

Physicochemical properties of nano-pore adsorbents to remove volatile organic compounds in aqueous phase

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The nano-pore adsorbents, which were waste-reclaimed (WR), shale-based (S), red clay-based (R), and blue kaolin-based (B), were investigated for their physicochemical characteristics to remove benzene, toluene, ethylbenzene and xylene (BTEX). The BET and SEM-EDS analysis showed that WR, which was a mixture of bottom ashes from a power plant and dredged soil, had the larger surface area (i.e. $14.56 \text{ m}^2 \text{ g}^{-1}$), the rougher surface topography, and the higher total iron (i.e. 5.46 wt% of Fe) than others. Moreover, the XRD analysis indicated that all adsorbents contained aluminum-silica complexes and particularly, both S and WR also contained the iron complexes. The iron complex on S was identified as hercynite (FeAl_2O_4) while two types of iron oxides (i.e. hematite ($\alpha\text{-Fe}_2\text{O}_3$) and maghemite ($\gamma\text{-Fe}_2\text{O}_3$)) existed on the surface of WR. Langmuir model generally predicted BTEX adsorption on nano-pore adsorbents and the calculated adsorption capacities (Q_0) of WR for BTEX were 0.752, 3.793, 1.678, and 4.902 mg g^{-1} , respectively. In addition, the more BTEX was removed in $\text{H}_2\text{O}_2/\text{WR}$ system than others. This relatively high BTEX removal by $\text{H}_2\text{O}_2/\text{WR}$ could be explained by both the larger surface area and catalytic property, which was mainly from the coexistence of hematite and maghemite. Moreover, even though S had hercynite, it did not show much catalytic properties to degrade BTEX in solution.

Key words: nano-pore adsorbents, waste-reclaimed adsorbent, hematite, maghemite, BTEX removal, hydrogen peroxide.

Introduction

Benzene, toluene, ethylbenzene and xylene (BTEX) are the major organic contaminants that threaten the environment, and they have been treated by various treatment technologies [1-5]. Generally, adsorption has been recognized as the one of dominant mechanisms for BTEX removal, and the application of various types of adsorbents and their modifications has been investigated [6-9].

The numerous studies on BTEX adsorption revealed that adsorption process could depend on the complexity of surface characteristics of adsorbents and the pH of solution [10], and examples of adsorbents used to treat BTEX contamination were activated carbon, zeolite, clay mineral, and their modifications. Activated carbon and its modifications are widely used as effective adsorbents to remove organic contaminants from water, and it was suggested that the adsorption mechanism of BTEX occurred through π - π interaction between the aromatic ring and the carbonyl groups on activated carbon [8]. Moreover, lowering surface acidity reduced the polarity of the surface and increased adsorption of hydrophobic BTEX [10]. However, efforts have continued to reduce production/regeneration cost of activated carbons [10, 11].

Zeolite, the one of well-known adsorbents, has been generally applied to remove cationic transition metals such as Pb^{2+} and Cd^{2+} , and the surfactant-modified zeolite was synthesized to remove non-polar organic and anionic inorganic contaminants [6]. The surfactant-modified zeolite showed that its adsorption capacity was increased by increasing either surfactant loadings or carbon composites of the surface that these provided hydrophobic properties [12-14]. Moreover, an air sparging technique was suggested to be applied to regenerate surfactant-modified zeolite without loss of adsorption capacity [12].

Organo-clays, which were prepared from clay minerals (i.e. Na-montmorillonite, sericite, hectorite, and bentonite) by cation exchange with various types of organic cations including surfactants, showed that these organic cations expanded the interlayer of clays to increase the adsorption of contaminants including BTEX [7, 15-17]. Moreover, it was suggested that organo-clays could be applied for groundwater contamination because of their properties such as retention capacity and hydrophobicity, even though they have disadvantages in terms of cost, biodegradability, and recyclability [17].

Moreover, waste materials as potential adsorbents have recently attracted attention because they reduce waste disposal costs [9, 18, 19]. The various waste materials have shown great potential for the adsorption of various contaminants, and the physical and chemical stability of waste-reclaimed adsorbent should be investigated and proved before their utilization [19].

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In addition to the adsorption mechanism, degradation using chemical oxidants has been widely practiced, and hydrogen peroxide with soluble iron, iron chelates, or iron minerals have been applied to remove organic contaminants [20-22]. The heterogeneous catalytic decomposition of hydrogen peroxide (Fenton-like reaction) has an advantage of high efficiency at neutral or nearly neutral pH [22, 23], and various types of materials like iron minerals, clays, silicas, zeolites, and activated carbons have shown their catalytic characteristics to generate strong oxidant, hydroxyl radical (HO) [23-26].

The present study investigated the feasibility of nano-pore adsorbents (i.e. waste-reclaimed (WR), shale-based (S), red clay-based (R), and blue kaolin-based (B)) for BTEX removal. First of all, adsorbents (e.g. physical/chemical characteristics, XRD, and SEM) were analyzed. After that, these four types of adsorbents were tested for BTEX adsorption and the catalytic characteristics were evaluated when hydrogen peroxide was used at the initial pH of 7.0.

Experimental Procedures

Benzene was purchased from Yakiri Pure Chemicals, ethylbenzene from Junsei Chemicals, and toluene and xylene (i.e. a mixture of three xylene isomers) from Carlo Erba Reagenti. Dichloromethane for BTEX extraction was purchased from Fisher Scientific. Hydrogen peroxide (H_2O_2), sulfuric acid (H_2SO_4), and titanium (IV) sulfate hydrate ($\text{Ti}(\text{SO}_4)_2 \cdot n\text{H}_2\text{O}$) were purchased from Junsei Chemicals. The nano-pore adsorbents (i.e. waste-reclaimed (WR), shale-based (S), red clay-based (R), and blue kaolin-based (B)) were supplied from the center for resource processing of solid wastes (CRP-SW) at Kyonggi University. These adsorbents were washed ten times in de-ionized water with a resistivity of $18 \text{ m}\Omega \text{ cm}$ (Millipore system, Young-Lin Co., Korea), then dried at 105°C for 1 day, and stored in a desiccator.

All reaction samples were prepared in duplicate, and the average value was used for analysis. The initial pH of all solutions was adjusted to 7.0 by adding 5 N NaOH or 1 N HNO_3 . Benzene, toluene, ethylbenzene, and xylene solutions were prepared by adding the required volume of pure chemicals and stirring for 12 hrs in a 2.8 L stainless steel reactor with no headspace. The concentrations of contaminants were 100 mg L^{-1} , respectively.

The experiments for adsorption isotherms were conducted using the bottle-point method. The amount of nano-pore adsorbents was fixed as 2.5 g (i.e. the volume of solids was approximately 1.6 mL) and the concentrations of contaminant (B, T, E, X) were varied from 10 to 100 mg L^{-1} . Each contaminant solutions were filled in a 40 mL glass vial with no headspace (i.e. actual volume of vial was $45.4 \pm 0.5 \text{ mL}$ when $n = 7$). These batch reactors were mounted on the

shaking incubator (LPN-0201F-S, Hanbaek ST.) for 12 hrs at 200 rpm and 25°C . The preliminary test for adsorption kinetic showed that equilibrium was reached within 10 h for B, T, E, and X solutions with all adsorbents. After 12 hrs of shaking, 5 mL of a sample was extracted with 5 mL of dichloromethane. The loss of BTEX in the control sample was negligible.

The concentration of hydrogen peroxide was 500 mM, and the amount of adsorbents in a 40 mL batch reactor was 2.5 g, and the total volume of solution was 40 mL. Experimental systems were prepared including control (only contaminants), adsorbents/contaminants, oxidant/contaminants, and adsorbents/oxidant/contaminants. These prepared systems were mounted on the shaking incubator at 200 rpm and 25°C . Samples were retrieved at each sampling times, and an extraction vessel containing 5 mL of dichloromethane was used for extraction of 5 mL sample. Moreover, the sorbed contaminants were also analyzed by removing additional 30 mL solution from reaction vial and adding 5 mL of dichloromethane into a vial remaining 5 mL solution and adsorbents.

Extraction was conducted with 4 min of vortex mixing (TTS3, IKA) followed by 20 min of sonication (8510R-DTH, Banson). Then, the extracted BTEX was detected by using HP-6850 gas chromatograph equipped with a flame ionization detector. The chromatograph was equipped with a capillary column (J & W scientific, Model HP-5; $30.0 \text{ m} \times 530 \mu\text{m} \times 0.88 \mu\text{m}$), and injector and detector temperatures were maintained at 200 and 340°C , respectively. The oven temperature was started at 40°C , which was held for 2 min, ramped at $10^\circ\text{C min}^{-1}$ to 100°C followed by ramping at $40^\circ\text{C min}^{-1}$ to 280°C , and held for 2 min at 285°C . Nitrogen was the carrier gas and a split ratio of 10:1 was used. The colorimetric method (Milton Roy Spectronic 20⁺) was used to detect hydrogen peroxide at 467 nm [27]. The reagent for the residual H_2O_2 detection was titanium sulfate, and 2 mL : 2 mL of reagent: sample was rapidly mixed and kept in dark place for 2 min. The pH was measured by using a pH-200L meter (ISTEK). Moreover, XRD analysis was conducted to identify crystalline materials on the surface of adsorbents, and X-ray diffraction curves were recorded using a Denki D/MAX RINT 2000 diffractometer with Cu $K\alpha$ radiation at 40 kV and 100 mA. Scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM-EDS, Jeol JSM-6330F) was used for surface analysis.

Results and Discussion

Characteristics of nano-pore adsorbents

The manufacturing processes for nano-pore adsorbents (i.e. waste-reclaimed (WR), shale-based (S), red clay-based (R), and blue kaolin-based (B)) were followed briefly. First of all, raw materials were

Table 1. Chemical compositions (%) of nano-pore ceramic substrates.

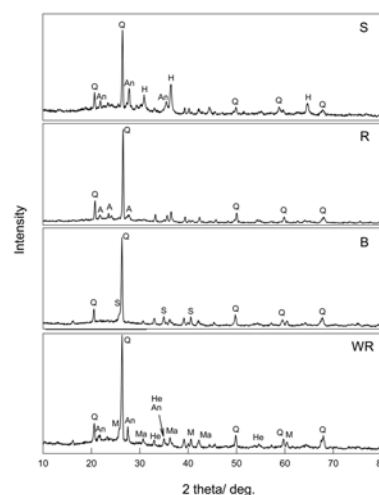
	WR ^a		S ^b	R ^c	B ^d
	Bottom ash	Dredged soil			
SiO ₂	45.58	70.71	51.34	57.86	59.52
AlO ₃	18.60	14.38	20.55	19.08	23.34
Fe ₂ O ₃	8.08	3.82	15.40	7.07	3.72
CaO	2.17	0.79	5.32	0.20	0.27
MgO	0.78	0.18	2.23	1.04	0.39
Na ₂ O	0.18	2.51	0.31	0.09	1.39
K ₂ O	0.51	2.70	2.54	2.54	2.89
TiO ₂	1.33	0.80	0.89	0.92	0.2
P ₂ O ₅	0.24	0.03	0.32	0.17	0.02
MnO	0.05	-	0.23	0.22	-
ZrO ₂	0.33	-	-	-	-
SO ₃	-	-	0.49	-	-
C	18.07	-	0.17	-	-
Ignition loss	4.07	4.08	0.14	11.03	8.21
Total	100.00	100.00	99.94	100.22	99.95

^a: Waste-reclaimed (bottom ash: dredged soil = 7 : 3).^b: Shale-based.^c: Red-clay-based.^d: Blue kaolin-based.**Table 2.** Physical properties of nano-pore ceramic substrates.

Shape	BET surface area (m ² g ⁻¹)	Total pore volume (mm ³ g ⁻¹)	Pore diameter (nm)	Specific gravity	Porosity (%)
WR Spherical	14.56	6.72	1.41	1.41	30.1
S Spherical	2.23	1.13	2.03	0.75	40.27
R Irregular	0.34	0.17	1.95	1.44	44.5
B Irregular	0.36	0.18	2.00	1.05	53.63

pulverized until they were below 150 μm . After that, in the molding process, a pelletizer was used for WR and S (spherical), and a compression molding was used for R and B followed by the cutting process (cubic). These molded lumps dried at 110–120 $^{\circ}\text{C}$ for 48 hrs. Calcination for WR and S was done by rotary kiln at 1125–1150 $^{\circ}\text{C}$ for 15 min and for R and B by shuttle kiln at 1125 $^{\circ}\text{C}$ for 2 hrs. The 4.75–9.60 mm particles of adsorbents were collected and used for experiments.

Waste-reclaimed adsorbent (WR) was prepared by mixing two raw materials, one of which was bottom ash generated from a power plant and the other was dredged soil. The ratio of bottom ash to dredged soil was 7 : 3. Shale-based adsorbent (S) was prepared by the expanded shale, which is also used for lightweight construction material. Red clay-based (R) was a mixture of red clay and sawdust. The ratio based on weight percentages of red clay and sawdust was 95 : 5.

**Fig. 1.** XRD patterns of nano-pore ceramic substrates. [Q: quartz (SiO₂), An: anorthite (CaAl₂Si₂O₈), H: hercynite (FeAl₂O₄), A: albite (NaAlSi₃O₈), He: hematite (α -Fe₂O₃), S: sillimanite (Al₂SiO₅), M: mullite (3Al₂O₃ • 2SiO₂), and Ma: maghemite (γ -Fe₂O₃)].

Blue kaolin-based adsorbent (B) was produced by mixing kaolin and surfactant, which was sodium lauryl sulfate (SLS). The role of this anionic surfactant was the same as that of sawdust, which makes a large pore when calcination has progressed. The chemical compositions of raw material and physical characteristics of adsorbent are shown in Table 1 and 2.

In Table 1, chemical compositions of raw material in WR showed that bottom ash had relatively high percentages of TiO₂ (1.33%) and carbon element (18.07%) and dredged soil showed the higher SiO₂ (70.71%). S had relatively high percentages of Fe₂O₃ (15.40%) and CaO (2.23) while R had the highest ignition loss (11.03%). Moreover, B showed the highest Al₂O₃ (23.34%) and K₂O (2.89%). The ratio of Si/Al (i.e. SiO₂:Al₂O₃) of WR (bottom ash: dredged soil), S, R, and B were (2.45 : 4.92), 2.50, 3.03, and 2.55, respectively. According to the physical characteristics in Table 2, the pore diameter of all tested adsorbents was ranged from 1.41 to 2.03 nm. Moreover, WR had a large surface area and total pore volume while R and B had a small surface area and total pore volume. However, the porosity in R and B were higher than others and this was caused by using sawdust or surfactant. These physical characteristics implied that WR had relatively smaller pores than R or B in both external and internal surfaces while R and B had relatively large pores in surfaces. In addition, S showed the lowest specific gravity, 0.75, which indicated that S was lighter than water.

XRD analysis (Fig. 1) indicated that quartz was the most common mineral for all nano-pore adsorbents, and several types of aluminum-silica complexes (e.g. anorthite (CaAl₂Si₂O₈), albite (NaAlSi₃O₈), sillimanite (Al₂SiO₅), or mullite (3Al₂O₃ • 2SiO₂)) were also detected. In addition to these minerals, WR had two types of iron minerals, which were hematite (α -Fe₂O₃)

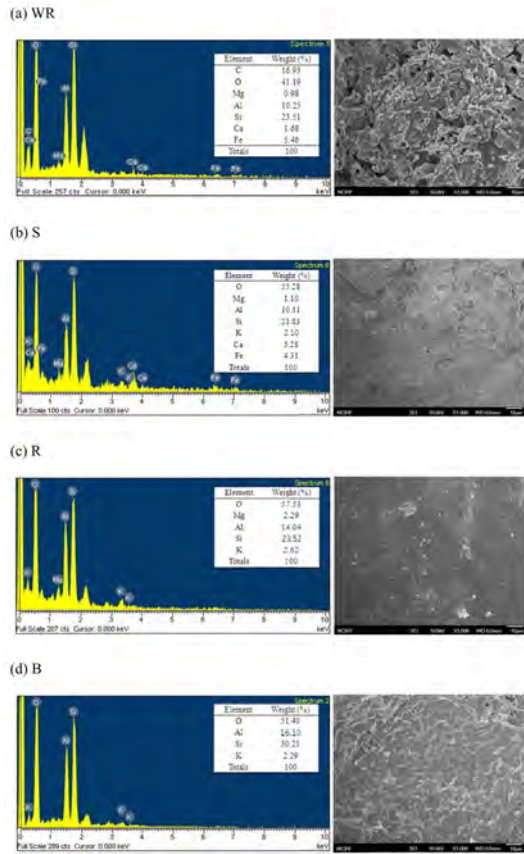


Fig. 2. The results of SEM-EDS analysis for nano-pore ceramic substrates.

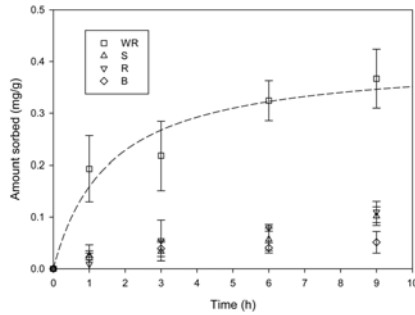


Fig. 3. The average BTEX uptakes on nano-pore ceramic substrates (Initial conc. of contaminants = 100 mg/L, the amounts of substrate = 2.5 g, and initial pH = 7.0). The line in figure was a guideline for experimental data.

and maghemite ($\gamma\text{-Fe}_2\text{O}_3$), and S had an iron-aluminum complex, hercynite (FeAl_2O_4). SEM-EDS analysis (Fig. 2) confirmed that iron (Fe) was detected only on the surface of WR and S, and it revealed that WR had the highest weight percentages of Fe (i.e. 5.46 wt %). Moreover, WR had the roughest surface among adsorbents.

Adsorption isotherms

Three isotherm models (i.e. linear sorption, Langmuir, and Freundlich, as shown in Eq. (1), (2), and (3), respectively) were used for B, T, E, and X

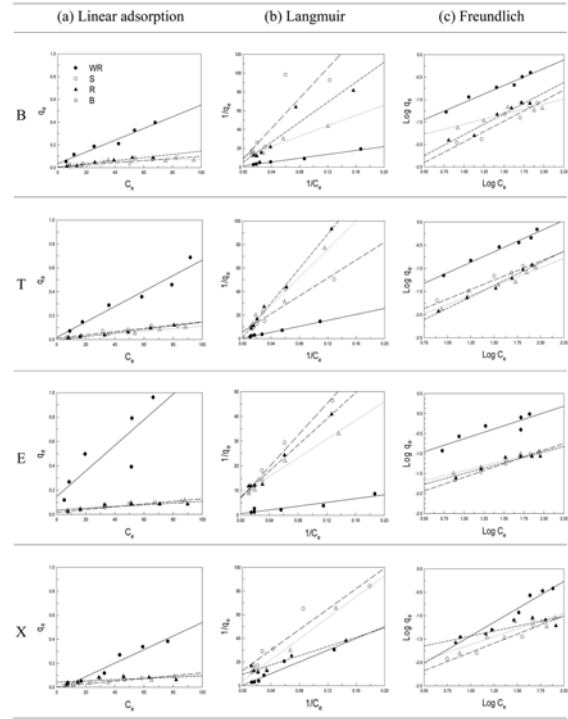


Fig. S1. Linear sorption, Langmuir, and Freundlich isotherms.

adsorptions on nano-pore adsorbents (i.e. WR, S, R, and B).

$$q_e = K_L C_e \quad (1)$$

$$q_e = Q_0 \frac{b C_e}{1 + b C_e} \quad (2)$$

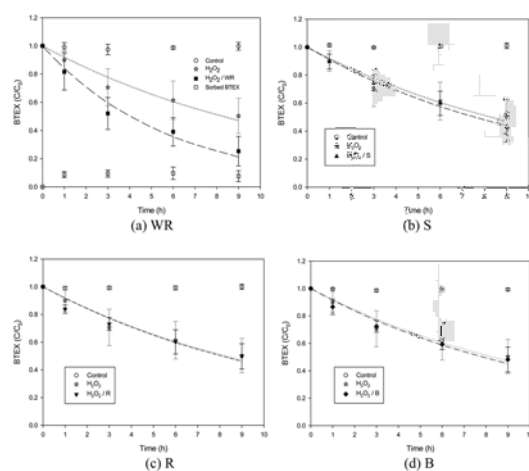
$$q_e = K_F C_e^{1/n} \quad (3)$$

where q_e is the amount of contaminant adsorbed per solid (mg g^{-1}) at equilibrium, K_L is the linear adsorption equilibrium constant (L g^{-1}), and C_e is the equilibrium contaminant concentration (mg L^{-1}); Q_0 and b represent the adsorption capacity at complete surface coverage (mg g^{-1}) and affinity constant (L mg^{-1}), respectively; K_F and n represent the capacity of solids to adsorb contaminant ($(\text{mg g}^{-1})(\text{L mg}^{-1})^{1/n}$) and the intensity of adsorption (dimensionless), respectively. The linearized forms of each model were used to calculate coefficients, and those values are shown in Table 3 (Also see Fig. S1. in Supplementary data).

According to results in Table 3, Langmuir model predicted BTEX adsorptions on WR, R and B with high reliability (r^2) while both linear and Freundlich model did the same for S. Based on Langmuir model, the order of adsorbents for BTEX adsorption was $\text{WR} > \text{R} > \text{S} > \text{B}$. This relatively high adsorption capacity of WR could be explained by the large surface area. The calculated adsorption capacities (Q_0) for BTEX were 0.752, 3.793, 1.678, and 4.902 mg g^{-1} , respectively, which indicated that the order of adsorption on WR was $\text{X} > \text{T} > \text{E} > \text{B}$. Even though the orders for the tested adsorbents (i.e. WR, S, R and B) were slightly

Table 3. Coefficients of linear sorption, Langmuir, and Freundlich models.

targets		Linear sorption		Langmuir			Freundlich		
		K_L (L g ⁻¹)	r^2	Q_0 (mg g ⁻¹)	b (L mg ⁻¹)	r^2	K_F (mg g ⁻¹)(L mg ⁻¹) ^{1/n}	$1/n$	r^2
WR	B	0.005	0.961	0.752	0.013	0.982	0.015	1.317	0.966
	T	0.006	0.944	3.793	0.002	0.994	0.010	1.105	0.979
	E	0.011	0.725	1.678	0.016	0.894	0.052	1.535	0.798
	X	0.006	0.943	4.902	0.001	0.933	0.003	0.858	0.941
S	B	0.001	0.917	0.111	0.011	0.732	0.001	1.084	0.910
	T	0.001	0.933	0.169	0.016	0.872	0.003	1.219	0.939
	E	0.001	0.963	0.146	0.021	0.985	0.005	1.470	0.992
	X	0.001	0.960	0.078	0.030	0.912	0.003	1.284	0.952
R	B	0.001	0.940	0.188	0.010	0.902	0.002	1.074	0.948
	T	0.001	0.978	1.279	0.001	0.995	0.001	1.015	0.984
	E	0.001	0.678	0.137	0.028	0.983	0.009	1.860	0.891
	X	0.001	0.320	0.104	0.049	0.915	0.014	2.361	0.692
B	B	4.33E-4	0.422	0.090	0.041	0.921	0.010	2.225	0.751
	T	0.001	0.955	0.242	0.007	0.966	0.003	1.257	0.949
	E	0.001	0.874	0.124	0.043	0.972	0.012	1.980	0.956
	X	0.001	0.591	0.248	0.009	0.952	0.005	1.502	0.811

**Fig. S2.** BTEX removal by H₂O₂ with/without (a) WR, (b) S, (c) R, and (d) B. The sorbed BTEX on S, R, and B were less than 1%, so they were not presented in figures, and the prediction lines of the first-order kinetics were used for guide.

different from each other, it is generally accepted that the orders of BTEX adsorption were depended on materials [7, 8, 14].

BTEX removal by nano-pore adsorbents with hydrogen peroxide

BTEX removal on nano-pore adsorbents was evaluated, and controls indicated the negligible loss of BTEX. The average BTEX uptakes by WR, S, R, and B were 0.37, 0.10, 0.11, and 0.05 mg g⁻¹, respectively, when it was conducted for 9 hrs reaction time. This indicated that waste-reclaimed adsorbent (WR) could

Table 4. Removal percentages, residual H₂O₂, and the final pH of BTEX degradation with H₂O₂ and nano-pore ceramic substrates.

[H ₂ O ₂]	Types of substrates	Removal percentages, % (Sorbed ^a , %s)				Residual ^b (%)	pH ^c
		B	T	E	X		
500 mM	None	43	38	51	67	97	4.3
	WR	66 (13)	65 (14)	83 (8.4)	85 (6.6)	83	4.6
	S	50	47	62	70	97	6.3
	R	42	44	55	61	96	5.1
	B	49	40	58	59	96	5.7

^a: the highest values of sorbed BTEX within 9 hrs reaction [The values for S, R, and B were not presented because the sorbed BTEX on them were less than 1.0%].

^b: the average value of residual H₂O₂ in BTEX degradation experiments.

^c: the average value of final pH in BTEX degradation experiments.

be a possible adsorbent for low BTEX contamination.

Along with adsorbability of nano-pore adsorbents, BTEX removals in the systems containing H₂O₂ with/without adsorbents were evaluated. Total BTEX removals were shown in Supplemental data (Fig. S2). Parameters (i.e. removal percentages, the percentages of residual H₂O₂ concentration, and final pH) were presented in Table 4.

As shown in Table 4, H₂O₂ decomposition in the system with WR was 17% while those in other systems ranged from 3 to 4%. In accordance with that, total BTEX removal by H₂O₂/WR was higher than that by others. Moreover, even though less than approximately

6.6 to 14% of BTEX was still adsorbed, the catalytic characteristic of WR was observed. Due to the existence of both hematite ($\alpha\text{-Fe}_2\text{O}_3$) and maghemite ($\gamma\text{-Fe}_2\text{O}_3$) on WR, $\text{H}_2\text{O}_2/\text{WR}$ system could be considered as a Fenton-like system (i.e. reaction between H_2O_2 and iron minerals). It was reported that the mixture of hematite and maghemite could degrade bisphenol A (BPA) more favorably than hematite or maghemite alone [28]. Moreover, Hanna et al. [29] reported that the catalytic activities of ferric oxides (i.e. goethite and maghemite) were higher than ferrous oxide (i.e. magnetite) when they were existed with quartz. None or less catalytic property of S, which had hercynite (FeAl_2O_4) could be explained by the results of Hanna et al. [29]. In addition, because of the lower pH after 9 h reaction time in $\text{H}_2\text{O}_2/\text{WR}$, the effect of the dissolved Fe was checked. The dissolved total Fe in the $\text{H}_2\text{O}_2/\text{WR}$ system was 0.05 mg L^{-1} , which was analyzed by ICP-AES (OPTIMA 4300DV). This less amounts of the dissolved Fe (i.e. 0.05 mg L^{-1}) from WR could not affect BTEX degradation as catalyst. It was reported that hydroxyl radical ($\text{OH}\cdot$) rapidly reacted with H_2O_2 to produce perhydroxyl radical ($\text{HO}_2\cdot$) at $[\text{Fe(II)}]/\text{H}_2\text{O}_2 \ll 1.0$, and perhydroxyl radical was known for a less significant reductant [30, 31]. Therefore, the catalytic activity of WR could be due to a mixture of iron oxides.

Conclusions

Four types of nano-pore adsorbents, which were waste-reclaimed (WR), shale-based (S), red clay-based (R), and blue kaolin-based (B) were tested for their adsorption ability and catalytic characteristics. WR showed the high adsorption capacity because of its relatively large surface area, and the higher BTEX degradation by $\text{H}_2\text{O}_2/\text{WR}$ was observed due to iron oxides, which were hematite ($\alpha\text{-Fe}_2\text{O}_3$) and maghemite ($\gamma\text{-Fe}_2\text{O}_3$). Moreover, even though S had hercynite (FeAl_2O_4), it did not show catalytic property. Therefore, these adsorptive and catalytic characteristics of WR could be valuable in terms of both the reduction of waste materials and the remediation for low concentration of BTEX.

Acknowledgment

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Appendix A. Supplementary data

Supplementary data associated with this article can be found in the online version.

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