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Catalytic activities of zeolite compounds for decomposing aqueous ozone

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Abstract

The advanced oxidation process (AOP), chemical oxidation using aqueous ozone in the presence of appropriate catalysts to generate highly reactive oxygen species, offers an attractive option for removing poorly biodegradable pollutants. Using the commercial zeolite powders with various Si/Al ratios and crystal structures, their catalytic activities for decomposing aqueous ozone were evaluated by continuously flowing ozone to water containing the zeolite powders. The hydrophilic zeolites (low Si/Al ratio) with alkali cations in the crystal structures were found to possess high catalytic activity for decomposing aqueous ozone. The hydrophobic zeolite compounds (high Si/Al ratio) were found to absorb ozone very well, but to have no catalytic activity for decomposing aqueous ozone. Their catalytic activities were also evaluated by using the fixed bed column method. When alkali cations were removed by acid rinsing or substituted by alkali-earth cations, the catalytic activities was significantly deteriorated. These results suggest that the metal cations on the crystal surface of the hydrophilic zeolite would play a key role for catalytic activity for decomposing aqueous ozone.

Key words: ozone; advanced oxidation process; catalyst; hydroxyl radical; zeolite

Introduction

Ozonation and advanced oxidation processes (AOPs) have recently emerged as an important class of technologies for the oxidation and destruction of a wide range of organic pollutants in water and wastewater (Legrini et al., 1993; Alvares et al., 2001; Zhou and Smith, 2001; Oppenländer, 2003). The AOPs are characterized by a variety of radical reactions that involve combinations of chemical agents (e.g., O₃ and/or H₂O₂) and auxiliary energy sources (e.g., ultraviolet-visible (UV-Vis) irradiation, electronic current, γ -radiation, and ultrasound). These treatment processes can either eliminate organic pollutants completely through mineralization or convert them to the products that are less harmful for human health and the aquatic environment. AOPs are processes involving *in-situ* generation of highly reactive species such as the hydroxyl radical (HO[•]), which is the primary oxidant in AOPs, while the other radical and active oxygen species are superoxide radical anions (O₂^{•-}), hydroperoxyl radicals (HO₂^{•-}), singlet oxygen (¹O₂) and organic peroxy radicals (ROO[•]). Unlike many other radicals, HO is non-selective and thus readily attacks a large group of organic chemicals to convert them to less harmful intermediate products.

Alternative ozonation processes, i.e., catalytic ozonation, have been investigated for degradation of organics, which can be divided mainly into homogeneous and

heterogeneous processes. In homogeneous catalytic ozonation, transition metal ions were used together with bulk O₃ (Hordern et al., 2003). Heterogeneous catalytic ozonation involves the use of metal oxides such as TiO₂, Al₂O₃, SiO₂, MnO₂ and Fe₂O₃ and metals or noble metals such as Co, Cu, Pb, Ru, Pt and Pd supported on metal oxides (Liotta et al., 2009; Hordern et al., 2003). The activity of these catalysts was based on the catalytic decomposition of O₃ and the enhanced generation of HO. The efficiency of catalytic ozonation depended to a great extent on the catalysts and its surface properties as well as solution pH (Hordern et al., 2003). The role of metals on the surface of metal oxides in the process of heterogeneous catalytic ozonation in water was discussed by Legube and Karpel Vel Leitner (1999).

Recently, the degradation of organic compounds have been found to be enhanced by the simultaneous use of ozone and hydrophilic zeolites (Sano et al., 2007; Dong et al., 2008; Valdés et al., 2009a, 2009b) or hydrophobic zeolites (Reungoat et al., 2007; Chen et al., 2008; Merle et al., 2009; Amin et al., 2010; Reungoat et al., 2010). There have been agreement that catalysts enhance aqueous ozone decomposition by allowing the formation of HO. However, the mechanism of catalyzed ozone decomposition and the relationship between catalysts and their abilities to catalyze ozone decomposition have not been well understood. The purpose of this study is to evaluate the catalytic activities of various zeolites for decomposing aqueous ozone, and to clarify the mechanism of catalyzed

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aqueous ozone decomposition by the specific zeolites.

1 Materials and methods

1.1 Screening tests of various zeolites as catalysts for aqueous ozone decomposition

Table 1 shows the crystal structures, compositions and Si/Al ratios of various zeolite powders used in this study, which were purchased from TOSOH Corp., Japan. Since the average particle sizes of all powders were in the order of a few microns, the size classification was performed using a following procedure: 60 g of each powder was dispersed in 1 L purified water and settled for 5 min, then a resultant supernatant was discarded and the precipitate was dried at 80°C overnight.

Figure 1 shows an experimental set-up for evaluating the catalytic activities of various zeolite powders for decomposing aqueous ozone. O₃ was produced from dry pure oxygen using an ozone generator (OZG-05G, Shibata Chemical Co. Ltd., Japan). The concentration of O₃ (ca. 10,000 ppm) was checked using the O₃ analyzer (OZM-7000GN, Shibata Chemical Co. Ltd., Japan) during an experiment. O₃ gas was continuously bubbled into 1 L distilled water in a sealed glass container at 10°C for about 1 hr until saturation (ca. 11 ppm), then 30 g of zeolite powder was added to a container. By continuously bubbling O₃ gas and dispersing zeolite powder using a magnetic stirrer, the O₃ concentration of supernatant was determined using the iodine titration method after 1 min, 2 hr, 4 hr and 8 hr.

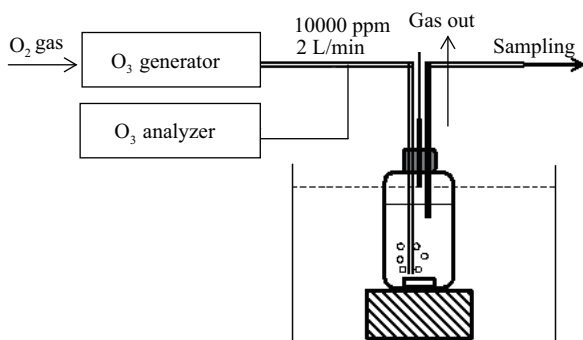


Fig. 1 Schematics of the experimental set-up for performing screening tests of various zeolites as catalysts for aqueous ozone decomposition.

1.2 Effects on ion-exchange

Three commercial A-type zeolite pellets in cylindrical shape with 1 mm diameter and 5 mm length, molecular sieve 3A ($K_{7.2}Na_{4.8}Al_{12}Si_{12}O_{48} \cdot xH_2O$), 4A ($Na_{12}Al_{12}Si_{12}O_{48} \cdot xH_2O$) and 5A ($Ca_{4.8}Na_{2.4}Al_{12}Si_{12}O_{48} \cdot xH_2O$), were purchased from Kishida Chemical Co. Ltd., Japan. The 60 g zeolite pellets were dispersed in 1 L distilled water or 1 mol/L HCl for 1 hr. After discarding the supernatant, the pellets were washed by distilled water once, and dried at 80°C overnight.

The 30 g zeolite pellets were filled in a glass column (ca. 44 cm³). Distilled water saturated by O₃ (ca. 11 ppm) produced by the procedure described above was passed through a column with various flow rates as shown in **Fig. 2**. The O₃ concentration of effluent was determined using the iodine titration method.

2 Results and discussion

2.1 Screening tests of various zeolites as catalysts for aqueous ozone decomposition

Figure 3 shows the change of O₃ concentration with time after dispersing zeolite powders. Clearly, zeolites are

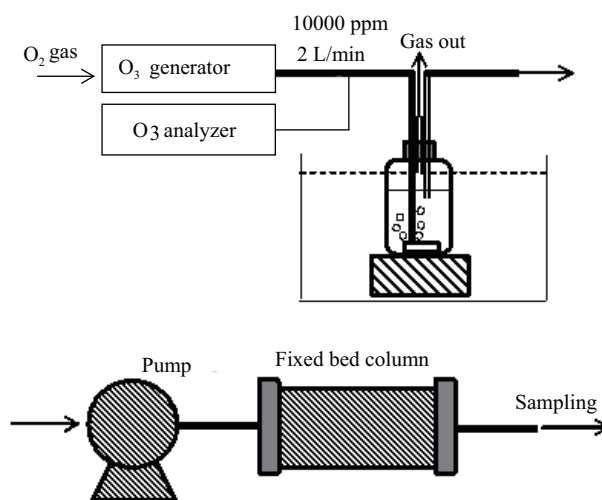


Fig. 2 Schematics of the experimental set-up for evaluating the catalytic activities of zeolite using the fixed bed column method.

Table 1 Crystal structures, compositions and Si/Al ratios of various zeolite powders

Type	Composition	Product name	Si/Al ratio	Na ₂ O (wt.%)
A type	Na ₁₂ Al ₁₂ Si ₁₂ O ₄₈ ·27H ₂ O	A-4	1	17
X type (Faujasite)	Na ₈₆ Al ₈₆ Si ₁₀₆ O ₃₈₄ ·240H ₂ O	F-9	1.23	15
Y type (Faujasite)	Na _n Al _n Si _{192-n} O ₃₈₄ ·xH ₂ O n: 48–76	HSZ-320NAA	5.5	12.5
		HSZ-320HOA	5.5	4
		HSZ-350HUA	10	0.1
Mordenite	Na ₈ Al ₈ Si ₄₀ O ₉₆ ·24H ₂ O	HSZ-642NAA	18	5
		HSZ-640HOA	18	0.05
Ferrierite	not shown	HSZ-720KOA	18	1.3

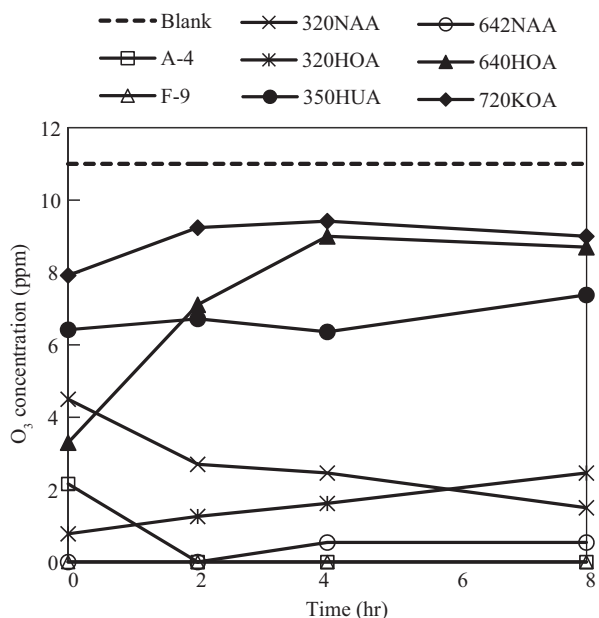


Fig. 3 Change of O_3 concentration with time after dispersing various zeolite powders.

divided into two groups. When a group including A-4, F-9, HSZ-320NAA, HSZ-320HOA and HSZ-642NAA (group 1) was dispersed in distilled water, O_3 concentration was maintained at relatively low level throughout the experiment. This suggests that these zeolites have high catalytic activity for decomposing aqueous ozone. In contrast, when another group including HSZ-350HUA, HSZ-640HOA and HSZ-720KOA (group 2) was dispersed in distilled water, O_3 concentration was maintained at a level close to the saturation (ca. 11 ppm). This suggests that these zeolites possess almost no catalytic activity for decomposing aqueous ozone. Zeolites are basically hydrophilic, but they turn hydrophobic when high Si/Al ratio reaches 8 or 10 (Ruthven, 1984). Zeolites with high Si/Al ratio are called high silica zeolites, and are known to have good adsorption properties for non-polar molecules. As seen from **Table 1**, the group 1 and 2 are hydrophilic (low Si/Al ratio and high Na_2O content (≥ 4 wt.%) and hydrophobic (high Si/Al ratio and low Na_2O content (≤ 1.3 wt.%) zeolites, respectively. Zeolites containing more than 10 wt.% Na_2O , A-4 and F-9, showed the highest catalytic activity among the hydrophilic zeolites, suggesting that the content of alkali cations irrespective of the crystal structure be critical for the catalytic activity. The reason that O_3 concentration decreased once after 1 min in the case of HSZ-640HOA would be due to the adsorption of O_3 on the surface of hydrophobic zeolite (Fujita et al., 2004), since O_3 is a non-polar molecule. The current results are consistent with the previous report that high silica zeolites can catalyze ozone decomposition into radical species in the gas phase (Monneyron et al., 2003) but apparently not

in water (Reungoat et al., 2007).

2.2 Effects of ion-exchange

Figure 4a, b and c show the O_3 concentration after passing through the fixed bed column filled with molecular sieve 3A, 4A and 5A, respectively, using various flow rates. These figures also demonstrate the catalytic activities of the hydrophilic zeolite treated by a distilled water or 1 mol/L HCl. As clearly seen from these figures, the substitution of Na^+ by H^+ (treated by 1 mol/L HCl) significantly deteriorates the catalytic activity for decomposing aqueous ozone, which are not consistent with the previous works that the treatment by acid solution (Valdés et al., 2009a)

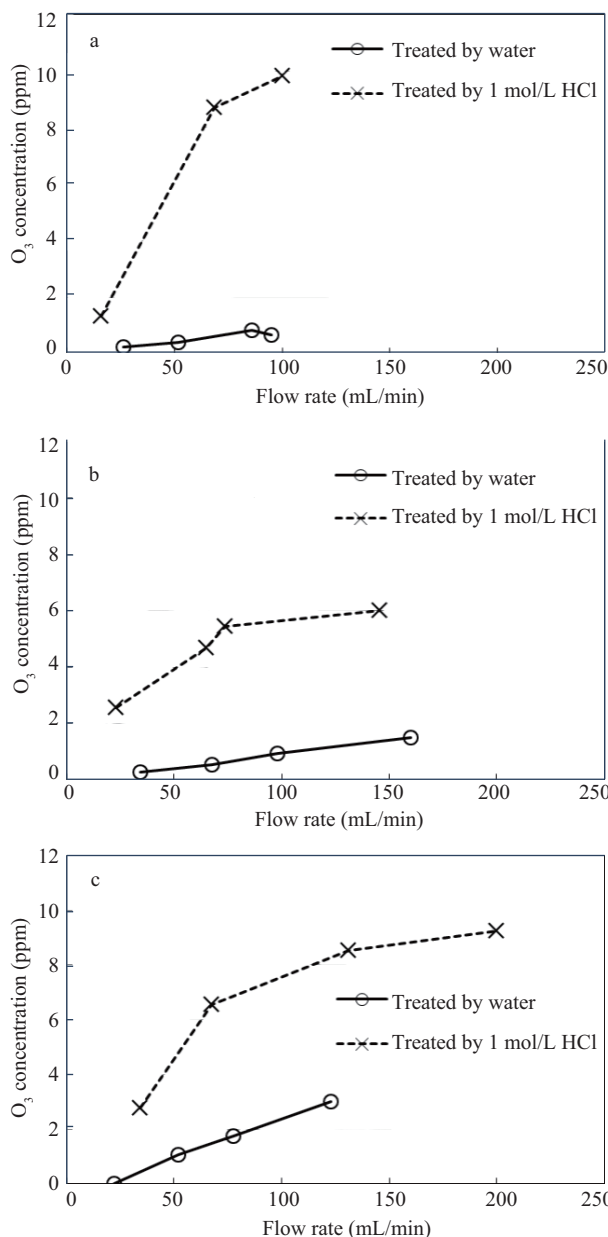


Fig. 4 O_3 concentration after passing through the fixed bed column filled with molecular sieve 3A (a), 4A (b) and 5A (c), respectively at various flow rates.

or water vapor (Dong et al., 2008) enhanced the catalytic activity due to the formation of the hydroxyl groups on the zeolite surface.

By comparing Fig. 4b with Fig. 4a and 4c, it would be concluded that the substitution of Na^+ contained in the molecular sieve 4A by K^+ (molecular sieve 3A) does not deteriorates the catalytic activity for decomposing aqueous ozone, but that by Ca^{2+} (molecular sieve 5A) does. It is well-known that the molecular sieve 3A or 5A is produced by the ion-exchange of Na^+ by K^+ or Ca^{2+} , respectively. Because all zeolites are the A-type (Si/Al ratio = 1) and are the most hydrophilic zeolites, the current results suggest that the catalytic activity for decomposing aqueous ozone depends not only on the Si/Al ratio but also on the metal cations in the zeolite crystal structure. The chemical formula of molecular sieve 5A ($\text{Ca}_{4.8}\text{Na}_{2.4}\text{Al}_{12}\text{Si}_{12}\text{O}_{48} \cdot x\text{H}_2\text{O}$) shows that Na^+ are partially substituted by Ca^{2+} . Then, it is speculated that the substituted Na^+ would be located on the micropore surface, and the remained Na^+ would be located inside the framework formed by SiO_4 and AlO_4^- tetrahedrons. It was reported that Na^+ protrude from the micropore surface of the A-type zeolite, and reduce the micropore size from 11 to 3.5 Å, while Ca^{2+} less protrude from the micropore surface and thus the micropore size is 4.2 Å (Gramlich and Meier, 1971). Thus, the position of metal cations relative to the framework formed by SiO_4 and AlO_4^- tetrahedrons connected by oxygen atoms would play a key role for the catalytic activity for decomposing aqueous ozone.

2.3 Proposed mechanism for ozone decomposition catalyzed by hydrophilic zeolites

From the results and discussion described above, a following mechanism would be proposed for explaining the catalytic activity for decomposing aqueous ozone on the surface of the hydrophilic zeolites containing alkali cations (Fig. 5). (1) A-type zeolites (Si/Al ratio = 1) are composed of the frameworks formed by the SiO_4 and AlO_4^- tetrahedrons connected by oxygen atoms. To charge-compensate the AlO_4^- tetrahedrons, metal cations should exist close to them. (2) The SiO_4 tetrahedrons have no polarization to be hydrophobic, while the AlO_4^- tetrahedrons strongly polarize due to the existence of metal cations to be hydrophilic. (3) O_3 molecules would readily adsorb on the hydrophobic SiO_4 tetrahedrons, since O_3 is a non-polar molecule. In the A-type zeolite crystal, the AlO_4^- tetrahedrons always exist beside the SiO_4 tetrahedrons, and adsorbed O_3 molecules would be polarized due to the electric field caused by the AlO_4^- tetrahedron and metal cation pair. (4) Polarized O_3 molecules would readily react with H_2O molecules to yield hydroxyl radicals according to the reaction:

The length between a AlO_4^- tetrahedron and Ca^{2+} is shorter than that between a AlO_4^- tetrahedron and Na^+ . Then, electric field caused by a AlO_4^- tetrahedron and

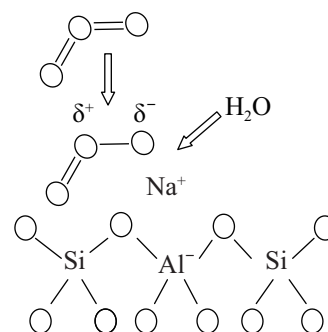


Fig. 5 Schematics explaining the catalytic activity for decomposing aqueous ozone on the surface of the hydrophilic zeolite containing alkali ion.

Ca^{2+} pair would be less than that caused by a AlO_4^- tetrahedron and Na^+ pair due to the difference of the cationic field strength. This is consistent with the results obtained in this study. The enhanced decomposition of 2,4-D (2,4-dichloro phenoxy acetic acid) by the AOP using hydrophilic zeolites as the heterogeneous catalysts has been confirmed (Ohta and Kitayama, 2011). The ESR (Electron spin resonance) studies for detecting and identifying the reactive oxygen species produced by the catalytic decomposition of aqueous ozone are on-going.

3 Conclusions

Using the commercial zeolite powders with various Si/Al ratios, metal cation contents and crystal structures, their catalytic activities for decomposing aqueous ozone were evaluated. Following conclusions are obtained in this study.

- (1) The hydrophilic zeolites (low Si/Al ratio) with high alkali cation contents in the crystal structures possess high catalytic activity for decomposing aqueous ozone.
- (2) The hydrophobic zeolites (high Si/Al ratio) have an ability to absorb ozone in water, but have almost no catalytic activity for decomposing aqueous ozone.
- (3) Metal cations on the crystal surface of the hydrophilic zeolite would play a key role for catalytic activity for decomposing aqueous ozone.

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