steam in order to clarify the effect of oxygen on reactivation. Continuous reactivation was carried out at constant temperature ($800 \pm 15^\circ\text{C}$), and the effect of residence time on reactivation was investigated.

Fig. 6 shows the effect of residence time on yield and properties of the reactivated carbon. In **Fig. 6**, yields with arrows were obtained from reactivation under coexistence of air, and the lengths of arrows indicate the amounts of decrease in yield due to burning off of the carbon with oxygen, assuming that all oxygen in the air reacted with carbon.

Fig. 6 indicates a considerable scattering of results, especially those of yields and strengths, evidently, due to the effect of coexistence of air. Decreases in yields and strengths of reactivated carbons obtained under coexistence of air are larger in comparison with those obtained with pure steam. Their

Fig. 6 Effect of Residence Time (θr) on Reactivation Yield and Properties of Reactivated Carbon—Continuous—

bulk density also decreases under coexistence of air.

From **Fig. 6**, the residence time required for regenerating S and MB to the levels as high as those of the virgin carbon is about 0.5~0.6 hr and the yield is about 92~90%.

The small change in bulk density makes it clear that the reaction of the spent carbon with steam does not increase the porosity of the carbon and that steam (or steam and air) gasifies only the exterior surface of the carbon. This tendency was also observed in batch reactivation, where the effect was larger than that in the previous report.¹⁾ This

result might have been caused by the characteristics of the carbon in the reactivity with steam or a mixture of steam and air.

The particle size of activated carbon decreases by reactivation as shown in **Fig. 7**. A screw feeder was used to charge the spent carbon into the reactor. **Fig. 7(C)** shows the particle size distribution of reactivated carbon RG-207 after it was subjected again to transport by the screw feeder but not subjected to reactivation. The size of particles decreased visibly in the process of transport. Abrasion of carbon particles by reactivation reaction itself was small as shown in the previous report.¹⁾ Therefore, the transferring of activated carbon with the screw feeder may be the main cause of decrease in the particle size of the carbon by abrasion. Thus, a further development or selection of an appropriate feeding equipment for activated carbons is necessary to prevent abrasion of carbon particles.

The ash components in spent carbon cannot be removed completely by reactivation as shown in **Table 3**. The result is similar to that obtained by batch reactivation.

The loss of fine particles of carbon in the dust collector (cyclon) is one of the characteristics of fluidized bed reactors. In the present study, it amounted to 5% of the feed for the case of the longest reactivation period. Pulverization of carbon by the screw feeder also is one of the causes of the loss that must be further investigated.

3.5 Relationships between Yields and Properties of Reactivated Carbons

Fig. 7 Particle Size Distribution

Fig. 8 shows the relationships between yields and properties of reactivated carbons obtained by both batch and continuous reactivation with steam.

A slight difference is found in the results. The degree of improvement of S and MB by batch reactivation is slightly greater than that by continuous reactivation. The main reason for this may be the apparent decrease in the reactivation yield due to the burning off of the carbon with the coexisting air and the loss of the pulverized carbon particles. Continuous operation with the co-current two-hearth fluidized bed gives approximately the same regeneration effects for S and MB as by batch reactivation. The carbon reactivated with a yield of about 90% has fully restored the value of S and MB in both systems of reactivation.

The observed smaller change in bulk density and the larger decrease in strengths by continuous reactivation may be due to the effect of coexisting air.

In these experiments, bulk density also is observed to be in good correlation with such properties as S and MB^{1),10)}.

3.6 Adsorption Properties of Reactivated Carbons for Phenols

Adsorption properties of reactivated carbons, H-37, RG-204 and RG-208, for phenols in the FCC wastewater effluents were examined.

Figs. 9 and **10** show equilibrium adsorption capacities and adsorption rates of the reactivated carbons for phenols, respectively.

Fig. 8 Relationships between Reactivation Yields and Properties of Reactivated Carbons

Fig. 9 Adsorption Isotherm for Phenols

Initial concentration of phenols : 120 ppm as phenol
Activated carbon : 1.0 g, Wastewater : 500 ml

Fig. 10 Adsorption Rate for Phenols

Equilibrium adsorption capacities of the reactivated carbons are a little smaller than the virgin carbons, but their adsorption rates are approximately equal or somewhat larger. It should be noted that the difference of adsorption properties between RG-204 and RG-208 is small in spite of the larger discrepancy in their reactivation yields.

Breakthrough curves of the reactivated carbons for phenols are shown in **Fig. 11**.

From **Fig. 11**, capacities of RG-208 and H-37 for treatment of phenols were almost equal to that of the virgin carbon, and the capacity of RG-204 was a little larger.

There was no difference in the adsorption capacity for phenol treatment between H-37 reactivated in the batch system and RG-208 reactivated in the continuous system; moreover the yields (95~96%) of the two carbons were nearly equal.

From the above discussion, carbon reactivated to the level of 95~96% yield would be satisfactory

Activated carbon : 10g, Phenols : 120 ppm as phenol,
Flow rate : 10 ml/min/g-carbon

Fig. 11 Breakthrough Curves for Phenols

for phenolic wastewater treatments examined here whether it is reactivated in the batch system or in the continuous system. Such properties as S and MB of the carbons, however, are not fully recovered to the levels of the virgin activated carbon.

4 Conclusions

Continuous reactivation of spent activated carbon used for phenolic wastewater treatments (1 t of carbon per 1,000 m³ of wastewater containing 100~150 ppm phenols) was investigated with a 100 mmφ co-current two-hearth fluidized bed reactor for comparison with batch reactivation in the same reactor.

Heating in nitrogen regenerated specific surface area and methylene blue adsorption capacity to about 90% of those of the virgin activated carbon.

When steam was used as a reactivating gas, the specific surface area and methylene blue adsorption capacity of the carbon could be regenerated slowly but completely in both the continuous and batch experiments. But the levels of restoration attained by the continuous reactivation were a little less. This might be due to the apparent decrease in the reactivation yield based on the burning of the carbon by the coexisting air and the loss of the fine carbon particles produced in the screw feeder. It was supposed that almost the same effects of regeneration were obtained by continuous reactivation by using the co-current two-hearth fluidized bed reactor.

From the result of the slight restoration of bulk density, of specific surface area and of methylene blue adsorption capacity, it was considered that the porosity of the spent carbon was not recovered substantially even by reaction with steam. Certain characteristics of the carbon involved in reactivity with steam might have brought about these results, and further studies would be necessary to clarify the problem. Coexistence of air was one of the

factors that deleteriously affected yields and strengths of reactivated carbons.

The carbon obtained with a yield of about 95~96% was found applicable to treatments of phenolic wastewater effluents whether it was reactivated in the continuous system or in the batch system; however, such properties as specific surface area and methylene blue adsorption capacity were not regenerated to the levels of the virgin activated carbon.

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要 旨

活性炭の評価ならびに利用に関する研究 (第4報)

100 mmφ 流動床炉による活性炭の回分式および連続式再生の比較

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100 mmφ の流動床炉を用いて、フェノール類含有廃水処理に使用された活性炭の連続式再生を回分式再生と比較しながら検討した。回分式再生は通常の流動床型式において行なったが、連続式再生は河端ら⁴⁾が提案した併流2段流動床炉 (Fig. 1) によって実施した。活性炭は石炭系の市販品であった。

まず、使用済活性炭の熱分析を行なったところ、前報¹⁾における結果とは異なりほぼ連続的な重量減少が起こることがわかった (Fig. 2)。この時、メチレンブルー吸着力 (MB) はおよそ 90% の回復を示したことから、活性炭上の吸着物質のほとんどは単なる熱処理によって除去されると推定された。前報における結果との比較から、その原料および製法の違いに基づく活性炭の種類によって吸着物質の熱的な蒸発および分解特性が異なることが明らかになった。

水蒸気による回分式再生において、新活性炭の比表面積 (S) および MB とほぼ等しい値を有する再生炭が、90~95% の収率で得られた。しかし、S および MB の増加割合は、新活性炭をさらに水蒸気賦活した場合のそれらの増加割合と比較してかなり小さかった。これは吸着過程において増加した灰分の影響であると思われたが、さらに詳細な研究が必要である。またこの時、再生炭の強度は新活性炭のそれと比較して約 3% 小さく、この点においても活性炭の種類の差が明らかであった。

連続式再生においては、燃焼排ガスを熱源として用いることが多い工業的再生を想定して、水蒸気に若干の空気を混合することによって共存酸素の再生に与える影響をも調べた。酸素の共存は再生収率を低下させ、また強度およびかさ密度を低下させたが、S および MB に対してはあまり影響がなかった (Fig.

6)。再生炭の収率をおよそ 90% にすることによって、S および MB はほぼ完全に再生されると推定された。

連続式再生においては収率低下に対する S および MB の増加割合が、回分式再生の場合と比較して幾分か小さかった (Fig. 8)。しかしこれは、共存空気による炭素の燃焼およびスクリーフィーダーによって粉化された活性炭の飛び出しに基づく再生炭収率の見かけの減少に主因があると考えられ、併流2段流動床炉を利用した連続式再生は、本質的には回分式再生とほぼ同様の効果を与えると推定された。

連続式再生においても回分式再生においても、S および MB の増加傾向が小さかったことと関連するが、収率低下に対するかさ密度の減少割合は小さかった。このことから、使用済活性炭の再生においては、本来炭素の多孔質化を与えるべき炭素と水蒸気との反応が活性炭の表面のみで進行して炭素の多孔質化を促進しないという傾向があることが知られた。これに対しても、原料および製法の違いに基づく活性炭の種類の影響があると思われたが、なお詳細な検討が必要である。

次いで、連続式再生および回分式再生によって得られた再生炭の廃水中フェノール類に対する吸着特性を、新活性炭のそれと比較しながら調べた。平衡吸着能力においては、何れも新活性炭よりもやや小さかったが、吸着速度においては新活性炭と同程度もしくはそれ以上であった。また、連続通水試験によって破過曲線を測定したところ、収率 95~96% で得られた再生炭は、それが連続式で再生されたか回分式で再生されたかによらず、またその S および MB は新活性炭の値にまで回復していないにもかかわらず、ほぼ新活性炭と同程度のフェノール類含有廃水処理能力を有することが知られた (Fig. 11)。

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Keyword

Activated carbon, Adsorption capacity, Continuous reactivation, Phenolic wastewater