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Investigation into COD Component in the Wastewater at Sea Reclamation Waste Disposal Site and Its Influence on the Coagulation-Sedimentation and the Activated Carbon Adsorption

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Abstract: The relationship between COD component and the Coagulation-Sedimentation or the activated carbon adsorption, which were performed at the existing effluent water treatment facility, was investigated effectively to treat COD of the wastewater at the sea reclamation waste disposal site. COD could decrease by only bubbling because COD except for organic matters was contained in waste and wastewater. It is effective to measure the ion property of organic-COD in some waste and select an adequate coagulant. However, it was not necessary that the ion property was related to COD removal ratio. In the activated carbon adsorption, the batch method was very effective but it is necessary to examine the solid-liquid separation after the treatment. Besides, the classification of disposal site for COD treatment and the introduction of the additional charge system calculated on the effluent treatment cost were considered.

Keywords: Sea Reclamation Waste Disposal Site, Leachate, COD Component, Coagulation-Sedimentation,
Activated Carbon Adsorption

1. Introduction

Various wastes as sludge or cinder are carried and are reclaimed at a reclamation waste disposal site in proportion to the variety and the complication of industry. Then, it is considered that hardly degradable organic matters, metals and chemical substances exist in leachate. In fact, COD concentration of wastewater at Hibikinada reclamation waste disposal site in Kitakyushu City becomes over 100 mg L^{-1} at the end of reclamation and it is difficult to decrease at below 20 mg L^{-1} (aiming value) by the existing effluent treatment (contact oxidation – the Coagulation-Sedimentation with $FeCl_3$ – sand filtration – particulate activated carbon adsorption). As this countermeasure, the pile construction [1], the Coagulation-Sedimentation with powder activated carbon, the penetration of wastewater into the reclaimed wastes [2, 3]

and the purification by a little stream [4] were examined. But, they were not practical. The reason is considered that the COD components in wastes can be hardly treated with normal coagulant or activated carbon.

Then, we examined COD and total organic carbon (TOC) in 9 kinds of wastes which were carried and reclaimed at the disposal site and contained high concentration of COD. The relationships between the COD components in 6 kinds of wastes and the Coagulation-Sedimentation or the activated carbon adsorption were examined. Further, we also examined the selection and the design of appropriate coagulant or activated carbon and the prospective system for the acceptance of waste.

2. Materials and Methods

2.1. Reagents

The strong acid cation-exchange resin and the strong basic anion-exchange resin were a Wako Pure Chemical Industrials Dowex 50Wx8 (100-200 mesh) and a Dowex 1x8 (100-200 mesh), respectively.

$FeCl_3$ anhydride and polyaluminum chloride (PAC) were obtained from Wako Pure Chemical Industries and Kurosaki Chemical Industry, respectively. The strong cation-polymer coagulant, the nonion-polymer coagulant, the strong anion-polymer coagulant and the inorganic nonion-coagulant were a MT Aqua Polymer Aronfloc C-508 (polyacrylic ester), a Accofloc N-100S (polyacrylamide), an Accofloc A-150 (polyacrylamide) and a Noatech Supernammit, respectively.

The granular activated carbons were a Taihei Chemical Industrial Yashicoal S.SC, a Yashicoal M.MC, a Yashicoal L.LC, a Procoal CM and a Procoal GN. Their properties were shown in Table 1.

The standard molecular weight reagents were a VARIAN PL 2070-0100 (polyethylene glycol; molecular weight: 106-21,030) and a PL 2080-0101 (polyethylene oxide; molecular weight: 15,120-859,500). Other reagents were equally with JIS K 0102 [5]. The pure water was purified using a Japan Millipore Milli-Q SP.

Table 1. Properties of each activated carbon.

	Yashicoal S.SC	Yashicoal M.MC	Yashicoal L.LC	Procoal CM	Procoal GN
Material	Coconut shell	Coconut shell	Coconut shell	Coal	Coal
Activation	Steam	Steam	Steam	Steam	Steam
Drying loss (%)	3.9	1.5	2.6	2.0	0.5
Particle size (mm)	0.50-0.25	2.36-0.50	4.75-2.36	2.36-0.50	4.75-3.35
Infill rate (g mL^{-1})	0.48	0.47	0.49	0.43	0.46
Specific surface area (m^2 g^{-1})	1,050	1,157	960	1,137	1,228
Average fine pore size (Å)	17.9	18.4	17.8	20.0	18.7
Fine pore volume (cm^3 g^{-1})	0.469	0.532	0.428	0.570	0.574
I$_2$-adsorption (mg g^{-1})	1,200	1,140	1,100	1,080	1,100
Benzene-adsorption (%)	36.9	34.8	34.1	35.3	36.1
Methylene blue-adsorption (mL g^{-1})	220	200	200	190	190

2.2. Instruments

pH meter and electric conductivity (EC) meter were a Horiba F-53 and a Toa Electronics CM-40S, respectively. TOC meter was a Shimadzu TOC-5000/an auto sampler ASI-5000A. Spectrophotometer was a JASCO NQF-720/U-630. Atomic absorption spectrometer (AAS) was a Varian AA240 FS. Ion chromatograph was a Nippon Dionex/an auto sampler DAS-80/a Shimadzu column oven CTO-10A. Jar tester was a Miyamoto Riken Industries JT-6D.

2.3. Samples

2.3.1. Wastewater at the Disposal Site

To investigate the wastewater at the disposal site, 2 drilling hole waters were sampled in December 2011.

2.3.2. Carried Waste

Above 10 kinds of wastes containing high concentration of COD are carried and are reclamated at the disposal site. Incineration residue of domestic waste (incineration plant),

Sludge A (intermediate waste treatment) and Sludge B (intermediate waste treatment) were collected in May 2009. Slag A (casting sand), Slag D (containing binder) and Dust (chelating and containing cement) were collected in June 2011. Other wastes were collected in 2008-2011.

2.4. Elution Test

The elution tests for Sludge C (intermediate waste treatment)), Cinder A (incineration ash), Cinder B (chelating), Slag A, Slag B (casting sand) and Slag C (casting sand) were also done and COD and TOC were measured. The elution test was equally performed with "the Verification Methods for Metals et. al. Contained in Industrial Waste" [6]. The ratio of waste (dry matter) and purified water or sea water was 3 w v^{-1} % and the mixture was shaken for 6-h.

2.5. Preparation of Test Solution

As the test solution for the component examination of COD, total nitrogen (T-N) and total phosphorous (T-P), the Coagulation-Sedimentation and the activated carbon

adsorption, 0.5 kg of waste was mixed with 17 L of purified water, the mixture was stand for a day and the supernatant was filtrated with a 1 μm glass fiber filter paper (GFP).

2.6. Bubbling

The bubbling for 50 mL of test solution was done at 200 mL min^{-1} for 30-min and COD and inorganic-COD were measured by the method in 2.9.1.

2.7. Coagulation-Sedimentation

2.7.1. Coagulation-Sedimentation with Several Kinds of Coagulant

Each 500 mL of the test solution prepared in 5 was placed in 6 of 1 L beaker and each 0-100 mg L^{-1} of coagulant was added. The prepared solutions were rapidly mixed for 1-min at 150 rpm and then were slowly stirred for 10-min at 50 rpm with a jar tester. After 30-min of quiescent settling, the supernatant was filtrated with a 1 μm GFP and the filtrate was analyzed.

2.7.2. Influence of Rapid Mixture

For Sludge A, the removal ratio was examined with 50 mg L^{-1} of FeCl$_3$ at 0-350 rpm of rapid mixture. The Coagulation-Sedimentation was performed at the best pH.

2.7.3. Influence of Anion

For Sludge A, the influence of anion was examined with 50 mg L^{-1} of FeCl$_3$ by adding 0-2.0 g of NaCl or 0-2.96 g of Na$_2$SO$_4$.

2.8. Activated Carbon Adsorption

2.8.1. Adsorption with a Column

The glass chromatocolumn (the inside diameter: 1 cm; length: 30 cm) was packed with 3 g of activated carbon and was washed with 400 mL of purified water. One hundred milliliter of test solution (TOC: 10 mg L^{-1}) was path through the column and the adsorption ratio of TOC was calculated.

For the adsorption of metal ions, each 100 mL of Ni$^+$ and Fe^{3+} solution (10 mg L^{-1}) was prepared. The solution was used for the column test.

2.8.2. Adsorption with a Jar Tester

Each 500 mL of Ni$^+$ and Fe^{3+} solution (10 mg L^{-1}) was placed in 6 of 1 L beaker and each 3 g of activated carbon was added. The prepared solutions were rapidly mixed for 1-min at 150 rpm and then were slowly stirred for 10-min at 50 rpm. After 30-min of quiescent settling, the supernatant was filtrated with a 1 μm GFP and the filtrate was analyzed.

2.9. Analytical Methods

2.9.1. COD and Inorganic-COD

COD was measured by JIS K 0102 [5]. COD value contains the inorganic reducing matters as H$_2$S, metals, etc. These matters were easy to be oxidized with KMnO$_4$. Then, we defined the COD without 30-min of heating as the inorganic-COD.

2.9.2. Ionic Properties of COD, TOC, T-N and T-P

The glass chromatocolumn (the inside diameter: 3 cm; length: 40 cm) was packed with 200 mL of the strong acid cation-exchange resin and was washed twice with 200 mL of 1 N HCl, 200 mL of purified water, 200 mL of 1 N NaOH and 200 mL of purified water in turn. In the same way, the chromatocolumn was packed with 200 mL of the strong basic anion-exchange resin and was washed twice with 200 mL of 1 N NaOH, 200 mL of purified water, 200 mL of 1 N HCl and 200 mL of purified water in turn. One hundred milliliter of test solution prepared in 5 was path through each column at 20 mL min^{-1}, each decreased concentration of COD, T-N and T-P were measured and each ratio of cation, nonion and anion was calculated.

2.9.3. Waste and Water Quality

Water content and ignition loss (IL) were measured by Guideline for Lake Environmental Survey [7]. pH, EC, COD, TOC, T-N, T-P, F$^-$, Br$^-$, Cl$^-$ and SO$_4^{2-}$ were measured by JIS K 0102 [5].

3. Results and Discussion

3.1. Water Quality of Leachate

3.1.1. Drilling Hole Water

The COD values of Drilling hole water A and Drilling hole water B were 125 and 196 mg L^{-1}, respectively. The ratios of inorganic-COD were 17.6 and 38.8%, respectively (Fig. 1). By bubbling for 30-min, the inorganic-COD in Drilling hole water B was removed. Forty point eight percent of nonion organic-COD was contained in Drilling hole water A. Twenty four percent of cation organic-COD and 22.4% of anion organic-COD were contained in Drilling hole water B. The ratios for TOC were nearly same as those for COD (Fig. 2).
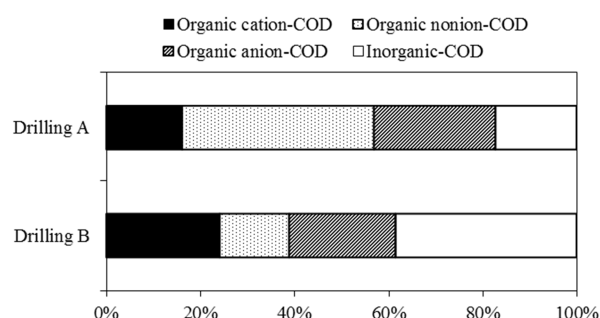

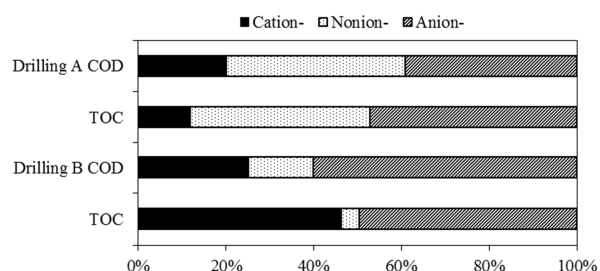**Fig. 1.** *Component ratio of COD in the drilling hole water.*

Fig. 2. *Component ratios of COD and TOC in the drilling hole water.*

3.1.2. Elution Test for Carried Waste

Table 2 showed the results of COD and TOC in the elution test with sea water for 6 wastes containing large amount of organic matters. The COD values were 50.2 (Cinder A) - 480 mg L^{-1} (Slag C) and the TOC values were 40.6 (Cinder B) - 740 mg L^{-1} (Slag C). The ratios of COD/TOC were 0.65 (Slag C) - 1.9 (Sludge C).

The results of the elution tests with purified water for Incineration residue of domestic waste, Sludge A and Sludge B were shown in Table 3. pH was 9.52-10.1. Ten point nine milligrams per liter of T-N and 1.52 mg L^{-1} of T-P eluted

from Sludge A at high concentration. High concentration of Cl$^-$ (1,230 and 105 mg L^{-1}) and SO$_4$$^{2-}$ (710 and 570 mg L^{-1}) eluted from Incineration residue of domestic waste and Sludge B, respectively. High concentration of F$^-$ (9.9 mg L^{-1}), Br$^-$ (1,650 mg L^{-1}), Cl$^-$ (3,330 mg L^{-1}) and SO$_4$$^{2-}$ (1,870 mg L^{-1}) eluted from Sludge A. Then, the EC level (131-930 mS m^{-1}) of these wastes was high.

3.1.3. Component Ratios of COD, TOC, T-N and T-P in the Carried Waste

***Table 2.** Results of the elution test for the wastes containing large amount of organic matters.*

	Sludge C	Cinder A	Cinder B	Slag A	Slag B	Slag C
COD (mg L^{-1})	100	50.2	73.1	245	220	480
TOC (mg L^{-1})	53.3	47.8	40.6	260	290	740
COD/TOC	1.9	1.1	1.8	0.94	0.76	0.65

***Table 3.** Results of the elution test for the wastes.*

	Incineration residue of domestic waste	Sludge A	Sludge B
Water content (%)	9.0	27.6	18.9
IL (%)	5.2	19.8	6.7
pH	10.0	10.1	9.52
EC (mS m^{-1})	370	930	131
COD (mg L^{-1})	30.9	48.5	15.6
TOC (mg L^{-1})	26.4	94.9	4.9
COD/TOC	1.2	0.51	3.2
T-N (mg L^{-1})	0.40	10.9	0.92
T-P (mg L^{-1})	0.60	1.52	0.57
T-P/T-N	1.5	0.14	0.62
F$^-$ (mg L^{-1})	0.9	9.9	1.4
Br$^-$ (mg L^{-1})	3.9	1,650	2.6
Cl$^-$ (mg L^{-1})	1,230	3,330	105
SO$_4$$^{2-}$ (mg L^{-1})	710	1,870	570

Figure 3 showed the component ratios of COD in Incineration residue of domestic waste, Sludge A and Sludge B. Thirteen point four to 33.5% of inorganic-COD was contained. Incineration residue of domestic waste contained much organic cation-COD (43.2%) but Sludge A (2.2%) and Sludge B (3.1%) contained small amount of organic cation-COD. As the reclamation ratio at the disposal site was about 40% of incineration residue of domestic waste, about 30% of construction waste and about 10% of sludge, the drilling hole water contained relatively large amount of organic cation-COD. Sludge A and Sludge B contained much nonion-COD (29.4 and 30.9%, respectively). Anion-COD was 33.8-36.1%.
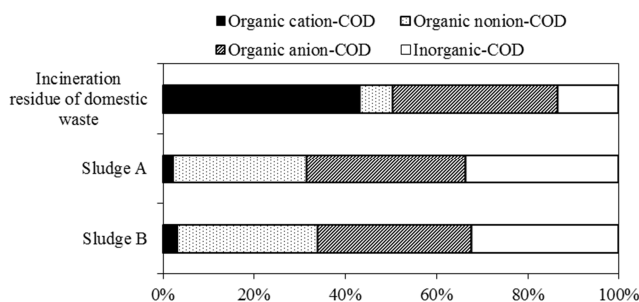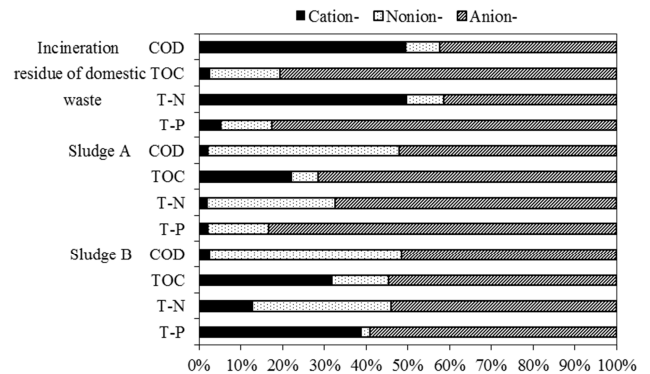

Fig. 4. Component ratios of COD, TOC, T-N and T-P in the carried waste.

Figure 4 showed the component ratio of COD, TOC, T-N and T-P. The ratio of TOC was different from that of COD. In Incineration residue of domestic waste, cation-TOC (2.5%) was a little and nonion-TOC (16.9%) or anion-TOC (80.6%) was much. In Sludge A and Sludge B, nonion-TOC (6.5 and 13.6%, respectively) was a little and cation-TOC (22.1 and 31.8%, respectively) or anion-TOC (71.4 and 54.6%, respectively) was much. Generally, the part of N becomes cation and the part of P becomes anion. In Incineration residue of domestic waste, cation-T-N (49.7%) or anion-T-N (41.4%) was much and nonion-T-N (8.9%) was a little. In

Fig. 3. Component ratio of COD in the carried waste.

Sludge A and Sludge B, cation-T-N (1.9 and 12.8%, respectively) was a little and nonion-T-N (30.8 and 33.3%, respectively) or anion-T-N (67.3 and 53.9%, respectively)

was much. As for T-P, cation-T-P (5.2 and 2.1%, respectively) was a little in Incineration residue of domestic waste and Sludge A. In Sludge B, cation-T-P (38.8%) was much.

Table 4. Change (%) of COD by bubbling.

	Incineration residue of domestic waste	Sludge A	Sludge B	Slag C	Slag D
Inorganic-COD	13.4	33.5	32.2	14.4	40.8
COD after bubbling	93.5	106	119	100	91.7
Change of COD by bubbling	-6.5	+6	+19	0	-8.3

Table 5. Maximum removal ratio (%) with each coagulant.

		FeCl₃	PAC	Aronfloc C-508	Accofloc N-100S	A-150	Supernammit
Incineration residue of domestic waste	COD	22	37	27	19	17	26
	TOC	24	28	25	6	16	27
	T-N	31	34	44	22	28	14
	T-P	25	38	22	31	27	11
Sludge A	COD	18	24	7	35	12	10
	TOC	4	9	7	16	9	11
	T-N	8	5	14	28	27	4
	T-P	7	10	12	21	22	5
Sludge B	COD	16	29	18	39	21	17
	TOC	29	31	17	26	19	22
	T-N	4	14	29	22	22	16
	T-P	17	21	18	25	23	15
Slag C	COD	84	62	62	75	56	69
	TOC	55	92	91	92	91	89
	T-N	-	-	-	-	-	-
	T-P	76	77	82	64	62	65
Slag D	COD	13	13	15	17	15	13
	TOC	12	11	14	13	11	9
	T-N	-	-	-	-	-	-
	T-P	-	-	-	-	-	-
Dust	COD	-	-	-	-	-	-
	TOC	-	-	-	-	-	-
	T-N	-	-	-	-	-	-
	T-P	-	-	-	-	-	-

(☐ : 50% ≦)

3.2. Change of COD by Bubbling

Drilling B contained 38.8% of inorganic-COD and the COD was removed by bubbling. Then, the effect of bubbling was performed on the wastes (Table 4). Though these wastes contained 13.4-40.8% of inorganic-COD, only 6.5% (Incineration residue of domestic waste) and 8.3% (Slag D) of COD were removed by bubbling. On the contrary, 6 and 19% of COD increased in case of Sludge A and Sludge B, respectively. As the drilling hole water was in anaerobic condition for a long time, much reducing matters generated and corresponded inorganic-COD was removed by bubbling.

3.3. Coagulation-Sedimentation

3.3.1. Removal Ratios with Several Kinds of Coagulant

Table 5 showed the maximum removal ratios with several kinds of coagulant. In Slag C, relatively high removal ratios (COD: 56-84; TOC: 55-92; T-P: 62-82%) were obtained with all coagulants. In the other wastes, those of COD, TOC, T-N and T-P were below 39, 31, 44 and 38%, respectively.

In Incineration residue of domestic waste, Sludge A and Sludge B, the relationship between the removal ratio and the

component ratio was examined. Maximum removal ratios of COD, TOC, T-N and T-P were 39, 31, 44 and 38%, respectively. As 33.8-36.1% of organic anion-COD was contained (Fig. 3), 7-37% of COD was removed with the cation-coagulant (FeCl₃, PAC and Aronfloc C-508). Especially, the removal ratios with PAC (24-37%) were high. The removal ratio with organic cation-coagulant Aronfloc C-508 (7%) was low. In Sludge A and Sludge B which contained 29.4 and 30.9% of organic nonion-COD, the removal ratios of COD with organic nonion-coagulant Accofloc N-11S showed the highest values of 35 and 39%, respectively. In Incineration residue of domestic waste which contained 7.3% of organic nonion-COD, 19% of COD was removed with Accfloc N-100S. But, only 17% of COD was removed with organic anion-coagulant Accofloc A-150 in spite of containing 43.2% of organic cation-COD. Though Sludge A and Sludge B contained small amount of organic cation-COD (2.2 and 3.1%, respectively), 12 and 21% of COD were removed with Accofloc A-150, respectively. The removal ratio was also affected by the interaction among organic matters.

T-N in Slag C and T-N and T-P in Slag D could not be

removed or increased by adding the coagulant. As Dust contained much minute carbon particles (1 μm >), the effect of Coagulation-Sedimentation could not observed.

3.3.2. Influence of Rapid Stirring Speed on Removal Ratio

The coagulation effect is also affected by stirring speed. The removal ratios of COD, TOC, T-N and T-P were examined with 50 mg L^{-1} of $FeCl_3$ at 0-350 rpm of rapid stirring speed for Sludge A. But, the ratios were almost same as at 150 rpm.

3.3.3. Influence of the Coexistence with Anion

The influence of Cl^- was examined with 50 mg L^{-1} of $FeCl_3$ by adding 0-2.0 g of NaCl for Sludge A. The ratios of TOC and T-P did not change very much. However, the ratios of COD (19.2%) and T-N (29.8%) increased. The effect of salting out is considered. Similarly, the influence of SO_4^{2-} was examined with 50 mg L^{-1} of $FeCl_3$ by adding 0-2.96 g of Na_2SO_4. The ratio of only T-P (15.7%) increased.

3.4. Activated Carbon Adsorption

3.4.1. Adsorption Ratio of Standard Molecular Weight Reagent as TOC

Figure 5 showed the adsorption ratios of standard molecular weight reagents as TOC with several kinds of activated carbon. Above 90% of adsorption ratio was obtained on the whole molecular weight in case of Yashicoal S.SC which particle size was the smallest (Table 1). In this column test, particle size largely affected than specific surface area, average fine pore size and fine pore volume. In case of other activated carbon, lower molecular weight reagents hardly adsorbed because they were more hydrophilic. The higher molecular weight is and the higher adsorptivity is because it becomes less hydrophilic. Further, the higher molecular weight is and the lower adsorptivity is because the adsorbate cannot go into the fine pore. The peak was observed in the range of 10^2-10^3. In case of Procoal CM and Procoal GN, the peak was also observed in the range of 10^5-10^6.
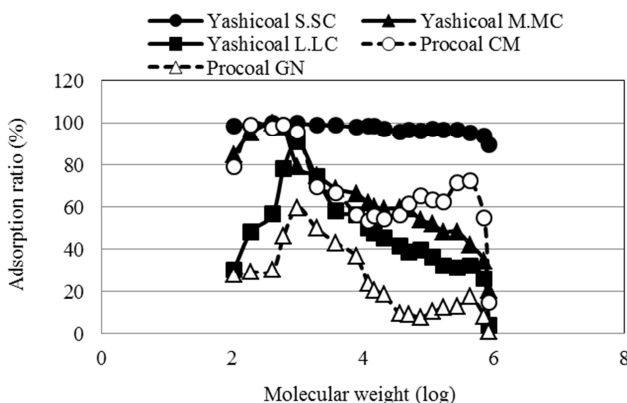
Fig. 5. Adsorption ratio of each standard molecular weight reagent as TOC.

Initial TOC: 10 mg L^{-1}, 100 mL; activated carbon: 3 g.
There are a few differences of adsorptivity on each activated carbon for I_2, benzene and methylene blue (Table 1).

The reason is that the adsorption test was performed by the batch method [8]. The column adsorption test was performed because the actual treatment was performed by the adsorption tower system. If the solid-liquid separation method such as the sloped-precipitation pipe method [9] would be introduced, the adsorption by the batch method can be performed and the increase of adsorptivity would be expected.

3.4.2. Adsorption Ratios of Metal Ions

The adsorption tests of metal ions were performed with Yashicoal S.SC, Yashicoal M.MC and Procoal GN. In case of column adsorption test, the ratios of Ni^+ and Fe^{3+} were 100, 18.4 and 0% and 52.3, 90.2 and 23.2%, respectively (Fig. 6). On the other hand, all activated carbons showed 100% of ratio for Fe^{3+} in case of jar test. The contact with activated carbon also effects on the ratio in case of metal ions (chemical adsorption).
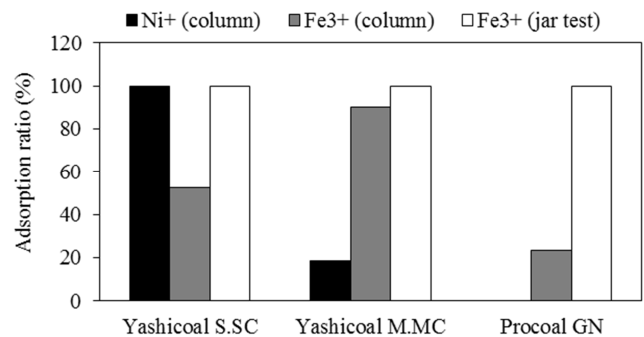
Fig. 6. Adsorption ratio of metal ions.

Initial metal ions: Ni^+ 10 mg L^{-1}, Fe^{3+} 10 mg L^{-1}, 100 mL (column) or 500 mL (jar test); activated carbon: 3 g.

3.4.3. Adsorption Ratio of TOC in Wastes

The column adsorption tests for Incineration residue of domestic waste, Sludge A, Sludge B, Slag C, Slag D and Dust were performed. The adsorption ratios of TOC with Yashicoal S.SC, Yashicoal M.MC and Procoal GN were 55.5-92.7, 51.4-84.9 and 12.5-39.8%, respectively (Fig. 7). The ratios of TOC in Slag D and Dust were lower. It is also considered that the ratios of TOC in wastes increase by the batch method.
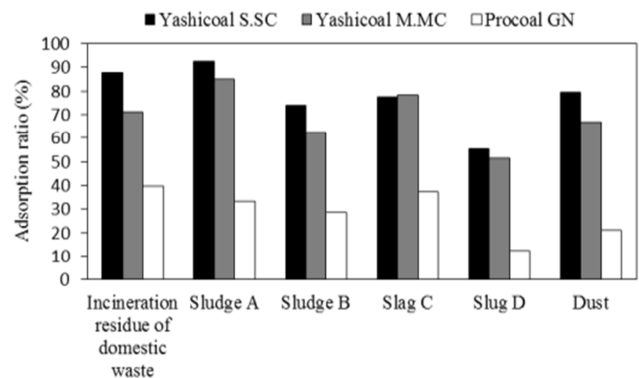
Fig. 7. Adsorption ratio of TOC.

Initial TOC: 10 mg L^{-1}, 100 mL; activated carbon: 3 g.

4. Conclusion

In order to treat COD in the wastewater at the sea reclamation waste disposal site effectively, the relationship between COD component and the Coagulation-Sedimentation or the activated carbon adsorption, which were performed at the existing effluent water treatment facility, was investigated.

(1) As the COD except for organic-COD was contained in waste and wastewater, COD value could be reduced by bubbling.

(2) In some waste, COD can be treated effectively by examining the ionic property of organic-COD and using a proper coagulant.

(3) Though the activated carbon adsorption by the batch method was very effective, it is necessary to use the effective solid-liquid separation.

(4) The classification of disposal site for COD treatment like recycling should be considered.

(5) Though the membrane treatment was effective, large amount of concentrated wastewater must be treated [10].

(6) The countermeasures against Cl- and hazardous substances [11-15] are necessary to discharge into the sewerage.

(7) The introduction of the additional charge system calculated on the effluent treatment cost was considered.

Acknowledgements

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