Ultraselective and ultrasensitive detection of trimethylamine using MoO₃ nanoplates prepared by ultrasonic spray pyrolysis

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Abstract

An ultraselective and ultrasensitive trimethylamine (TMA) sensor was achieved using MoO₃ nanoplates-prepared by ultrasonic spray pyrolysis followed by a heat treatment at 450 °C. The small and thin MoO₃ nanoplates with gas-accessible structures showed an unusually high response to 5 ppm TMA (ratio of resistance to air and gas = 373.74) at 300 °C with detection limit as low as 45 ppb. Moreover, the ratios of the cross-responses to interfering gases (i.e., 5 ppm C₂H₅OH, CO, CH₄, C₂H₆, H₂, and NO₂) to the response to 5 ppm TMA were extremely low (0.008–0.016). The source of the ultraselective and highly sensitive detection of TMA with negligible interference from other gases is discussed with respect to the acid/base properties, size, and morphology of the MoO₃ sensing materials.

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1. Introduction

Trimethylamine (TMA) is secreted during the decomposition of trimethylamine N-oxide (TMAO) in dead fish and sea creatures [1,2], and induces skin burns, headaches, nausea, and irritation of the eye and respiratory system. The concentration of TMA increases with the decay of the dead fish and marine products. The fish is regarded as “decayed” when the concentration of TMA is greater than 10 ppm [3]. However, the TMA concentration is dependent upon the sensing conditions (e.g., distance from fish or marine products and number of decaying fish). Thus, the ability to sense TMA concentrations of less than 10 ppm is also necessary to estimate the degree of decay of fish or for highly sensitive instantaneous screening of decaying marine products. The National Institute for Occupational Safety and Health, USA, specifies permissible exposure limits of 10 ppm for 10 h for long-term exposure and 15 ppm for 15 min for short-term exposure [4]. Accordingly, detection of ppm levels of TMA is important to estimate fish freshness and identify harmful environments.

The chemiresistive variation of n-type oxide semiconductors such as SnO₂ [5], ZnO [6], TiO₂ [7], In₂O₃ [8], Fe₂O₃ [9], WO₃ [10], and MoO₃ [11] is used to detect trace concentrations of reducing gases. Simple sensor structure, high sensitivity, facile integration, and cost-efficiency are major strengths of oxide semiconductor gas sensors. However, the negatively charged oxygen adsorbed on the surface can react with a range of reducing gases, which often renders the discrimination of different gases difficult. To date, various oxide semiconductors have been explored to achieve selective and sensitive detection of TMA; these include ZnO doped with Al₂O₃, TiO₂, and V₂O₅ [12], WO₃ [13], Ru-doped SnO₂ [14], LaFeO₃ [15], ZnO–In₂O₃ composites [16], Cr₂O₃-decorated ZnO [17], and CdO–Fe₂O₃ [18]. Among these, WO₃ hollow spheres, which were prepared by the present authors, exhibited very high selectivity and responses to TMA [13]. Although, the selectivity of the WO₃ hollow spheres to TMA was among the highest of the reported oxide semiconductor gas sensors, the cross-responses to 5 ppm NH₃ (R₈/R₈ = 12.3, R₉: resistance in air, R₈: resistance in gas) and 5 ppm C₂H₅OH (R₈/R₈ = 4.8) were 21.6% and 8.4%, respectively, of that to 5 ppm TMA (R₈/R₈ = 56.9), which impedes the ultraselective detection of TMA.

Selective detection of a specific gas can be achieved by manipulating the sensing temperature [19], loading catalytic materials to promote the sensing reaction of a specific gas [20,21], compositional control of sensing materials with different acidities and basicities [22], and employment of a catalytic filtering layer [23]. The selective detection of TMA by WO₃ hollow spheres occurred via the strong interaction between basic TMA gas and acidic WO₃. MoO₃ is also an acidic oxide [24] and has the same orthorhombic crystal structures as WO₃. From this perspective, MoO₃ is potentially a good candidate material for the selective detection of TMA.
2. Experimental

Hexammonium heptamolybdate tetrahydrate (\((\text{NH}_4)_6\text{Mo}_7\text{O}_{24}4\text{H}_2\text{O}\), Junsei, 99%) (3.5310 g) and citric acid monohydrate (\(\text{C}_6\text{H}_8\text{O}_7\cdot\text{H}_2\text{O}\), Sigma-Aldrich, 99%) (2.1014 g) were dissolved in 1 L of distilled water and stirred for 3 h. The solution was used to prepare particles via spray pyrolysis. Citric acid was added to induce the hollow morphology of the spheres during spray pyrolysis [43]. The spray pyrolysis system consists of a droplet generator, quartz-tube reactor, and particle-collecting chamber. Six ultrasonic generators (resonant frequency: 1.7 MHz) were used to generate a plethora of droplets. The droplets were carried into the high-temperature quartz-tube reactor (inner diameter: 55 mm) by a carrier gas (air, 40 L/min). Mo-containing precursor spheres were prepared through the pyrolysis of droplets containing molybdicenium precursors and collected on a Teflon bag filter in the particle-collecting chamber. The temperature of the chamber was held at ~250 °C to prevent water condensation. Details of the experimental setup were previously reported [44]. The reactor temperature was maintained at 700 °C. As-prepared precursor particles were heated at 450 °C for 2 h, washed five times with distilled water, and dried in air at 70 °C for 24 h. For simplicity, the specimen prepared by spray pyrolysis and subsequent heat treatment is referred to as “Mo-SP.”

For comparison, the following three specimens were also prepared. For the first specimen, 5 g of \((\text{NH}_4)_6\text{Mo}_7\text{O}_{24}4\text{H}_2\text{O}\) was heat-treated at 450 °C for 2 h, washed five times with distilled water, and dried in air at 70 °C for 24 h to prepare MoO₃ powders (referred to as “Mo-HT”). For the second specimen, MoO₃ powders were prepared using the method reported by Dhage et al. [45]; \((\text{NH}_4)_6\text{Mo}_7\text{O}_{24}4\text{H}_2\text{O}\) (6 g), urea (\(\text{NH}_2\text{CONH}_2\), Junsei, 99%) (1.2 g), and sodium dodecyl sulfate (\(\text{C}_{12}\text{H}_{25}\text{O}_7\cdot\text{Na}\), Sigma-Aldrich, 99%) (0.12 g) were dissolved in 200 mL of distilled water. The solution was titrated with nitric acid (HNO₃, Samchun, 60.0%) to a pH of 3.0. The precursor powders were prepared by ultrasonic transduction of the solution for 30 min. The MoO₃ specimens (referred to as “Mo-UT”) were then heat-treated at 450 °C for 2 h, washed five times with distilled water, and dried in air at 70 °C for 24 h. For the third specimen, 5 g of commercial molybdicenium(VI) oxide powder (MoO₃, Sigma-Aldrich, 99.5%) was heat-treated at 450 °C for 2 h, washed five times with distilled water, and dried in air at 70 °C for 24 h. This specimen is referred to as “Mo-CS.”

The phases and crystal structures of the particles were analyzed by X-ray diffraction (XRD, Rigaku D/MAX-2500/PC) using Cu Kα radiation. The morphology of the particles was observed using field-emission scanning electron microscopy (FE–SEM, S-4800, Hitachi Co. Ltd., Japan). The surface areas were measured using the Brunauer–Emmett–Teller method (BET, Tristar 3000, Micrometrics Co. Ltd, USA). Small amounts of MoO₃ powders (0.01 g) were ultrasonically dispersed in distilled water (2 mL), and 0.2 μL aliquots of this slurry were dropped five times onto an alumina substrate (1.5 mm × 1.5 mm × 0.25 mm) with two Au electrodes. The substrate was dried at 60 °C for 20 min between the additions of each droplet. The sensor was heat-treated at 400 °C for 3 h to remove any moisture. A flow-through technique with a constant flow rate of 500 cm³/min was used, and a four-way valve was employed to switch the gas atmospheres. The responses of the sensors to 5 ppm TMA, \(\text{C}_2\text{H}_5\text{OH}, \text{H}_2, \text{CO}, \text{NH}_3, \text{NO}_2\), and toluene were measured at 300–400 °C by switching between the mixture gases (i.e., 5 ppm TMA, \(\text{C}_2\text{H}_5\text{OH}, \text{H}_2, \text{CO}, \text{NH}_3, \text{NO}_2\), and toluene, all in air balance) and dry synthetic air. The \(R_d/R_g\) (\(R_d\): resistance in air, \(R_g\): resistance in gas) and \(R_d/R_v\) values were used as the gas responses to reducing \((\text{TMA}, \text{C}_2\text{H}_5\text{OH}, \text{H}_2, \text{CO}, \text{NH}_3, \text{NO}_2\), and toluene) and oxidizing \((\text{NO}_2)\) gases, respectively. The dc–2 probe resistance of the sensor was measured using an electrometer interfaced with a computer.

3. Results and discussion

The as-prepared Mo-containing precursors showed weakly crystalline peaks in the X-ray diffraction patterns (Fig. 1a). After the heat treatment at 450 °C for 2 h, the compound converted into a crystalline orthorhombic MoO₃ (α-MoO₃) phase (JCPDS no. 05-0508) (Fig. 1b). The Mo-HT, Mo-UT, and Mo-CS powders after heat treatment also showed crystalline α-MoO₃ phases (Fig. 1c–e). The low crystallinity of the as-prepared Mo-precursors despite the high reaction temperature (700 °C) is likely due to the short residence time (~3 s) of the droplets in the reactor during spray pyrolysis. The increase in crystallinity caused by the heat treatment at a
lower temperature (450 °C) for a longer period of time (2 h) supports this. By applying Scherrer’s equation to the (1 1 0), (0 4 0), and (0 2 1) peaks, the crystallite sizes of the Mo-SP, Mo-HT, Mo-UT, and Mo-CS powders were calculated to be $52.05 \pm 11.95, 89.92 \pm 21.28$, $99.34 \pm 16.57$, and $131.21 \pm 14.79$ nm, respectively. Although the Scherrer’s analysis is generally valid at the particle size smaller than 100 nm, these values indicate that the crystallites of the Mo-SP powder are the smallest among the specimens.

Spherical Mo-containing precursors with clean surfaces were prepared by spray pyrolysis of droplets (Fig. 2a). The Mo-containing precursors were identified to be the hollow spheres from TEM analysis (Fig. 2b) and thickness of shell ranged from 35 to 50 nm (Fig. 2c). After the heat treatment at 450 °C for 2 h, the samples comprised thin plates (edge size: 0.5–2 μm, thickness: ∼100 nm) or small particles (50–100 nm) (Fig. 2d and e). High crystallinity of α-MoO₃ nanoplatelet was confirmed by high resolution TEM image and selected area electron diffraction pattern (Fig. 2f). α-MoO₃ has an anisotropic structure containing layers parallel to the (0 1 0) crystal plane [46], which leads to the formation of α-MoO₃ nanoplatelets and nanobelts [11,46–49]. Accordingly, the loss of the spherical morphology during the heat treatment can be attributed to the transformation from curved hollow shells into flat nanoplates. We tried to prepare MoO₃ hollow spheres by heat treatment of Mo-containing hollow spheres but failed probably because of significant grain growth and densification at heat treatment temperature (450 °C) due to the low melting point of MoO₃ (795 °C). Thus, the gas sensors using MoO₃ hollow spheres could not be measured. The Mo-SP, Mo-HT, Mo-UT, and Mo-CS powders had plate-like morphologies (Fig. 2g–i). The plates in the Mo-HT, Mo-UT, and Mo-CS specimens are ∼0.25, ∼0.40, and ∼2.20 μm thick (Fig. 2g–i), respectively, and are significantly thicker than those in the Mo-SP specimen (∼0.10 μm). Moreover, the order of the edge sizes of the plates (i.e., Mo-SP < Mo-HT < Mo-UT < Mo-CS) is consistent with the order of the thicknesses of the plates. This is also in agreement with the tendency of the crystallite sizes, which were attained from X-ray diffraction, although the crystallites are smaller than the edges of the nanoplates because of the co-existence of small particles and coarse nanoplates.

The gas responses of the Mo-SP sensors were measured at 300–400 °C (Fig. 3). The response to 5 ppm TMA was maximized at

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**Fig. 2.** SEM and TEM images of (a–c) precursor spheres prepared by spray pyrolysis, (d–f) Mo-SP powders, (g) Mo-HT powders, (h) Mo-UT powders, and (i) Mo-CS powders.

**Fig. 3.** Gas responses ($R_d/R_g$) of the Mo-SP sensor to 5 ppm TMA, $C_2H_5OH$, $H_2$, CO, $NH_3$, $NO_2$, and toluene as a function of sensor temperature.
300 °C ($R_g/R_a = 374.74$) and then decreased to 96.54 with increasing sensor temperature to 400 °C. The 90% response times, the time to reach 90% variation of sensor resistance, upon exposure to 5 ppm TMA at 300–400 °C were short (2–3 s). In contrast, the 90% recovery times at 300, 325, 350, 375, and 400 °C upon exposure to air were 7157, 7021, 2338, 2667, and 2027 s, respectively. This indicates that the desorption of strongly adsorbed species or the adsorption of oxygen with negative charge takes relatively long time although the detailed mechanism should be studied further. The gas response was not measured at the lower sensing temperature because of the high sensor resistance and sluggish recovery kinetics. In contrast, the cross-response to 5 ppm C₂H₅OH was very low ($R_a/R_g = 6.09–9.62$) and the cross-responses ($R_a/R_g$ or $R_g/R_a$) to 5 ppm H₂, CO, NH₃, NO₂, and toluene were negligible (1.05–4.31). This clearly shows that TMA can be detected in an ultrasensitive and ultrasensitive manner using MoO₃ nanoplates prepared by ultrasonic spray pyrolysis with a subsequent heat treatment.

Fig. 4. Gas responses ($R_a/R_g$) of (a-1) Mo-SP, (b-1) Mo-HT, (c-1) Mo-UT, and (d-1) Mo-CS sensors to 5 ppm TMA, C₂H₅OH, H₂, CO, NH₃, NO₂, and toluene at 300 °C, and selectivities of (a-2) Mo-SP, (b-2) Mo-HT, (c-2) Mo-UT, and (d-2) Mo-CS sensors toward TMA ($S_{TMA}/S_{gas}$: response to TMA; $S_{gas}$: response to interfering gas).

To investigate the reason for the selective and sensitive detection of TMA, the gas responses of four different specimens were measured at 300 °C (Fig. 4): The responses of Mo-SP, Mo-HT, Mo-UT, and Mo-CS sensors to 5 ppm TMA were 373.74, 39.30, 11.93, and 33.92, respectively (Fig. 4a-1–d-1). The pore size distributions and surface areas were analyzed using nitrogen adsorption–desorption isotherms (Fig. 5). The BET surface areas of the Mo-SP, Mo-HT, Mo-UT, and Mo-CS specimens were 9.27, 2.36, 1.13, and 0.24 m²/g, respectively, which are consistent with the variation of the crystallite sizes determined by X-ray diffraction and the edge sizes and thicknesses determined via SEM analysis. The Mo-SP specimens displayed significantly higher pore volumes than the Mo-HT, Mo-UT, and Mo-CS specimens over the entire pore size range (2–90 nm), which indicates the high gas-accessibility of the nanoporous Mo-SP specimen. In n-type oxide semiconductor gas sensors, smaller or thinner nanostructures with higher surface area to volume ratios result in greater electron depletion, which induces a higher chemiresistive variation [50–52]. Moreover, significant agglomeration between primary particles or nanostructures often reduces the gas response because the particles near the surface of the secondary agglomerates shield the inner particles from reactions with the analyte gas [53,54]. From this perspective, the high gas response of Mo-SP in the present study is mainly attributed to effective electron depletion as a result of the thin plate structures and high gas reactivity due to gas-accessible nanoporosity. However, additional parameters such as the preferred orientation of the nanoplates may play a minor role in the gas-sensing reaction considering the higher response and selectivity toward TMA of the Mo-CS sensor than the Mo-UT sensor despite its low surface area and pore volume.

The ratios between the responses to TMA and other interfering gases (i.e., $S_{TMA}/S_{gas}$) were calculated as a measure of the selectivity for TMA. Mo-SP had the highest $S_{TMA}/S_{gas}$

![Fig. 5. Pore-size distributions of Mo-SP, Mo-HT, Mo-UT, and Mo-CS powders, as determined from nitrogen adsorption and desorption isotherms.](image-url)
values \(S_{\text{TMA}}/S_{\text{gas}} = 61.37–203.12\) (Fig. 4a-2); those of Mo-HT \(S_{\text{TMA}}/S_{\text{gas}} = 5.38–31.19\) (Fig. 4b-2), Mo-UT \(S_{\text{TMA}}/S_{\text{gas}} = 6.45–11.70\) (Fig. 4c-2), and Mo-CS sensors \(S_{\text{TMA}}/S_{\text{gas}} = 8.70–32.62\) (Fig. 4d-2) were significantly lower. Although the absolute \(S_{\text{TMA}}/S_{\text{gas}}\) values vary, all four sensors showed the highest response to TMA (Fig. 4a-1–d-1). Thus, the selective detection of TMA can be, in general, attributed to the characteristics of the sensing materials.

X-ray photoelectron spectroscopy (XPS) was performed to examine the oxidation state of Mo. The binding energies of Mo 3d\(_{3/2}\) peaks of Mo-SP, Mo-HT, Mo-UT, and Mo-CS specimens are 235.69, 235.85, 235.78, and 235.74 eV, while those of Mo 3d\(_{3/2}\) peaks are 232.57, 232.73, 232.67, and 232.65 eV, respectively (Fig. 6), clearly showing that all the specimens are MoO\(_3\) [55–57]. It is worthwhile to note that the binding energies of Mo 3d\(_{3/2}\) and Mo 3d\(_{3/2}\) peaks in Mo-SP specimen are 0.05–0.16 eV lower than those in Mo-HT, Mo-UT, and Mo-CS specimens. This indicates that the slight change of oxidation state cannot be excluded from the possible reasons for high gas response in Mo-SP sensor although further detailed study is necessary to confirm this.

The gas-sensing reaction of chemiresistive oxides comprises various steps including adsorption/desorption, a surface reaction, and a charge-transfer process. Accordingly, the acid-base interaction between the analyte gases and sensor surface as well as the electron transfers during the gas-sensing reaction should be investigated as important parameters for the gas response. The acidic MoO\(_3\) in the present study is highly reactive with basic gases such as TMA and NH\(_3\). The responses of the Mo-HT, Mo-UT, and Mo-CS sensors to NH\(_3\) in the present study were second highest, which led to relatively low \(S_{\text{TMA}}/S_{\text{NH3}}\) values (Fig. 4b-2–d-2) although Mo-SP deviated from this trend (Fig. 4a-2). This can be explained in part by the preferred adsorption of NH\(_3\) onto the surface of acidic oxides [58], and is consistent with the reports that MoO\(_3\) nanostructures showed high responses to NH\(_3\) [26–30] and that increased with the density of acid centers for MoO\(_3\)-loaded SnO\(_2\) sensors [59]. This result is also in good agreement with our previous report [13] that acidic WO\(_3\) hollow spheres showed the highest response to TMA and the second highest response to NH\(_3\) among C\(_2\)H\(_5\)OH, CO, CH\(_4\), C\(_3\)H\(_8\), H\(_2\), and NO\(_2\) analyte gases.

In order to investigate the effect of acid/base properties of MoO\(_3\) sensing materials on selective detection of TMA, the MoO\(_3\) sensor loaded with 1 at\% of La\(_2\)O\(_3\) have been prepared by ultrasonic spray pyrolysis and subsequent heat treatment at 450 °C for 2 h. The responses of sensor to 5 ppm TMA, C\(_2\)H\(_5\)OH, H\(_2\), CO, NH\(_3\), and Toluene at 300 °C were 8.22, 3.35, 2.35, 2.12, 2.58, and 1.34, respectively. Significant deterioration of selectivity and gas response to TMA by loading basic La\(_2\)O\(_3\) supports again that acidic property of MoO\(_3\) plays the key role in selective and sensitive detection of TMA. TMA has three methyl groups replacing the hydrogen atoms in NH\(_3\). Also, the nitrogen atom in TMA has a lone pair of electrons that can be donated to form bonds, which provides a method for adsorption onto Lewis-acid (Mo ion) sites. In addition, compared to the three hydrogen atoms in NH\(_3\), the three methyl groups in TMA can react more easily with the higher concentration of negatively charged oxygen on the surface, which significantly enhances the gas response. Currently, it is still unclear why the response of the Mo-SP sensor to NH\(_3\) is lower than that to C\(_2\)H\(_5\)OH. However, considering both the preferred adsorption of the gas molecule and consequent charge transfer, the charge-transfer process that occurs upon the reaction between C\(_2\)H\(_5\)OH and negatively charged oxygen seems to be more dominant in the gas response of the Mo-SP specimen than the preferred adsorption of NH\(_3\). The change of nanostructures that contain preferred specific crystallographic planes to being highly reactive toward gases [60] is a possible reason for this, although further detailed study is necessary.

To determine the low detection limit of TMA, sensing transients of the Mo-SP sensor to 0.25–5 ppm TMA at 300 °C were measured (Fig. 7). The resistance of the sensor decreased with increasing TMA concentration and recovered to the base air level upon exposure to air. From the log–log plot between the gas response and TMA concentration, the low detection limit of TMA was determined to be <45 ppb when \(R_0/R_2 > 1.5\) was used as the criterion for gas sensing; this value is significantly lower than the concentration of TMA required to monitor fish freshness and environmental conditions.

Ethanol is one of the most ubiquitous and representative indoor gases produced by culinary activities, cleaning products, and alcoholic beverages [61], and many oxide semiconductors such as SnO\(_2\) [62], ZnO [63], In\(_2\)O\(_3\) [21], Fe\(_2\)O\(_3\) [9], WO\(_3\) [10], and Cr\(_2\)O\(_3\) [63] show a high gas response to C\(_2\)H\(_5\)OH. Accordingly, the selectivity of the present sensors toward TMA over ethanol is very important.

To assess the gas-sensing characteristics of our sensor relative to those in the literature, the responses to TMA and \(S_{\text{TMA}}/S_{\text{ethanol}}\) values of the sensors from related reported works are summarized in Table 1 [12–18,40]. Many papers report gas-sensing characteristics to >100 ppm [64–74]. Thus, for comparison of the gas-sensing characteristics within a similar TMA concentration range, the responses
to <10 ppm TMA in the literature were used. To the best of our knowledge, the MoO3 sensors in the present study show the highest response to 1–5 ppm TMA and highest selectivity for TMA over ethanol. Note that the $S_{\text{TMA}}/S_{\text{ethanol}}$ values of the MoO3 sensor in the present study (61.4) and WO3 hollow spheres from our previous contribution (11.8) are significantly higher than those reported in other papers (1.4–3.27). The response to C2H2O by oxide semiconductors is also dependent on the acid/base properties of the sensing materials. Jinkawa et al. [75] reported that the response to C2H2O can be significantly enhanced by the addition of basic oxides. They suggested the following two different reactions to explain this phenomenon: (1) C2H2O is dehydrogenated into more active CH3CHO ($g$) + H2 ($g$) at the surface of basic oxides, or (2) C2H2O is dehydrated into less active C2H4 ($g$) + H2O ($g$) at the surface of acidic oxides. The high $S_{\text{TMA}}/S_{\text{ethanol}}$ values, i.e., low cross-responses to ethanol, are attributed to the decomposition of C2H2O into less active C2H4 ($g$) + H2O ($g$) at the acidic surface of the present MoO3 sensor.

4. Conclusions

MoO3 nanoplates were prepared by ultrasonic spray pyrolysis followed by a heat treatment to demonstrate ultrasensitive and ultrasensitive detection of TMA. The thin and hollow configuration of the Mo-precursor spheres resulted in less agglomeration and restrained particle growth during the heat treatment. The response of the MoO3 nanoplates to 5 ppm TMA ($R_0/R_2 = 374.74$) was significantly higher than that of the three reference sensors that used coarse MoO3 plates prepared by the heat treatment of precursors, an ultrasonically treated solution, and commercial powders, respectively. The ultrahigh TMA response of the MoO3 nanoplates is due to the significant electron depletion caused by the thin configuration of the nanoplates and effective diffusion of the analyze gases toward the entire sensing surface via nanoporous gas-accessible structures. The sensor showed ultrahigh selectivity for TMA with negligible cross-responses to other gases ($S_{\text{gas}}/S_{\text{TMA}} = 0.008–0.016$), which is attributed to the increased adsorption of basic TMA onto the acidic MoO3 surface and effective electron transfer by the active interaction between the three methyl groups in TMA and negatively charged oxygen adsorbed on the MoO3 surface.

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References


Biographies

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