

Adsorption and Oxygen Plasma-Driven Catalysis for Total Oxidation of VOCs

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Abstract— Various catalysts have been evaluated for the optimization of cyclized system. The potential of the tested catalysts were evaluated in terms of adsorption capacity of VOC and enhancement factor (EF). Silver supported zeolites (Ag/MOR, Ag/HY and Ag/MS-13X) were found to be effective catalysts by their large EF values of about 100 and the large adsorption capacity as well. Optical microscope-image intensifier-CCD camera was applied to observe the generation of discharge plasma on the surface of zeolites. The supported Ag nanoparticles promoted the generation of discharge plasma over the wide range of the zeolite surface. The spread of the discharge plasma on the surface of zeolites was found to be highly correlated to the EF and the CO₂ selectivity as well. This new finding provides us very important insight into the better understanding of the low temperature activation of the catalysts by nonthermal discharge plasma.

Keywords— VOC, oxygen plasma, catalyst, zeolite, enhancement factor

I. INTRODUCTION

Emission of volatile organic compounds (VOCs) and their related environmental problems are ubiquitous in many industrial working places, indoor air, and even open air. Among various characteristics of VOCs, such as large vapor pressure and direct health impact (carcinogenic, teratogenic, and mutagenic), their contributions to the formation of photochemical smog and the secondary organic aerosol (SOA) are considered to be important environmental issues in air pollution, especially in urban areas. Since the LA smog episode in the early 1950s, VOCs has been attracted concerns from scientists, governmental department of environment, and publics as well. Emission standards and regulations related to VOCs and HAPs (hazardous air pollutants) are getting stringent in many countries over the world. Generally, the approaches toward the pollution control can be divided into two major groups of source reduction and end-of-pipe (EOP) treatment. The source reduction includes the modification of process, changing chemicals to safer ones, and recycling before they are released to the open air. Basically this idea is quite close to the green chemistry. Although the source reduction approach seems to be environmental friendly and expected to be main stream in the future, significant time and investments will be required to meet the stringent upcoming regulations by the source reduction alone. From a practical viewpoint, the EOP treatment is more reasonable way to meet the environmental goals at present stage. There is therefore an urgent need to develop more effective and inexpensive techniques for the treatment of VOCs.

The nonthermal plasmas (NTPs) have been investigated for the removal of various air pollutants for many years [1-3]. Many laboratory scale experiments in 1990s have demonstrated the possible use of NTPs for the decomposition of VOCs. Except for the saturated hydrocarbons, the decomposition efficiencies higher than 90% have been achieved for the most of VOCs.

One of the remarkable recent trends in the NTP application for air pollution control is the combination of NTP with catalyst in either configuration of single-stage [4-8] or two-stage [9, 10]. The major reasons in using hybrid plasma system are the reduction of energy consumption (i.e. high energy efficiency), less NO_x formation and acceptable material balance. Some of the two-stage process operates the catalyst bed at higher temperature up to 400-500 °C [11-14]. The authors have been investigated the decomposition of VOCs using single-stage plasma-driven catalysis (PDC) system, and found the strong dependence of the PDC system to the oxygen partial pressure in the treating gas mixtures. Based on this finding, the authors have proposed the cyclized system, which use adsorption of VOCs and the oxygen plasma decomposition of the adsorbed VOC in a cycling mode.

In the present work, the potential of zeolites for the cyclized system has been tested. Two important parameters used for the evaluation are adsorption capacity of benzene and the enhancement factor. Optical microscopy-image intensifier-CCD camera system was used for the direct observation of discharge plasma on the surface of catalysts. Especially we focused on the interaction between the supported metal catalyst and the discharge plasma over the zeolites. The correlation between the catalytic activity and the physical characteristics in plasma generation on the surface of catalyst will be also discussed.

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II. EXPERIMENTAL

A. Setup and Measurement

Since the main objective of this work is to evaluate various catalysts for optimization of the cycled system, all the experiments were done with the flow-through type PDC reactor. Figure 1 shows the details of the PDC reactor. The PDC reactor was basically similar to a cylindrical surface discharge reactor. A coil-type stainless steel electrode with 35 turns and 0.45 mm diameter was set inside the tube as a high-voltage electrode, and silver paste was painted on the outer surface of the quartz tube as a ground electrode. In this surface discharge-like configuration, strong electrical field to form electrical discharge plasma is established between the ground electrode and the coil electrode on the inner surface of the quartz tube, so the plasma generation will be less sensitive to dielectric constants of the packing materials (i.e. catalysts in this case) compared to configuration of the dielectric barrier discharge (DBD). However, plasma can be spread over the surface of catalyst, and these effect will be discussed later. The inner diameter and the effective length of the quartz tube were 11mm and 150 mm, unless otherwise noted. Catalysts in the PDC reactor were placed within the effective length of the reactor. This configuration rendered all the catalysts exposed to the plasma and also allowed to ignore additional adsorption or additional chemical reactions outside the plasma region. The used catalysts were in the forms of pellets or spherical beads with size range from 1.6 mm to 3.5 mm. The tested catalyst materials include TiO_2 , $\gamma\text{-Al}_2\text{O}_3$, and zeolites such as ferrierite (FER; Tosoh Co.), mordenite (MOR; Tosoh Co.), HY (Tosoh Co., GL-Science), MS-13X (Sigma-Alcrich Co.). The specific surface areas (i.e. BET area) of the zeolites were in the range of 270–540 m^2/g . Silver supported catalysts were prepared by impregnation method with AgNO_3 as a starting material. The size and the shape of the silver catalyst were measured by transmission electron microscopy (TEM, Topcon Co., Model EM002B). Details of the catalysts are reported elsewhere [6].

The oxygen partial pressure-dependent behaviors of various catalysts for the decomposition of benzene were tested with the flow-through PDC reactor at 100 °C. Benzene was used as a model compound of VOC. The concentration of benzene was adjusted by changing either the temperature of water bath or the N_2 flow rate purging the bubbler. The PDC reactor was energized by an AC power supply consisting of a high voltage amplifier (Tek 20/20B) and a function generator (Tektronix AFG310). A Fourier transform infrared (FTIR) spectrometer (Perkin Elmer, Spectrum One) equipped with a long optical path length (6.4 mm) was used for the gas measurements of both the reactants and products.

Discharge power of the PDC reactor was measured using the automated V-Q Lissajous program (Insight Co. Ver. 1.72). A condenser (about 100 nF) was connected in series to the ground line of the plasma reactor. The

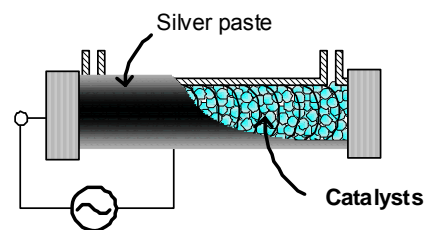


Fig. 1. Schematic diagram of the PDC reactor.

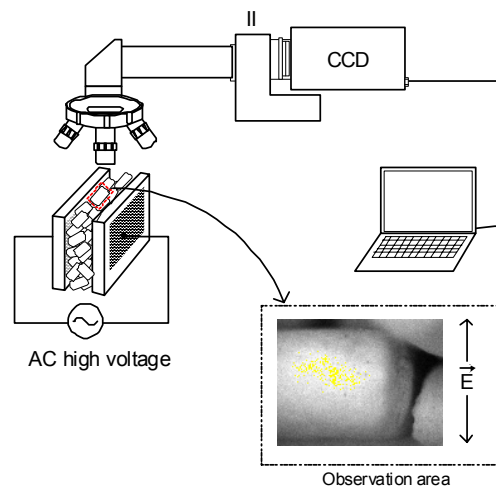


Fig. 2. Observation system of the discharge plasma on the surface of catalyst. (II; image intensifier)

charge Q (i.e. time-integrated current) was measured with a 10:1 voltage probe (Tek P6139A). Specific input energy, which is the ratio of discharge power (P_{dis} in watt) to the gas flow rate (Q_f in L/min), is one of important scaling parameters of NTP processing.

$$\text{SIE} = \frac{P_{\text{dis}}}{Q_f} = \frac{\text{discharge power (W)}}{\text{gas flow rate (L/min)}} \times 60 \quad (1)$$

The unit of Wh/Nm^3 is usually used for a large-scale experiment. The conversion factor from Wh/Nm^3 to J/L is 3.6 at normal temperature and atmospheric pressure. There is no general consensus on the terminology of the P/Q_f , so a number of different names are found in the literature; (specific energy input, specific deposited energy, specific power input, specific discharge input, adsorbed discharge energy, energy deposition, specific energy density, energy input density, corona energy density, discharge energy density, input energy density etc.). The term of P/Q_f is basically identical to the specific corona power, which was used in the electrostatic precipitator (ESP) [15, 16]. Both the specific input energy and the specific corona power are useful parameters, especially for scaling-up of the system, because they provide intuitive insights on total energy consumption for given flow rates. In the case of the cycled operation equivalent specific input energy (SIE_{eq})

was introduced for the evaluation of the energy consumption.

$$\text{SIE}_{\text{eq}} \text{ (J/L)} = \frac{(P_{\text{dis}})_{\text{Ave}}}{(Q_{\text{Ads}})_f} \times \frac{T_{\text{Oxy}}}{T_{\text{Ads}}} \times 60 \quad (2)$$

Here, $(P_{\text{dis}})_{\text{Ave}}$ and $(Q_{\text{Ads}})_f$ indicate the average discharge power of oxygen plasma during the decomposition of adsorbed VOC and gas flow rate during the adsorption, respectively. T_{Oxy} and T_{Ads} indicate the period of oxygen plasma and adsorption, respectively. Since all the experiments in this works have been conducted in a flow-through reactor, specific energy indicates SIE unless otherwise noted.

Figure 2 illustrates the setup for the microscopic observation of the discharge plasma on the surface of catalyst. Optical observation system consisted of an XY stage, optical microscope, image intensifier (Hamamatsu Photonics, C9016-03) and a CCD camera (Hamamatsu Photonics, C8484-05G). A small plane type DBD reactor of 4 mm gap was set on the XY stage. This DBD reactor was energized with a neon transformer, capable of delivering up to 40 kV_{pk-pk} at commercial frequency of 50 Hz. Four lenses with different magnification were attached to the revolver. The observation area can be adjusted by changing the optical lens. For example, the observation area of the 5X lens was 2.0 mm×2.6 mm, which corresponds to the size of single pellet. In all cases, electrodes were located at the top and the bottom of the observation area. The measured digital images were recorded on a PC.

B. Cycled System (Adsorption + Oxygen Plasma)

The cycled process is based on the highly oxygen content-dependent behavior of the PDC system for the decomposition of benzene [17]. Figure 3 shows the schematic diagram of cycled system consisted of two different processes. In the adsorption mode, VOCs are removed from the main exhaust gas stream by adsorption at room temperature without plasma application. When the catalyst bed reached adsorption equilibrium, then the gas flow was switched to the other catalyst bed to sustain the VOC removal by adsorption. Then the saturated catalyst bed is purged with oxygen before applying oxygen plasma to decompose the adsorbed VOCs. The two different processes in the cycled system require different properties of catalyst. In the case of adsorption mode, a large adsorption capacity is necessary. On the other hand, the regeneration mode requires high catalytic activity under oxygen plasma for the effective decomposition of adsorbed VOC. For the measurement purpose, the regeneration mode was operated with oxygen flow of 5–8 L/min. A small additional PDC reactor was set downstream of the main PDC reactor to decompose the desorbed VOC just after the O₂ plasma was turned on. In practical case, it will be beneficial to operate the regeneration mode in closed system after purging the catalyst bed with oxygen. In air plasma, there is a trade-off relation between the degree of VOCs

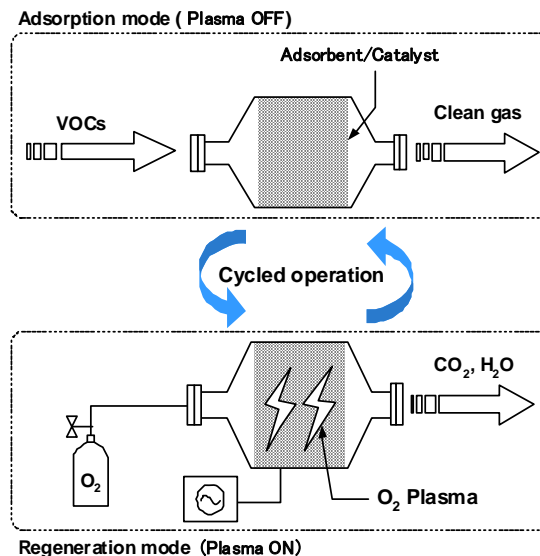


Fig. 3. Schematic diagram of the cycled system.

decomposition and the NO_x formation. So the determination of the maximum applicable specific input energy (SIE) is important in the flow-type PDC reactor. In the cycled system, however, formation of nitrogen oxides can be avoided because the plasma is only turned on under oxygen environment. This cycled operation itself is quite similar to that in the conventional adsorption process. The main difference between the cycled system and the conventional adsorption lies in the regeneration process. In the cycled system, the O₂ plasma decomposition of adsorbed VOC and the generation of adsorbent occur simultaneously. The major advantages of the cycled system can be summarized as follows.

- 1) Oxygen plasma exhibits higher energy efficiency than that of air plasma.
- 2) Free from nitrogen oxides formation
- 3) High CO₂ selectivity (100% for benzene decomposition using Ag/TiO₂)
- 4) Compact system due to the concentration of dilute VOC with a large volume to a limited space (on the surface of catalyst)
- 5) High power operation is possible (same reason in 2)
- 6) Flexible to the change of concentration and flow rate

III. RESULTS AND DISCUSSION

A. Catalyst Evaluation by Enhancement Factor

Two important factors of adsorption capacity and catalytic activity in oxygen plasma should be considered in selecting proper material for the cycled system. The adsorption capacity is mostly determined by the physicochemical properties of the materials such as specific surface area, size and shape of pores, acidity, and

the molar ratio of the consisting materials. On the other hand, little is known about the main reason on the high catalytic activity in oxygen plasma. In this work, enhancement factor was used to evaluate the potential of each catalyst for the cycled system. As is shown in Figure 4, the enhancement factor (EF) is defined as the ratio of decomposition efficiency of VOC with respect to the O_2 partial pressure at the same SIE.

$$EF = \frac{\eta_{Oxygen} - \eta_{Air}}{[O_2]_{Oxygen} - [O_2]_{Air}} \times 100 \quad (3)$$

Here, η_{Oxygen} and η_{Air} indicate the decomposition efficiency of VOCs in oxygen and in air, respectively. Since the cycled system is operated in pure oxygen, the larger the EF value, the higher the potential of catalyst for the cycled system. Evaluation of EF should be done at proper range of SIE according to the acceptable ranges of carbon balance and the degree of the destruction removal efficiency (DRE). Carbon balance becomes poor when the SIE is smaller than about 100 J/L. On the other hand, DRE becomes too high at SIE larger than about 200 J/L even at air-like mixture (i.e. 20% oxygen), so the balanced comparison becomes difficult. The same can be

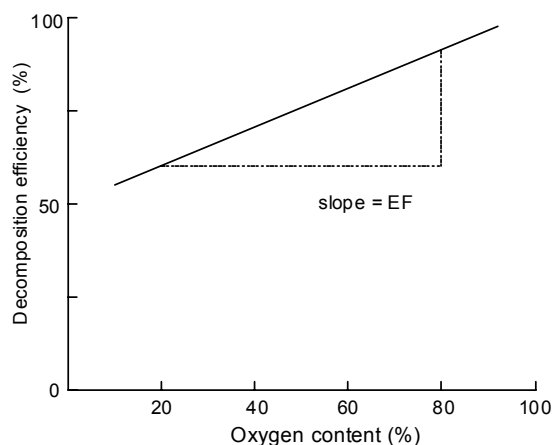


Fig. 4. Determination of enhancement factor.

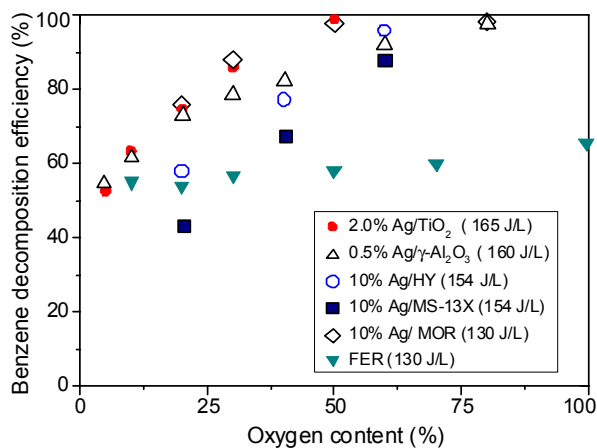


Fig. 5. Influence of oxygen on the decomposition of benzene ($[Benzene]_0 = 200$ ppm, $100^\circ C$)

applied for the oxygen content. In this sense, EF was evaluated with the SIE range of about 100–200 J/L and the oxygen content between 20%~80% in this study. The physical meaning of the enhancement factor is the slope of decomposition efficiency of VOC with respect to O_2 partial pressure at a given SIE.

It should be noted that this unique dependence on the O_2 partial pressure can be observed only with the PDC system. In the cases of the conventional thermal catalysis and DBD plasma alone were less dependent or independent of the oxygen partial pressure in the decomposition of benzene.

Figure 5 shows the effect of O_2 partial pressure on the decomposition of benzene according to the type of catalysts. It should be noted that the SIE shown in the figure indicates the values at 20% oxygen because the discharge power slightly changed with O_2 partial pressure even at a fixed voltage and frequency. For example, discharge power at 60% O_2 (9.72 W) was larger by about 0.6 W than that of the 20% O_2 (9.12 W) in the case of 10 wt% Ag/MS-13X at 30 kV_{pk-pk} and 500 Hz. As a general common trend, the decomposition efficiency of benzene linearly increased with oxygen partial pressures. However, the slopes depended on the types of catalysts. The Ag supported zeolites (MOR, MS-13X and HY) showed large enhancement in the benzene decomposition as the O_2 partial pressure increased. The oxygen content-dependent slopes of the Ag supported zeolites were quite similar to that of the Ag/TiO₂ catalyst.

B. Catalyst Optimization of Cycled System

Figure 6 summarizes the potential of the tested catalysts for the cycled system in terms of adsorption capacity and the enhancement factor. Recent experimental data are plotted together with in the previous data reported elsewhere [18]. Standard conditions for the evaluation are as follows; GHSV at 22000~60000 h⁻¹, 100 °C, 200 ppm benzene as model VOC and dry ($[H_2O]_0 < 100$ ppm) condition. As

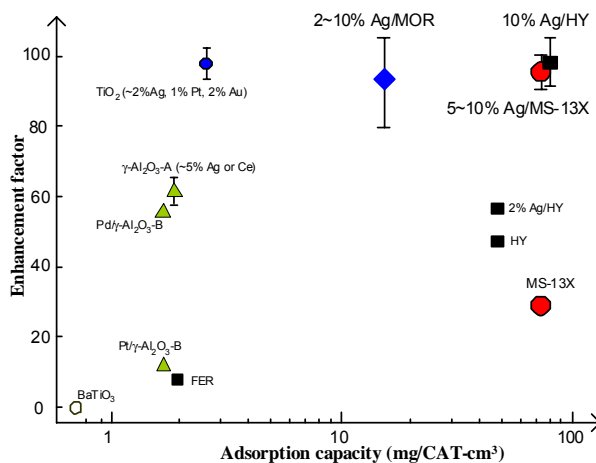


Fig. 6. Catalyst mapping for cycled system ($100^\circ C$, 200 ppm benzene)

TABLE I
CO₂ SELECTIVITIES OF THE TESTED ZEOLITES AT DIFFERENT OXYGEN CONTENT (BENZENE 200 PPM, 100 °C).

Catalyst	Supported Ag amount (wt%)	SIE (J/L)	S _{CO₂} in flow-through test (%)			S _{CO₂} in cycled system (%)
			20% O ₂	40% O ₂	60% O ₂	100 % O ₂
TiO ₂	0	165	65.5	69.2	71.0	–
	2	168	70.8	75.0	78.2	> 99%
MOR	2	128	61.7	62.0	64.1	–
	5	154	65.9	69.3	72.0	–
	10	120	75.0	80.3	83.6	–
HY	2	160	61.6	64.6	65.8	–
	10	154	68.9	71.6	72.9	–
MS-13X	5	158	71.1	76.2	76.4	–
	10	154	72.8	74.8	76.3	–

expected from the molecular size of benzene (5.9 Å), adsorption capacity was strongly related to the pore size of zeolite; MS-13X (10 Å) ≈ HY (7.4 Å) > MOR (6.7~7 Å) > ferrierite (4.3~5.5 Å). Interestingly all the tested materials exhibited positive enhancement factors as the O₂ partial pressure increased. In contrast, DBD plasma alone showed a negative EF values in the decomposition of benzene and toluene. Similar results have been reported with the different types of plasma alone process for the decomposition of NH₃ [19], dichloromethane [20], TCE and toluene [21]. Enhancement of benzene decomposition by increasing oxygen content has been first reported with Ag/TiO₂ catalyst [17]. The enhancement factor of the Ag/TiO₂ catalyst was about 100. In contrast to the large enhancement factor, the Ag/TiO₂ catalyst has small adsorption capacity of about 2 mg-C₆H₆/CAT-cm³, which make less attractive to be used in the cycled system. On the other hand, most of zeolites have relatively large specific surface area so that they can be served as good adsorbents. Moreover, characteristic pore sizes of the different zeolites can be attractive for the selective adsorption of certain molecule from their mixtures. The enhancement factors with the various zeolites were in the order of Ag supported zeolites (MOR, HY, MS-13X) >> 2 wt% Ag/HY > HY > MS-13X > FER. The enhancement factors with the Ag-supported MOR, HY and MS-13X were as good as or close to that of the Ag/TiO₂ catalyst. However, adsorption capacity of the Ag/MOR catalyst was about 16 mg-C₆H₆/CAT-m³, which is 8 times larger than that of the Ag/TiO₂ catalyst.

The Ag/MS-13X (75 mg-C₆H₆/CAT-m³) and the Ag/HY (80 mg-C₆H₆/CAT-m³.) exhibited about 40 times larger adsorption capacity compared to the Ag/TiO₂ catalyst.

Table 1 summarizes the selectivities of CO₂ for the flow-through tests of zeolite catalysts together with the 2 wt% Ag/TiO₂ as a reference catalyst. The CO₂ selectivity (S_{CO₂}) was determined as follows.

$$S_{\text{CO}_2} = \frac{[\text{CO}_2]}{[\text{CO}] + [\text{CO}_2]} \times 100 \quad (4)$$

As a common trend for all the tested catalysts, the CO₂ selectivity increased with the O₂ content in the gas stream. The CO₂ selectivity also increased as the supported amount of Ag increased. These observations are consistent with our previous works [17, 18]. The CO₂ selectivity of the Ag/TiO₂ catalyst was 78.2 % at 60% O₂ while > 99% in the case of the cycled system. Considering the similar CO₂ selectivities of the Ag supported zeolites (MOR, HY and MS-13X) to that of the Ag/TiO₂ catalyst, these zeolites will be also quite promising materials for the cycled system. The feasibility of the zeolite catalysts for the cycled system can be also confirmed by the large adsorption capacity and the high EF values. The potential of these zeolite catalysts will be further investigated by incorporating them into the cycled operation.

C. The Influence of Supported Silver Nanoparticles on the Plasma Generation on Zeolites

There have been many publications on the synergistic effect of the plasma-catalyst process. The important role of the supported metal catalysts has been explained by its catalytic activity, but their interaction with the discharge plasma is not well understood.

Figure 7 shows the influence of Ag nanoparticles on the plasma generation on the surface of two types of zeolites (MOR and MS-13X). The average size of the Ag

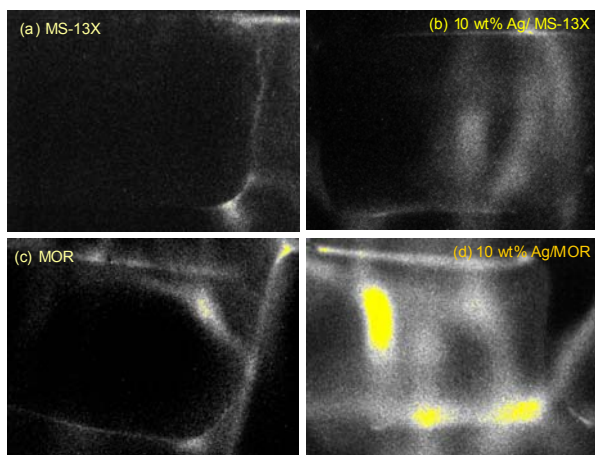


Fig. 7. Photos of discharge plasmas generated on the Zeolites; (a) MS-13X, (b) 10 wt% Ag/MS-13X, (c) MOR, (d) 10 wt% Ag/MOR (at all 33 kV_{pk-pk})

particles on both the zeolites was similar at about 3 nm~5 nm. AC high voltage of with 50 Hz was applied to the 4 mm gap DBD reactor filled with the catalysts. The electrodes were positioned at the top and the bottom in the figure. In the case of bare MS-13X (a) without Ag loading, plasma was mostly generated at the contact points, which is quite similar to the conventional ferroelectric (BaTiO₃) pellets packed bed reactor [22]. On the other hand, the discharge plasma easily spread on the surface of 10 wt% Ag supported MS-13X (b). The trend is more significant with the Ag/MOR catalysts (d). The similar influence of supported Ag on the plasma generation was also observed with the surface-discharge like configuration.

The remarkable influence of the Ag nanoparticles on the generation of discharge plasma can explain the different catalytic activities (see in Figure 6) of the bare and the Ag-supported zeolites. Both the Ag-supported zeolites exhibited much better catalytic activities, such as decomposition efficiency, CO₂ selectivity and carbon balance, compared to those of the bare zeolites. One generally accepted explanation of these enhancement is that the involvement of catalytic role by the supported metal catalyst (Ag in this case). However, the photos of discharge plasma clearly indicate that the supported Ag catalysts directly assist the generation of plasma over wide areas on the surface of catalyst. The enhanced plasma generation can interact with catalysts more effectively resulting in the enhancement of catalytic reaction. This new finding on the direct interaction of supported metal and the plasma generation provides an important insight into the smart design of the plasma-catalyst hybrid process.

IV. CONCLUSION

Various type of catalysts have been investigated in the flow-type PDC reactor to evaluate their potentials for

the cycled system. The two key factors used for the evaluation are the catalytic activity in oxygen plasma (i.e. enhancement factor) and the adsorption capacity. The catalytic activity of zeolite was found to be highly dependent on the presence of metal catalyst loading. The Ag supported zeolites (MOR, HY and MS-13X) exhibited similar enhancement factors to that of the Ag/TiO₂ catalyst, while the adsorption capabilities of the MOR and the MS-13X or NY were larger than the Ag/TiO₂ catalyst by 8 and 40 times, respectively. The influence of the metal catalyst loading was also evaluated by observing the shape of discharge plasma on the surface of catalyst. Discharge plasma expanded over wider areas on the Ag supported zeolites. The EF value and the CO₂ selectivity were also found to be highly correlated with the area of the plasma generation on the surface. This new finding provides us very important insight into the optimization of the plasma-driven catalyst process. The role of the supported metal catalyst on the performance of the plasma-driven catalyst should be explained not only by its catalytic activity but also by the influence on the generation of discharge plasma on the surface of catalysts.

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