

Sewage Sludge Carbonization for Adsorbents Use

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Abstract: In this study, optimum conditions of sewage sludge carbonization for adsorbent production and qualities of carbonized sewage sludge as an adsorbents were discussed. In order to study the characteristics on the digestion and surplus sewage sludge, proximate analysis (such as moisture, ash, combustibles, volatile matter and fixed carbon content), elemental analysis, and caloric value analysis of wet sewage sludge were performed and dried. For the carbonized sewage sludge obtained through the change of carbonization conditions, BET, pore size, and iodine adsorption performance tests were carried out to derive the optimum conditions of carbonization. As a result of the overall review on the fixed carbon content of dried sewage sludge and specific surface area (BET), pore size, and iodine adsorption performance of carbonized sewage sludge obtained, it is difficult to produce the adsorbent with good adsorption performance, but low-quality adsorbent is regarded to be possible. In spite of disadvantages, in the viewpoint of carbonizing the sewage sludge to produce an adsorbent, the optimum carbonization temperature was derived to be 350~450°C and the optimum carbonization time to be 30~60 minutes.

Key words: sewage sludge, carbonization, adsorbent, specific surface area(BET), iodine adsorption

I. Introduction

The world is currently facing a large economic and environmental crisis due to the high price of oil and global warming due to the depletion of fossil fuel resources. In these global economic and environmental crises, good energy is defined as the sustainable driving force for national development. Thus, a new and sustainable, competitive, and renewable energy policy is strongly promoted.

Studies related to the treatment and recycling of the sewage sludge generated in the sewage treatment process have progressed steadily, but it is regarded that a distinct technology has not yet been revealed. In particular, recycling of the sewage sludge through drying and carbonization has the advantages of enabling comparatively wide utilization and that treatment is easy for each purpose. However, from the perspective that equal quality assurance and utilization method of the product are not developed sufficiently, it

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is evaluated that the reliability and the stability of the technology is still insufficient and currently still in the development stage.

There were a lot of studies about producing adsorbents from sewage sludge, but there is still not sufficient data in our opinion.¹⁻⁴⁾ Moreover, the results have differentiated content that would be difficult for general application. For example, Park *et al.*¹⁾ reported that the maximum iodine adsorption was 123.5mg/g with the carbonized sewage sludge obtained for 30 minutes at 700°C, and 50% decolorization in the wastewater treatment was achieved when carbonizing for 15 minutes at 700°C as a optimum carbonization condition in the basic study result for utilizing carbonized sewage sludge as the adsorbent. Additionally, Jung *et al.*⁴⁾ reported differently that the optimum sewage sludge carbonization condition with a dried sewage sludge of 0.25~0.5mm diameter and moisture content of 10% is a carbonization temperature at 600°C, resulted in a maximum iodine absorption of 149.1mg/g and yield of 30.9%. And also, for the specific surface area, the carbonized sewage sludge increased greatly to 62.31m²/g compared to the 6.44m²/g of the dried sewage sludge, and the pore distribution of the carbonized sewage sludge was reported to be 20~100Å, and that the medium pore of 36.61Å mainly developed for the average pore diameter.

Therefore, in this study, re-examination was performed on the currently ambiguous state of optimum carbonization conditions for the sewage sludge adsorbent production to clearly define this matter. And also qualities of carbonized sewage sludge as an adsorbents were discussed.

II. Experimental method

To use the sewage sludge as the adsorbent, the tests were performed as follows in this study.

2.1. Used sewage sludge and subject facility

The samples used in the experiment are the dewatered sewage sludge generated by treating the domestic sewage in D City, Korea through the standard activated sludge method. After collecting the samples, they were dried to minimize the change in properties. For the drying condition, the samples were completely dried for 6 hours at 105 °C, and the dried samples were sealed and cold stored for analysis.

2.2. Analysis of the sewage sludge

Approximate analysis and elementary analysis and so on were carried out to review the basic characteristics of the sewage sludge. Also, after carbonization, BET specific surface area was measured, and iodine absorption test were performed to review the usability as the adsorbent. But activation of carbonized sewage sludge with steam or any chemicals did not carried out.

2.3. Carbonization equipment and process

The equipment used for carbonization in this study included carbonizing furnace, temperature measurement equipment, and nitrogen gas supplying equipment, as shown in Figure 1 below.

In the carbonization process, the samples were fully dried and fine grinded for the production of high quality adsorbent, because a great amount of moisture in the sewage sludge can reduce the hardness. more details, the dewatered sewage sludge was dried up to 10% of moisture content in the electric furnace at 105 °C, grinded and sieved with a diameter of 0.25~0.5mm for usage.

Before the carbonization process was performed, the inside of the carbonization reactor was replaced into inert atmosphere with the N₂ gas flow at 100mL/min and preheated to the constant carbonization temperature; when the carbonization temperature was reached, stabilization time was provided to maintain a constant inner temperature.

After the inside of the carbonization reactor was stabilized, the dried sewage sludge was inserted as the carbonizing sample was carbonized for 60 minutes at each carbonizing temperature. Here, the carbonizing temperature was changed from 250 to 650 °C to continue the test. Also, in the optimum carbonizing temperature derived, the carbonizing was changed at 20mins, 40mins, 60mins, 80mins, 100mins, and 120mins to review the effect according to carbonization time variation. To maintain the reducing atmosphere of the carbonization process, N₂ gas at 100mL/min was injected continuously.

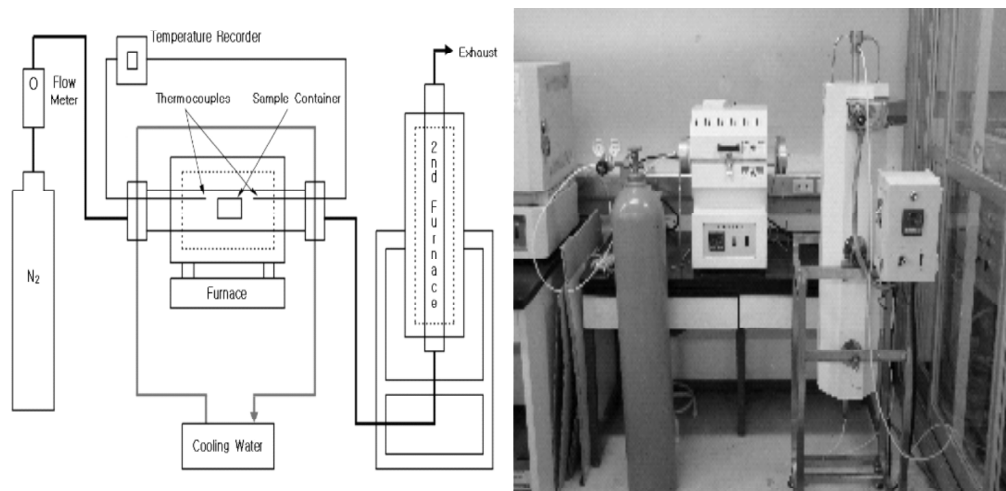


Figure 1: Schematic diagram and image of the carbonization equipment for the sewage sludge

III. Results and Discussion

3.1. Analysis of physicochemical properties of excess and digested sewage sludge

The results of analysis on physicochemical properties of excess and digested sewage sludge discharged by the sewage treatment plant in D City are shown as follows:

1) Proximate analysis

(1) *Moisture, combustibles and ash analysis:* The moisture, ash, combustibles content of wet sewage sludge were analyzed according to the ASTM^[5-7], and the results of analyzing are

shown in Table 1: in the case of the excess sludge, moisture content was approximately 77~80%, ash content about 3~6.5%, and combustibles 16~17%. In the case of the digested sludge, moisture content was approximately 79~82.5%, ash content about 6~9%, and the combustibles 11~13%.

Table 1
Moisture, ash, combustibles content of wet sewage sludge

Month	Excess sludge				Digested sludge			
	Moisture	Ash	Combustibles	Sum	Moisture	Ash	Combustibles	Sum
Feb.	80.05	3.03	16.92	100.00	82.39	5.95	11.66	100.00
Mar.	78.78	4.68	16.54	100.00	80.12	7.16	12.72	100.00
Apr.	78.39	4.68	16.93	100.00	82.37	5.68	11.95	100.00
May	77.64	6.47	15.89	100.00	81.12	7.44	11.44	100.00
Jun.	77.57	5.96	16.47	100.00	78.58	8.76	12.66	100.00
Jul.	68.66	17.21	14.30	100.00	66.47	20.63	13.71	100.00
Aug.	66.72	18.85	14.43	100.00	63.33	22.09	14.56	100.00
Oct.	78.63	5.82	15.54	100.00	70.34	13.23	16.38	100.00
Average	75.81	8.34	15.88	100.00	75.59	11.37	13.14	100.00

(2) *Analysis of the volatile matter and fixed carbon:* The analysis results of the volatile matter (VM) and fixed carbon (FC) of wet excess and digested sewage sludges are shown in Table 2. As the results of analyzing the volatile matter and the fixed carbon, the fixed carbon was approximately 3~5% for the excess sludge, and 2.5~3.5% for the digested sludge.

Considering this amounts of fixed carbon content, it specifies that carbonizing the wet excess and digested sewage sludges to produce the adsorbent is realistically difficult. Therefore, the carbonized sewage sludge obtained is considered to be possible for use as soil conditioner. Additionally, if it is dried properly without loss in the volatile matter, it is considered to be possible for use as a fuel due to the sufficient volatile matter in status of dried, can be seen below in section of calorific value analysis.

Table 2
Volatile matter and fixed carbon analysis results of the wet sewage sludge

Month	Excess sludge			Digested sludge		
	VM	FC	Sum	VM	FC	Sum
Feb.	12.17	4.75	16.92	8.79	3.10	11.89
Mar.	12.28	4.21	16.49	9.31	3.41	12.72
Apr.	12.87	3.78	16.65	8.57	8.05	16.62
May	12.66	2.97	15.63	8.65	2.64	11.29
Jun.	12.21	4.27	16.48	9.14	3.59	12.73
Jul.	12.44	1.86	14.30	12.41	1.31	13.72
Aug.	14.24	0.19	14.43	14.32	0.24	14.56
Oct.	15.42	0.12	15.54	16.28	0.10	16.38
Average	13.04	2.77	15.81	10.93	2.81	13.74

2) Elemental analysis

The elemental analysis results for combustibles of the dried excess and digested sewage sludge are shown in Figures 2 and 3. As the results of the elemental analysis, the elemental composition ratio of combustibles in the excess sludge was higher than that in the digested sludge except for S element.

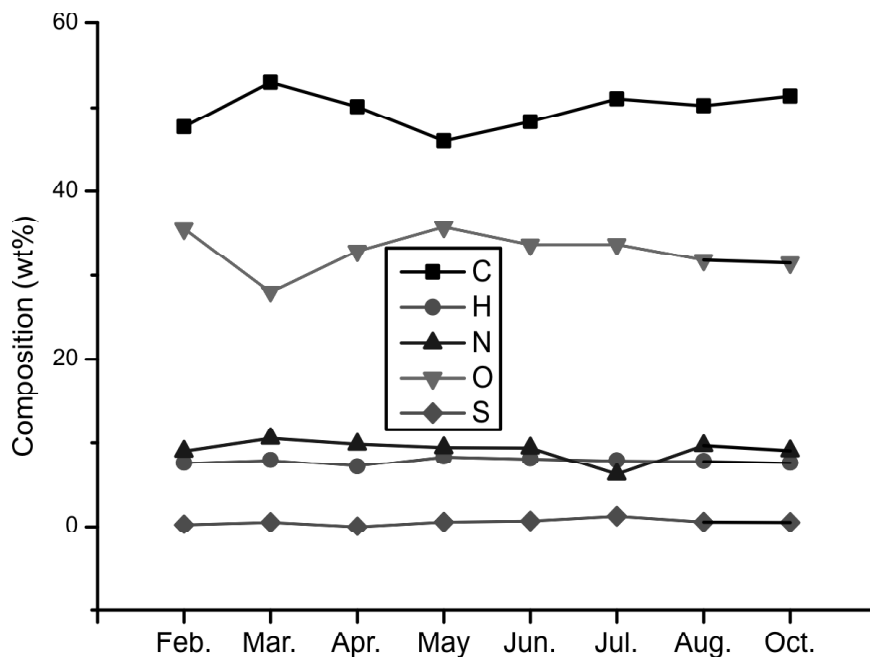


Figure 2: Elemental analysis results for combustibles of the dried excess sewage sludge

In the case of dried excess sludge, C was average 49.75%, O was average 32.69%, N was average 9.13%, H was average 7.7%, and S was average 0.54%. Otherwise, in the case of the dried digested sludge, C was average 49.75%, O was average 32.69%, N was average 9.13%, H was average 7.7%, and S was average 0.54%.

3) Calorific value analysis

The calorific values of the dried excess and digested sewage sludges were measured by using the bombe adiabatic calorimeter and calculated by using Dulong Eq., Scheurer-Kestner Eq. and Steuer Eq. is shown below in Figure 4 and 5. The calorific value of the dried excess sludge by using calorimeter was approximately 3,300~4,400kcal/kg for Feb. to Jun, but the calorific values for July and August were too low in the range of 2,200~2,400kcal/kg, due to be mixed it with soil etc in raining season. This results are in line with proximate analysis results.

The calorific value of the dried digested sludge by using calorimeter was approximately 2,800~3,800kcal/kg for Feb. to Jun, but the calorific values for July and August were also too low in the range of 1,800~2,000kcal/kg.

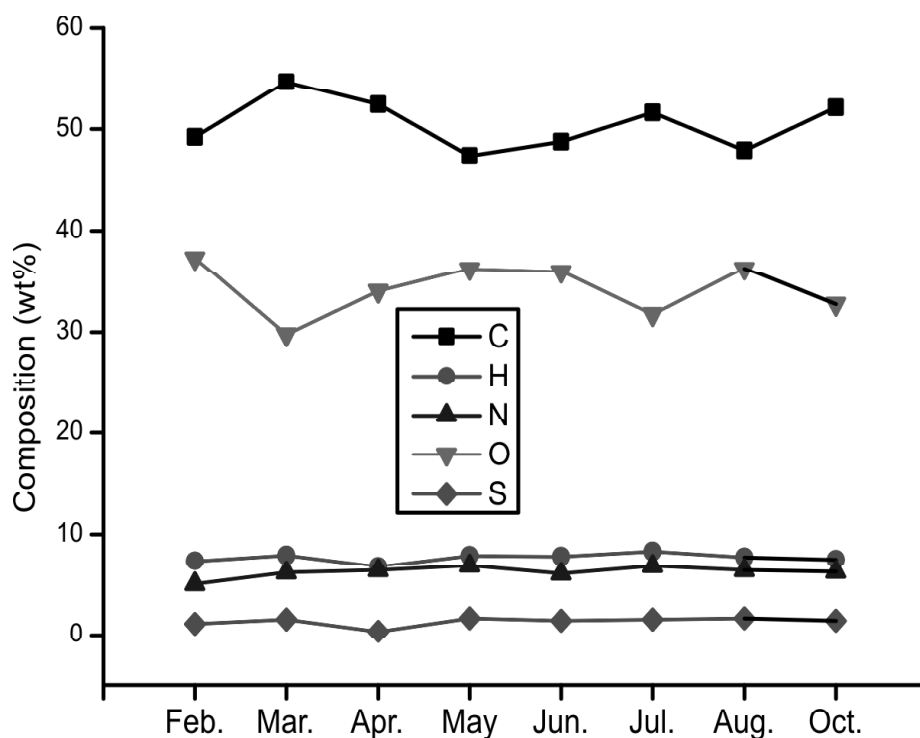


Figure 3: Elemental analysis results for combustibles of the dried digested sewage sludge

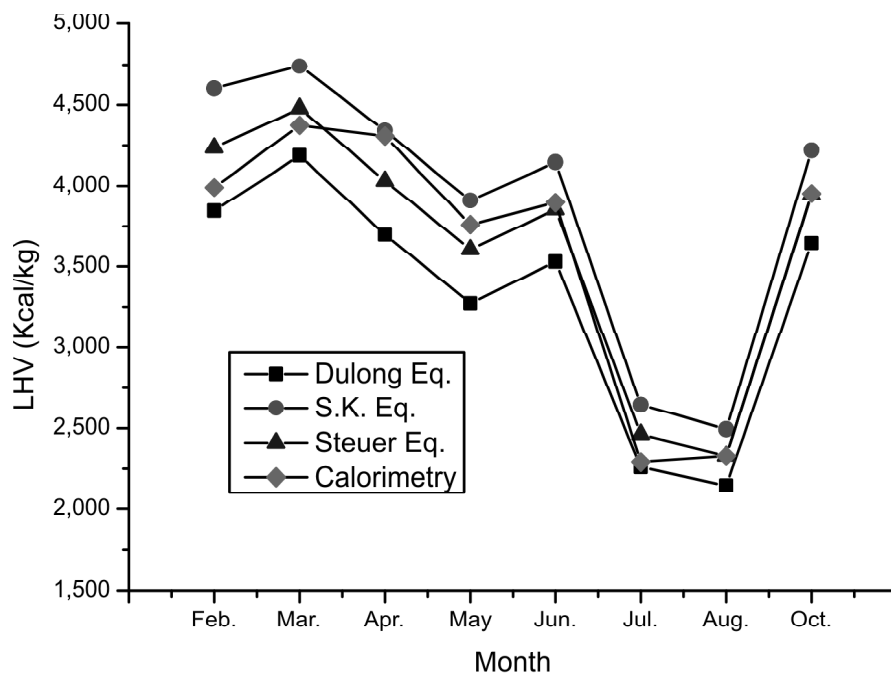


Figure 4: Calorific value analysis results for the dried excess sewage sludge

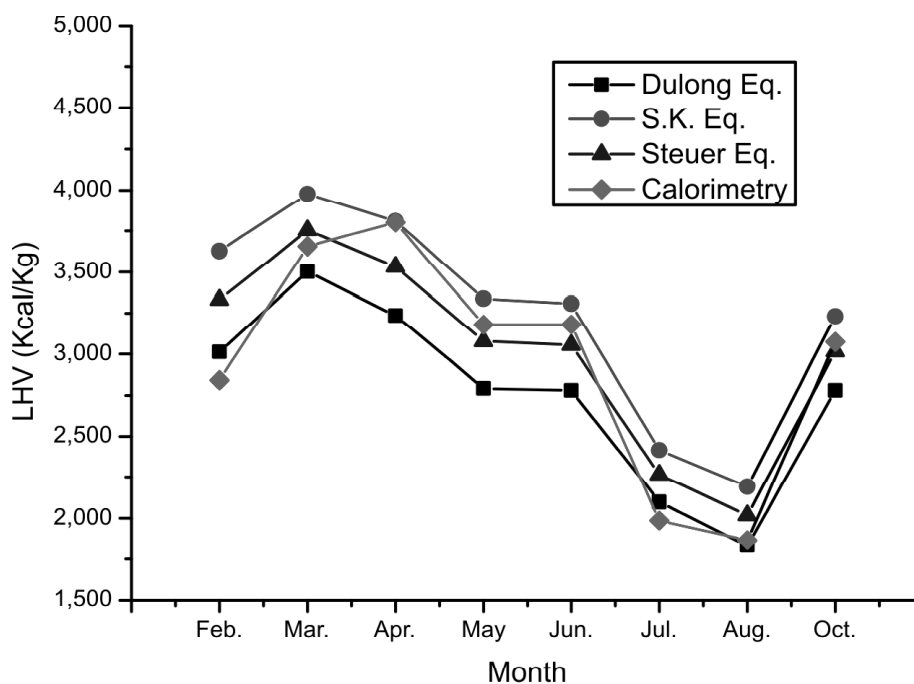


Figure 5: Calorific value analysis results for the dried digested sewage sludge

The calorific values measured by using bombe adiabatic calorimeter and calculated by using Scheurer-Kestner Eq. and Steuer Eq. are similar each other, but The calorific values calculated by using Dulong Eq. is lower of approximately 500~800 kcal than their values.

3.2. Analysis of physicochemical properties of carbonized excess and digested sewage sludge

1) BET specific surface area analysis

As the results of measuring the BET specific surface area of carbonized excess and digested sewage sludge, the specific surface area of the carbonized sewage sludge increased according to the increase in the carbonization temperature, and the maximum values were obtained with a carbonization time of 30~60 minutes at temperature of more than 450 °C. BET specific surface area of the carbonized sewage sludge was approximately 10~16m²/g compared to the approximately 3~5m²/g of the dried sewage sludge, but less than 62.31m²/g by Jung *et al.*⁴⁾ The BET specific surface area of commercial activated carbon is in the range of 800~1,200m²/g for water purification.

2) Pore size analysis

The average pore size of carbonized excess and digested sewage sludges were the largest of approximately 30~35Å at 250 °C, and decreased according to the increase in the

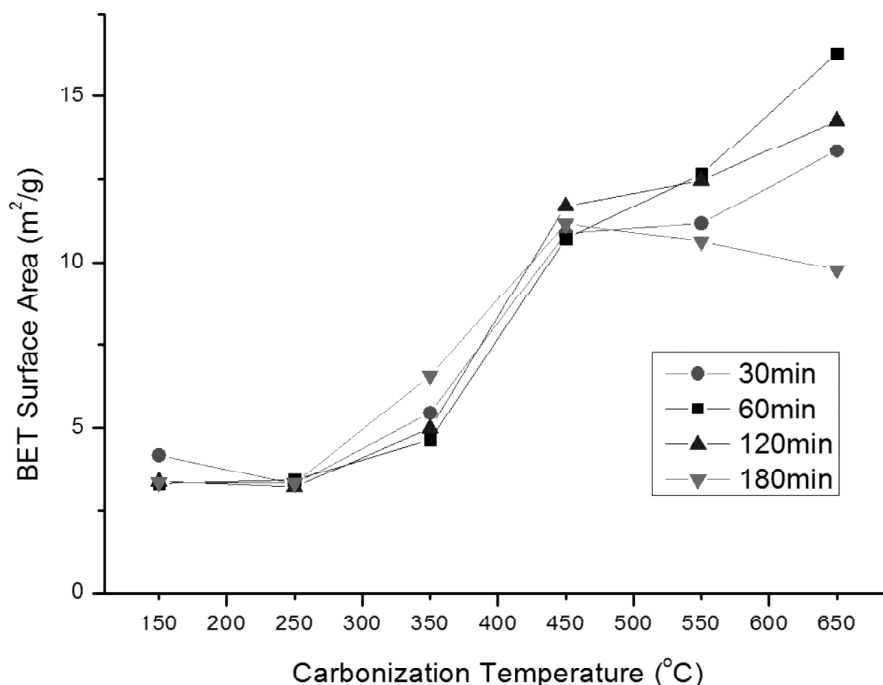


Figure 6: BET Specific surface area characteristics analysis result of the carbonized excess sludge according to the change in carbonization condition

carbonization temperature. There was almost no effect according to the carbonization time. Average pore size of the carbonized sewage sludge obtained from 450 °C was approximately 17 Å compared to the approximately 23~27Å of the dried sewage sludge, but less than approximately 36.61+! by Jung *et al.*⁴⁾ The average pore size of commercial activated carbon is in the range of 17~20Å for water purification.

3) Iodine adsorption performance tests

One of the measurement methods of evaluating the adsorbents are based on the iodine adsorption; therefore, the iodine adsorption performance of the carbonized excess and digested sewage sludge were reviewed according to the change in the carbonization condition. As the results of the iodine adsorption performance test of the carbonized excess sludge and the digested sludge, the optimum carbonization temperature were 350~450 °C and the optimum carbonization time was 30~60 minutes.

As shown in Figure 8 and 9, in the case of the carbonized excess sewage sludge, iodine adsorption performance of the carbonized sewage sludge obtained from 350 °C were approximately 180~240mg/g compared to the approximately 110~160mg/g of the dried sewage sludge at 150 °C, but it is higher than approximately 149mg/g by Jung *et al.*⁴⁾ The iodine adsorption performance of commercial activated carbon is approximately 1,000mg/g for water purification.

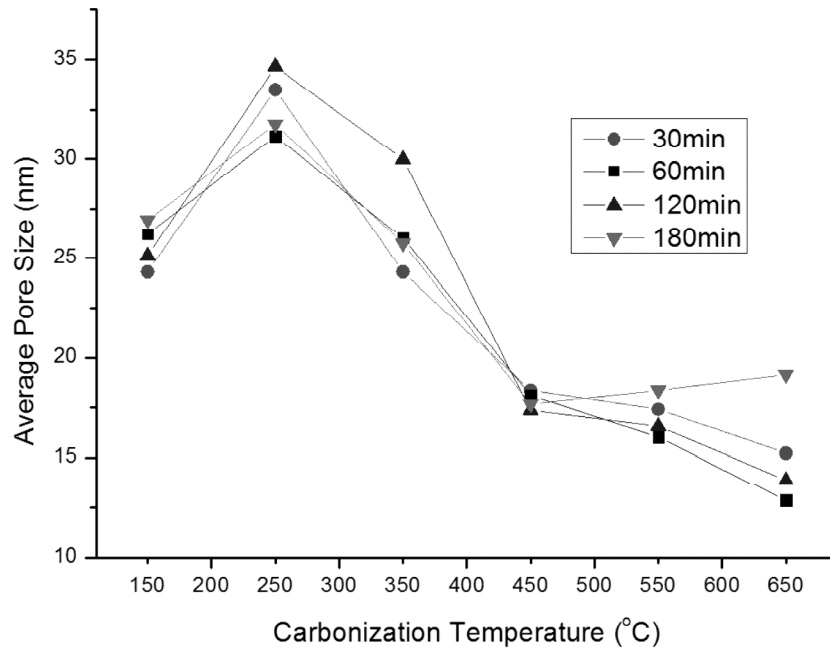


Figure 7: Average pore size analysis result of the excess sludge carbide according to the change in carbonization condition

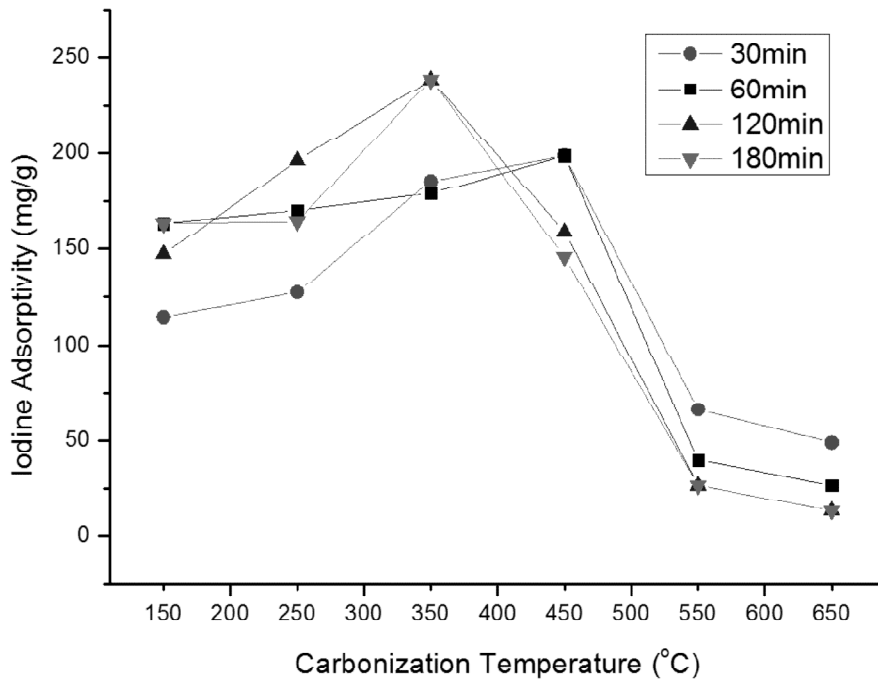


Figure 8: Iodine adsorption performance test results of the carbonized excess sewage sludge according to the change in carbonization condition

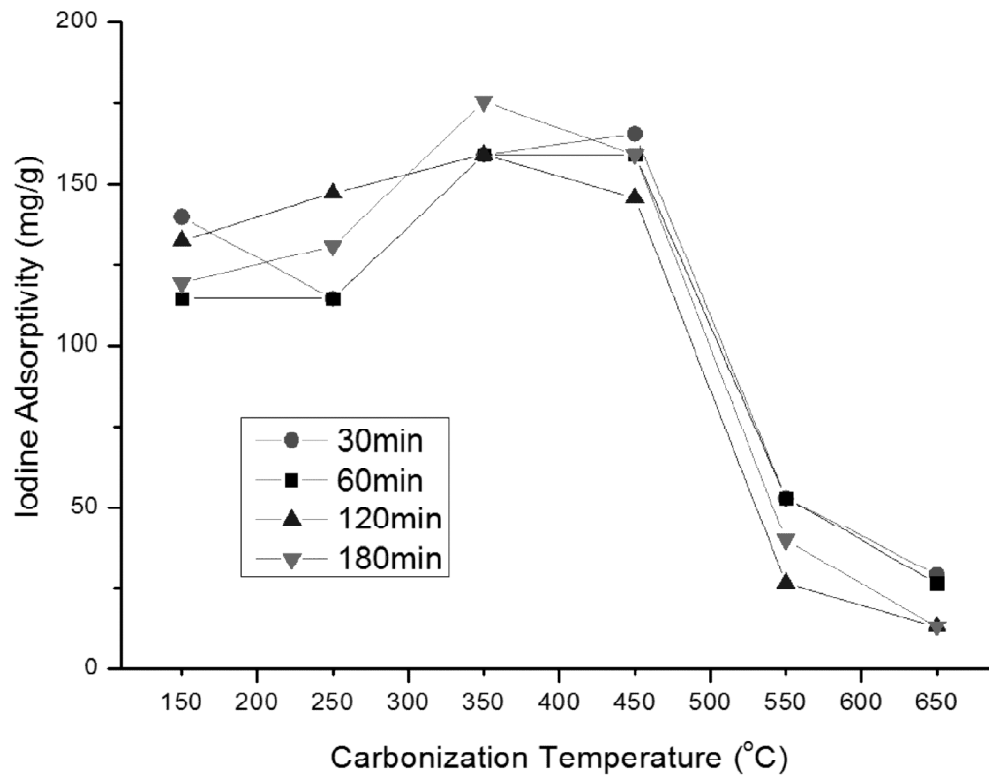


Figure 9: Iodine adsorption performance test results of the carbonized digested sewage sludge according to the change in carbonization condition

In the case of the carbonized digested sewage sludge, iodine adsorption performance of the carbonized sewage sludge obtained from 350 °C were approximately 160~175mg/g compared to the approximately 110~140mg/g of the dried sewage sludge at 150 °C, but it is almost similar to approximately 149mg/g by Jung *et al.*⁴⁾

4) Optimum carbonization condition for preparing absorbents

As the results of reviewing the BET specific surface area, pore size and the iodine adsorption performance test of the carbonized excess and digested sewage sludge obtained according to the change in the carbonization condition, the optimum carbonization temperature was 350~450 °C and the optimum carbonization time was derived in the range of 30~60 minutes, when using the sewage sludge as the raw material for adsorbent production.

VI. Conclusion

In the study of using sewage sludge to derive the optimum carbonization conditions for producing carbonized sewage sludge with absorption possibility, the results were obtained as shown below:

1. For the excess sludge, moisture was approximately 77~80%, ash content about 3~6.5%, and the combustible was 16~17%. For the digested sludge, moisture was approximately 79~82.5%, ash content about 6~9%, and combustible 11~13%.
2. The fixed carbon was approximately 3~5% for excess sludge and 2.5~3.5% for the digested sludge. Considering this amounts of fixed carbon content, it specifies that carbonizing the wet excess and digested sewage sludges to produce the adsorbent is realistically difficult. Therefore, the carbonized sewage sludge obtained is considered to be possible for use as soil conditioner. Additionally, if it is dried properly without loss in the volatile matter, it is considered to be possible for use as a fuel due to the sufficient volatile matter in status of dried.
3. The calorific values of the dried excess sewage sludge was approximately 3,800~4,400 kcal/kg, and the dried digested sewage sludge was about 2,800~3,800 kcal/kg. The calorific value of the excess sludge was approximately 500~1,000kcal higher per kg compared to the digested sludge.
4. BET specific surface area of carbonized sewage sludge increased as the carbonization temperature increased, and the maximum values were obtained at 30~60 minutes of carbonization time at temperature of more than 450 °C. BET specific surface area of the carbonized sewage sludge was approximately 10~16m²/g compared to the approximately 3~5m²/g of the dried sewage sludge.
5. The average pore size of carbonized sewage sludge was the largest of approximately 30~35Å at 250 °C, and decreased according to the increase in the carbonization temperature. Average pore size of the carbonized sewage sludge obtained from 450 °C was approximately 17Å compared to the approximately 23~27Å of the dried sewage sludge.
6. Iodine adsorption performance of the carbonized excess sewage sludge obtained from 350 °C were approximately 180~240mg/g compared to the approximately 110~160mg/g of the dried sewage sludge.
7. As the results of reviewing the BET specific surface area, pore size and the iodine adsorption performance test of the carbonized excess and digested sewage sludge, the optimum carbonization temperature was 350<“450! and the optimum carbonization time was derived in the range of 30<“60 minutes, when using the sewage sludge as the raw material for adsorbent production.

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