One-step Preparation of TiO_2–carbonized Medium-Density Fiberboard for Volatile Organic Compound Photodegradation

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Photocatalytic carbonized medium density fiberboard (MDF) was prepared by one-step carbonization with titanium tetraisopropoxide (Ti-tip) as a TiO_2 precursor. Anatase TiO_2 was formed at carbonizing temperatures of 600 to 800 °C. However, at > 900 °C, most crystalline TiO_2 was rutile. Ti-tip-treated carbonized MDF (c-MDF) showed outstanding formaldehyde reduction performance with complete removal of formaldehyde from the chamber in 1 to 3 days. However, with non Ti-tip-treated c-MDF, formaldehyde remained after 20 days. No toluene was detected after 3 h on 50% Ti-tip-treated c-MDF, while toluene was continuously detected with other samples (10% and 5% Ti-tip-treated c-MDF, and untreated c-MDF). After 9 h ultraviolet exposure, toluene was completely reduced on 10% and 50% Ti-tip-treated c-MDF; reduction was only 20% on untreated c-MDF. In addition, c-MDF/TiO_2 prepared at 800 and 900 °C had significantly higher photocatalytic performances compared to those obtained at lower carbonization temperatures. Based on the results, the combination of 10% Ti-tip treatment and carbonizing at 800 °C provided the optimum photodegradation capacity for formaldehyde and toluene.

Keywords: Photodegradation; Volatile organic compounds; Catalysis; Anatase; Carbonization; Medium density fiberboard

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INTRODUCTION

After the 1970s energy crisis, building and house designs emphasized improved airtightness to save energy, known as passive house or building. Unfortunately, such construction causes other issues such as sick house syndrome, induced by poor indoor air quality from pollutant accumulation and decreased fresh air intake (Arvela *et al*. 2014). Indoor air quality is a basic requirement for happiness in the home (WHO 2010).

With increasing concern for human health related to indoor air quality, government organizations and industries have begun seeking solutions to eliminate toxic chemical compounds. Various ventilation, filtration, and adsorption systems have been developed with charcoal, activated carbon, *etc*. Activated carbon has been prepared with various materials such as wood and wood products (particle board and MDF) by various methods (Lopez *et al*. 1996; Kercher and Nagle 2003; Gomes *et al*. 2016; Cansado *et al*. 2017).

Typical activated carbon has the form of powder or granule type. Byrne and Nagle (1997a,b) started to prepare and characterize monolithic activated carbon from carbonized wood for using advanced materials applications.

The carbonized MDF (c-MDF) had been attempted for use in electrical applications (Kercher and Nagle 2002). However, c-MDF was prepared in a small size, so it was not suitable for use as interior finishing material in a construction site. In 2009, a non-cracked and untwisted carbonized board (aggregated carbon in a panel shape) was developed in large size (800 mm (width) x 1200 mm (length) as indoor construction materials with the capacity to adsorb volatile organic compounds (VOCs) such as formaldehyde (Park *et al*. 2009). The carbonized board was prepared by heating MDF in an electronic furnace without air contact. According to Park *et al*. (2008), the carbonized board could reduce formaldehyde concentrations by 97%, but its reduction of toluene was only 20%. In order to improve the toluene decomposition performance of the carbonized board, titanium dioxide (TiO_2) was applied as a well-known versatile material used in photocatalytic applications.

Among the three crystal forms of TiO_2 of anatase, rutile, and brookite, anatase-type TiO_2 has attracted significant interest as a photocatalyst because of its reduction-oxidation (redox) capability under ultraviolet (UV) irradiation (Fujishima *et al*. 1999). Many scientists have researched the application of sol-gel type TiO_2 to charcoal powder because of its excellent abilities in removing waste and purifying contaminated water (Molinari *et al*. 2000; Wu *et al*. 2008; Wang *et al*. 2013). The photocatalytic activities of TiO_2 crystallite-activated carbon composites for toxic chemical degradation have been investigated (Takeda *et al*. 1995; Torimoto *et al.* 1997; Tokoro and Saka 2001). Moreover, various types of TiO_2-wood composites were developed including a carbonized wood-TiO_2 by use of a simple physical mixer (Doi *et al*. 2000). These composites utilized wood charcoal as an adsorbent and anatase TiO_2 as a photocatalyst. However, in most case of usage, TiO_2 is produced by sol-gel methods and then applied to flat objects by coating or spraying.

The objective of this study was to prepare anatase-type TiO_2 on c-MDF using a one-step carbonization method with titanium tetraisopropoxide (Ti-tip) as a precursor of TiO_2. The formaldehyde and toluene decomposition performances of the TiO_2-coated c-MDF were investigated.

EXPERIMENTAL

Materials

As the carbonized board (c-board) precursor, MDF of 1.2 cm in thickness (0.64 g/cm^2, E_1 grade, Sunchang Industry, Incheon, Korea) was cut into pieces measuring 130 cm × 260 cm. To prepare the photocatalyst, Ti-tip ($Ti[OCH(CH_3)_2]_4$) and isopropanol (IPA) were used. All chemicals were purchased from Daejung Chemical Co. (Daejung Chemical & Metals Co., Ltd., Sihung-si, Korea).

Photocatalyst Precursor Treatment and c-MDF Preparation

The photocatalyst precursor Ti-tip was diluted to 5%, 10%, and 50% (w/w) with IPA. Approximately 7 g of the Ti-tip solution was applied to the top of the MDF, which was placed in an oven at 60 °C for 3 h. Triplicate samples at each dilution were prepared. The Ti-tip-treated MDF was then carbonized in an electronic furnace at maximum temperatures of 600, 700, 800, and 900 °C (Fig. 1). The thermal schedule was 50 °C/h ramping followed by holding for 0, 0.5, 1, or 2 h at the maximum target temperature.

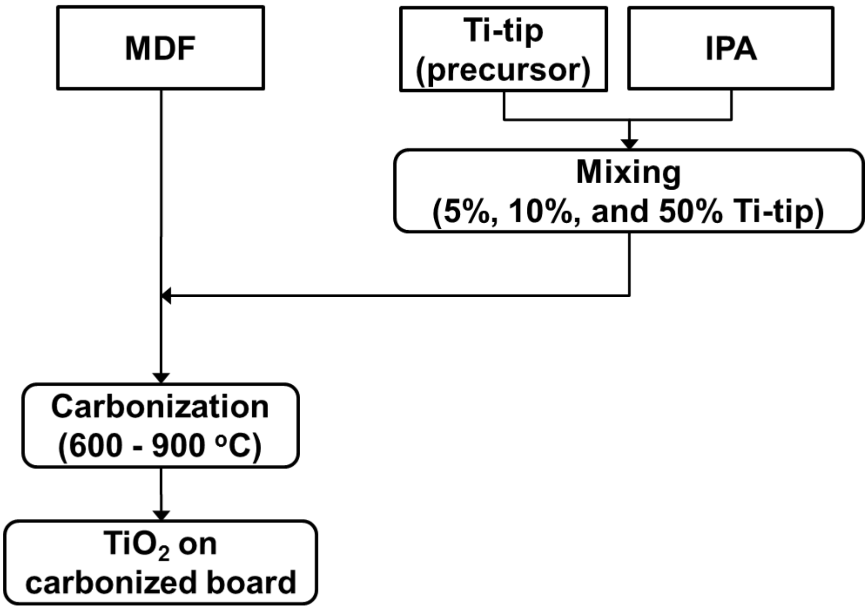


Fig. 1. Schematic of preparing TiO_2/c-MDF by one-step method

Crystallinity of Photocatalyst

The photocatalyst precursor Ti-tip was applied to the sample surface before carbonization. X-ray diffraction (XRD; D/Max-2500, Rigaku, Tokyo, Japan) was used to investigate the crystalline structure of TiO_2. After carbonization of the Ti-tip-treated MDF, part of the surface of each c-MDF specimen was peeled off and inserted into the mount.

Each sample was measured under the conditions of 40 kV and 30 mA in the range 5 to 80° from the starting angle. All patterns obtained were normalized and used. The surface of the Ti-tip-treated MDF after carbonization was also examined using scanning electron microscopy (SEM; JSM 5200 SEM, JEOL Ltd., Tokyo, Japan) and energy dispersive spectroscopy (EDS, JED-2300, JEOL Ltd., Japan). The test specimens were not coated and observed at magnifications of 200, 3500, and 10000×.

Photocatalytic Decomposition Performance of TiO_2/c-MDF

The formaldehyde and toluene reduction performances of the TiO_2-applied carbonized MDF (TiO_2/c-MDF) were tested by a modification of the ISO 16000-23 (2009) method. The top side of a 20 L chamber had a glass window to permit 360 nm UV light irradiation of the TiO_2/c-MDF. Samples were covered with aluminum tape, except for the Ti-tip-treated side, to avoid the effects from the non-treated surfaces such as the edges and back.

Formaldehyde reduction was conducted for 28 days under the testing conditions shown in Table 1. Toluene reduction was conducted by a modification of the 20 L chamber method (ISO 16000-23, 24 (2009)). The chamber was filled with 20 ppm toluene standard gas (Rigas Inc., Deajeon, Korea), and the inlet valve was connected to a N_2-filled Tedlar bag. Air sampling was performed through the outlet located on the top side to a Tenax-TA tube (Supelco, Bellefonte, PA, USA) for 1 L at 167 mL/min at 0, 1, 3, 5, 7, and 9 h of irradiation (Fig. 2). The toluene concentration in each air sample was analyzed by a gas chromatograph–mass spectrophotometer (GC/MS, Shimadzu, Kyoto, Japan) with thermal desorption (TD). Testing conditions are shown in Table 2.

Table 1. Sampling Condition and High-performance Liquid Chromatography (HPLC) Analysis Condition

List	Analysis condition
Chamber volume	20 L
Surface area	300 cm^2
Air flow	0.5 L/min
Sampling volume	10 L
Cartridge	2, 4-DNPH (Supelco)
HPLC	Shimadzu LC-20
Column	Nova-pac C18 4 μm (Waters, Milford, MA, USA)
Mobile phase	Water:acetonitrile (40:60 v/v)
Injection volume	10 μL
Light source	360 nm

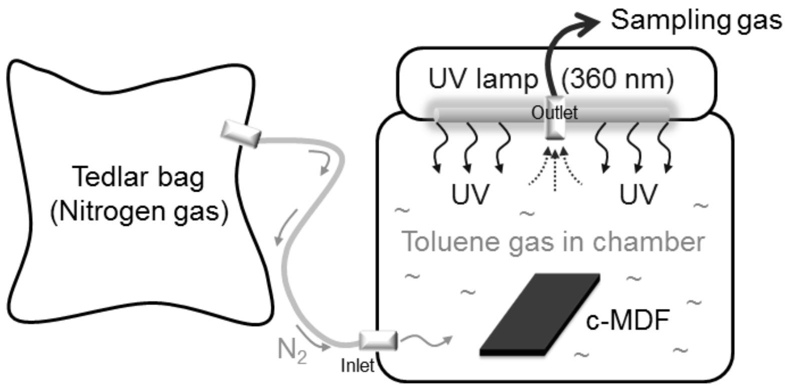


Fig. 2. Schematic of toluene reduction experiment

Table 2. GC-MS and TD Analysis Conditions

TD-20 (Shimadzu, Japan)		GC/MS (QP2010, Shimadzu, Japan)	
List	Condition	List	Condition
Desorption temp.	280 °C	GC column	VB-1 (0.32 mm × 60 m × 1 μm)
Desorption time	15 min	Initial	40 °C, 5 min
Cold trap temp.	−10 °C	1st ramp	10 °C/min, 80 °C, 16 min
2nd desorption temp.	300 °C	2nd ramp	20 °C/min, 200 °C, 4 min
Cold trap hold time	15 min	Column flow	1 mL/min
Cold trap packing	Tenax TA	MS source temp.	200 °C
Split	1:10	Detector type	EI (Quadrupole)
Valve temp.	210 °C	Mass range	35–350 amu
Transfer line temp.	250 °C	Electron energy	70 eV

RESULTS AND DISCUSSION

Preparation of c-MDF after Ti-tip Treatment

The amount of diluted Ti-tip applied to each specimen is shown in Table 3. The mass of IPA-diluted Ti-tip was 7.1 to 7.3 g. The amounts of Ti-tip actually present after oven-drying were 0.37, 0.75, and 3.67 g for the specimens treated with the 5%, 10%, and 50% dilutions, respectively. The Ti-tip solution treatment was correlated to areal loading weights of approximately 10.94 g/m^2, 22.19 g/m^2, and 108.58 g/m^2 for the treatment concentrations of 5%, 10%, and 50%, respectively.

Table 3. Average Weight of Ti-tip Applied on MDF

Concentration (%)	Applied Ti-tip/IPA (g)	Ti-tip (g)	Ti-tip (g/m^2)
5	7.12	0.37	10.94
10	7.32	0.75	22.19
50	7.21	3.67	108.58

Note: Area of MDF was 0.0338 m^2

The TiO_2/c-MDF specimens were successfully prepared with the same shapes as the original c-MDF boards; no negative effects were discovered in the surface color tone after Ti-tip treatment. With increasing Ti-tip concentration, the surface brightness became slightly greater than that of non-Ti-tip-treated c-MDF. This may be advantageous for application in distinguishing the treated and untreated sides. The shrinkages and weight reductions according to carbonization temperature are shown in Table 4. The averaged shrinkages and weight changes of the 5, 10, and 50% Ti-tip-treated c-MDFs were similar to those reported in the previous study, indicating no effects on the physical properties by Ti-tip treatment at varying concentrations.

Table 4. Summary of Average Shrinkages and Weights of Specimens by Carbonization Temperatures

	Temperatures (°C)	Width (%)	Length (%)	Thickness (%)	Weight (%)	Volume (%)	Density (%)
c-MDF	400	13.85	14.23	33.81	67.38	51.09	33.31
	600	20.00	18.77	38.57	71.68	60.08	29.05
	800	23.08	22.99	41.61	73.94	65.41	24.67
	1000	23.85	23.37	42.02	73.88	66.16	22.81
Ti-tip treated c-MDF	600	19.23	17.69	37.40	71.52	58.39	31.57
	700	20.00	19.23	33.93	70.37	57.31	30.59
	800	21.54	21.07	40.21	74.21	62.98	30.34
	900	33.59	21.46	30.64	73.59	63.82	27.00

Note: Data were collected and averaged from 5, 10, and 50% Ti-tip treated c-MDFs

Crystallinity of Photocatalyst

The formation of anatase TiO_2 was desired in this study. To the authors' knowledge, anatase TiO_2 has the highest photocatalytic activity toward toluene decomposition. The TiO_2 formed from Ti-tip by different carbonization procedures was examined to determine the best forming conditions. Figure 3 shows the results of crystal form analysis of 50% Ti-tip-treated c-MDF obtained by XRD. The 5% and 10% Ti-tip-treated c-MDF shows low peak intensities for TiO_2 because of the low concentrations of Ti-tip-treatment.

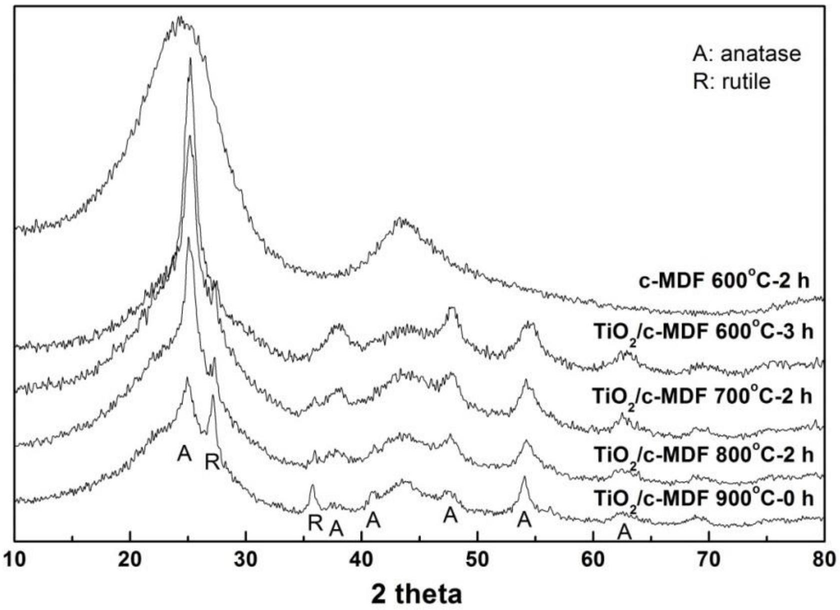


Fig. 3. XRD patterns of 50% Ti-tip-treated MDF *vs.* carbonization temperatures and times

The TiO_2 crystal phases in the 5% and 10% Ti-tip-treated c-MDF specimens were identified based on the 50% Ti-tip-treated c-MDF data. Under carbonization at 600 °C for 3 h, anatase appeared and remained present for samples carbonized at 800 °C for 3 h, while rutile appeared with 900 °C carbonization. No anatase was detected in the specimen carbonized at 900 °C for 0.5 h (Fig. 4). The XRD analysis suggests that anatase TiO_2 was mainly generated at 800 °C; therefore, carbonization at higher temperatures would be not recommended.

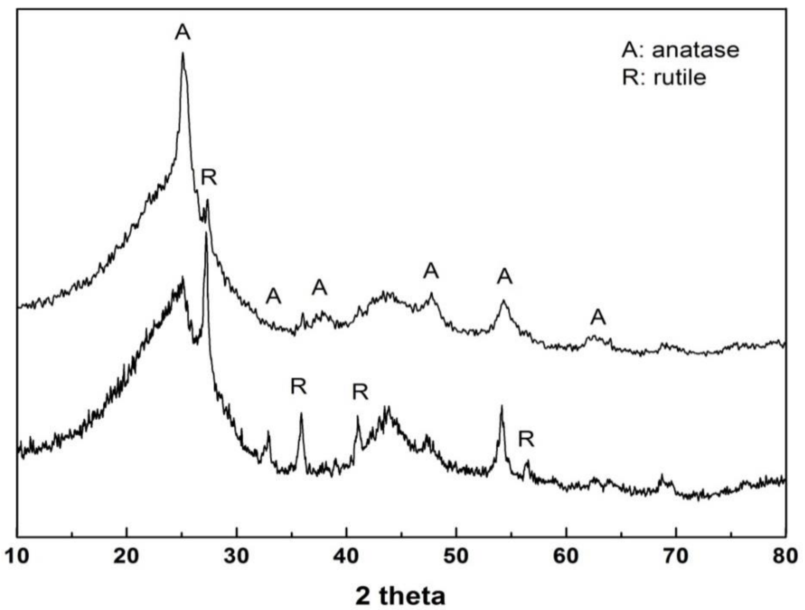


Fig. 4. XRD patterns of 50% Ti-tip-treated MDF after carbonization at 800 °C for 2 h (upper) and 900 °C for 0.5 h (lower)

SEM Observation of TiO_2/c-MDF

As shown in Fig. 5, on the 5% Ti-tip-treated c-MDF, TiO_2 crystals were present as individual particles with clearly observed wood fibers, while in the 10% Ti-tip-treated c-MDF, the TiO_2 crystals were aggregated in sea anemone-like clusters on the wood fiber. For the 50% Ti-tip-treated c-MDF, TiO_2 crystals formed a coating layer with individual TiO_2 crystals attached on the surface.

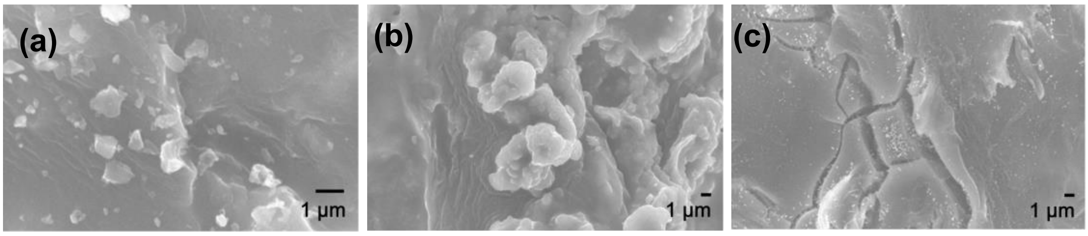
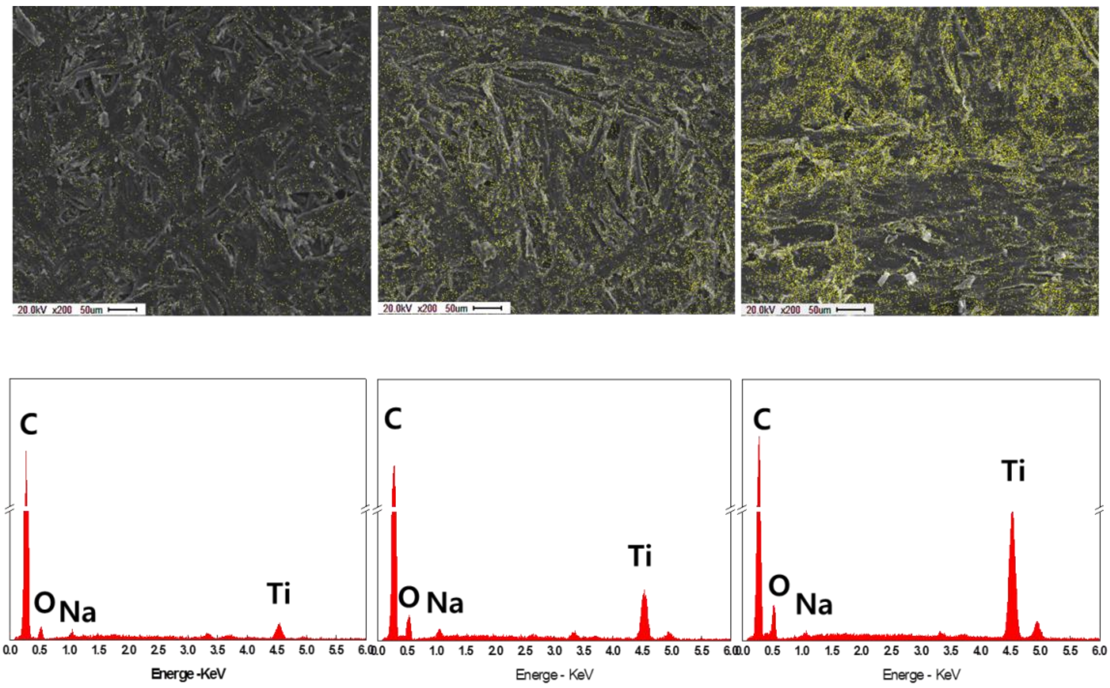


Fig. 5. SEM images of TiO_2/c-MDF; a) 5% Ti-tip treatment, b) 10% Ti-tip treatment, c) 50% Ti-tip treatment

Distribution of TiO_2 on c-MDF

The XRD analysis showed that Ti-tip was possibly converted to anatase TiO_2 with c-MDF simultaneously by the one-step method. Moreover, TiO_2 was well adhered to the carbonized wood fiber based on the SEM images. In this experiment, the distribution of TiO_2 on the c-MDF surface was analyzed.



5% Ti-tip 10% Ti-tip 50% Ti-tip

Fig. 6. Distribution of Ti (yellow dots) on c-MDF carbonized at 800 °C for 2 h

Figure 6 shows energy-dispersive X-ray spectroscopy (EDS) maps of the Ti distributions on the surfaces of the 5%, 10%, and 50% (TiO_2/c-MDF). Ti is evenly distributed on the specimen surfaces. Even Ti distribution was probably caused by the homogeneous diluted Ti-tip treatment. The Ti contents of the c-MDF surfaces were 1.9%, 3.6%, and 11.9% for the 5%, 10%, and 50% Ti-tip treatments, respectively. Theoretically, the total amount of Ti-tip applied should be on the TiO_2/c-MDF surface, but Ti loss was increased as the Ti-tip concentration was increased. This indicates that a large amount of Ti penetrated the MDF with the IPA used for Ti-tip dilution before IPA evaporation; therefore, some TiO_2 particles could not be detected by SEM-EDS. The loss of TiO_2 was increased for high-concentration Ti-tip-treated samples; therefore, the optimum concentration for Ti-tip treatment should be determined considering this loss after comparing the toluene decomposition performances.

Formaldehyde Decomposition Performance of TiO_2/c-MDF

Both Ti-tip treated and untreated c-MDF showed outstanding formaldehyde reduction performances for the 28 days of the study (Fig. 7).

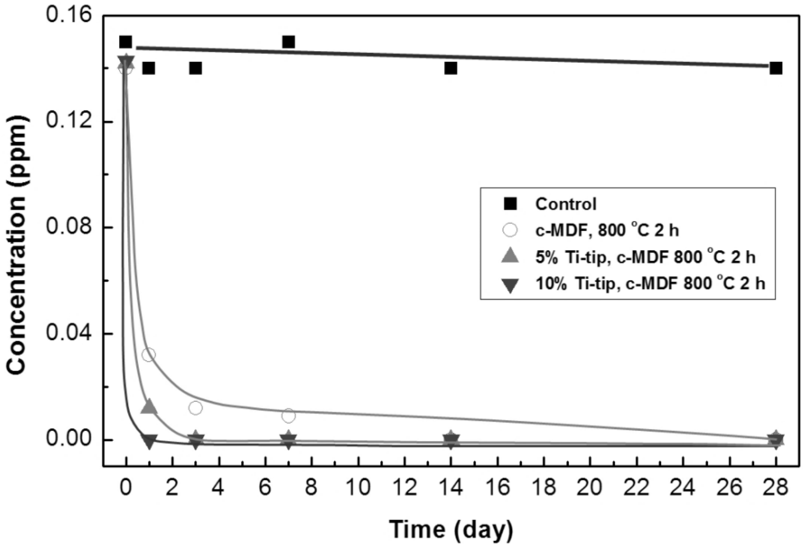


Fig. 7. Formaldehyde reduction by Ti-tip-treated and untreated c-MDF (Control indicates formaldehyde standard without sample)

However, in comparing Ti-tip-treated and untreated c-MDF, the 10% Ti-tip-treated c-MDF showed almost 100% reduction on day 1, whereas non-treated c-MDF showed 75% reduction on day 1. TiO_2 on c-MDF accelerated formaldehyde decomposition early in the testing period. At the end of the study (day 28), all samples showed almost 100% formaldehyde reduction. Therefore, it was concluded that TiO_2 does not interrupt formaldehyde reduction.

Toluene Decomposition Performance of TiO_2/c-MDF

The toluene decomposition performances of TiO_2/c-MDFs prepared at 800 °C for 2 h with different Ti-tip concentrations are shown in Fig. 8. The c-MDF showed lower toluene removal than the treated specimens. The 50% TiO_2/c-MDF showed the fastest toluene decomposition. Toluene was not detected in the 20-L chamber after 3 h with 50% TiO_2/c-MDF and after 5 h with 10% TiO_2/c-MDF. However, with the untreated c-MDF, more than 80% of toluene remained after 9 h (Fig. 8).

Figure 9 shows the toluene decomposition performance of c-MDF and 50% Ti-tip-treated MDF prepared at different carbonization temperatures. The 50% Ti-tip-treated MDFs after carbonization at 900 °C for 0 h and 800 °C for 2 h showed the highest toluene decomposition performance, with no difference observed between the two specimens. Therefore, the optimum carbonization conditions for achieving high photocatalytic activity is 800 °C with 2 h holding.

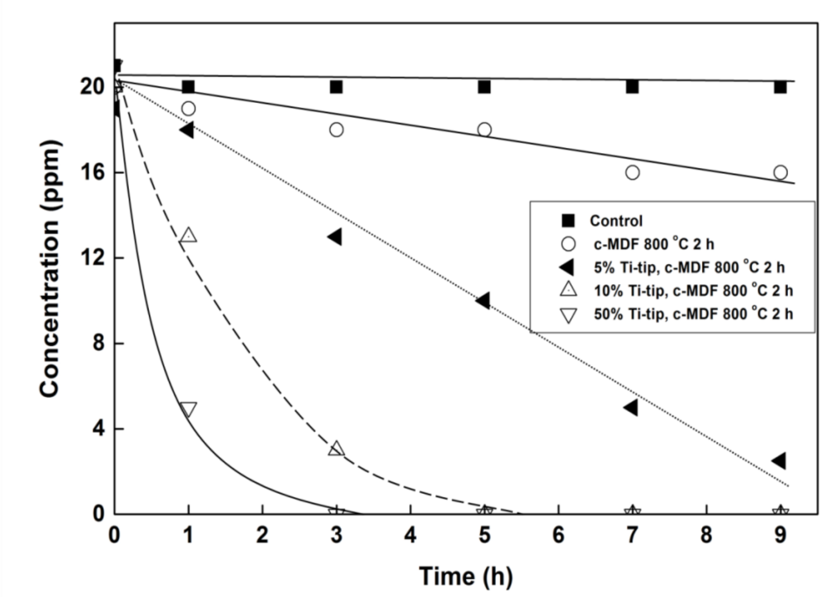


Fig. 8. Toluene decomposition performances of 5%, 10%, and 50% Ti-tip-treated MDF and untreated MDF after carbonization at 800 °C for 2 h (Control represents toluene standard without sample)

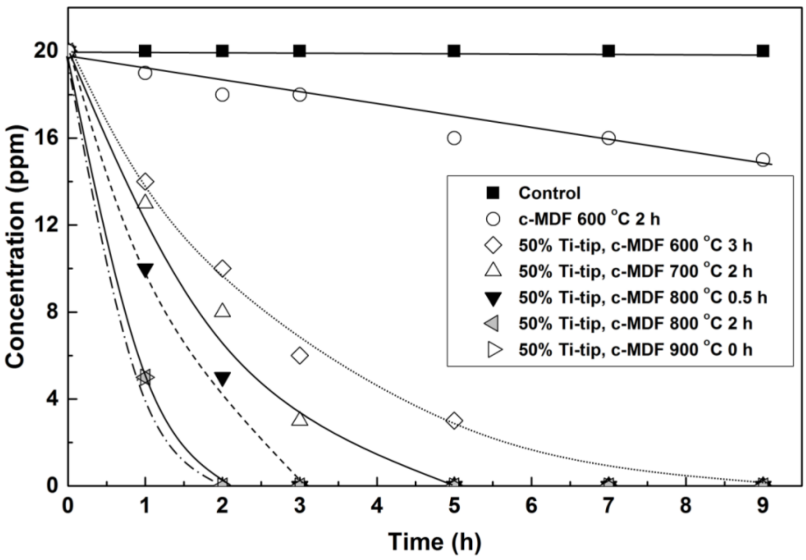


Fig. 9. Toluene decomposition performance of 50% Ti-tip-treated MDF and untreated MDF carbonized under different conditions (Control represents toluene standard without sample).

Meanwhile, in order to examine the persistence of the toluene decomposition performance, the same samples (10% Ti-tip, c-MDF 800 °C 2h) were repeatedly tested under the same conditions for 6 months. Outstanding toluene decomposition performance of TiO_2/c-MDF was maintained during the test period. At the each test, 20 ppm of toluene was 100% decomposed within 5 h by TiO_2/c-MDF.

CONCLUSIONS

1. A composite of carbonized medium density fiberboard with TiO_2 formed *in-situ* from titanium tetra-isopropoxide (TiO_2/c-MDF) was prepared by a one-step method of carbonizing MDF to which the TiO_2 precursor of titanium tetra-isopropoxide (Ti-tip) had been directed applied. During carbonization, Ti-tip was converted to TiO_2 and

MDF to c-MDF. The prepared TiO_2/c-MDF showed no apparent differences in dimensions compared to the c-MDF, but the surface became slightly brighter as the concentration of Ti-tip increased.

2. Anatase TiO_2 was identified at the carbonization temperatures of 600, 700, 800, and 900 °C. However, under carbonization at 900 °C for 0.5 h, rutile TiO_2 was mostly formed.
3. SEM analysis showed that using 5% Ti-tip yielded small particles of TiO_2. As Ti-tip concentration was increased to 10%, the TiO_2 particles self-aggregated in sea anemone-like shapes. For 50% Ti-tip treatment, most TiO_2 covered the c-MDF, with some TiO_2 nanoparticles (50 to 200 nm) observed on the surface of the TiO_2 layer. Moreover, Ti was uniformly distributed on the surfaces of the c-MDFs with all Ti-tip concentrations.
4. TiO_2/c-MDF showed an outstanding reduction effect toward both formaldehyde and toluene. For formaldehyde, the reduction rate on TiO_2/c-MDF was much faster than that on non-treated c-MDF.
5. Toluene was completely decomposed by TiO_2/c-MDF in 3 to 9 h under UV light, but only 20% toluene was removed by c-MDF after 9 h exposure. The photocatalytic activity toward toluene decomposition of the TiO_2/c-MDF was retained for 6 months.
6. Anatase TiO_2 was successfully formed by thermal treatment in this study. However, size of TiO_2 appeared to be of macro-size, so it needs to be prepared at nano-size for better photocatalytic activity.
7. TiO_2/c-MDF prepared by one-step method showed outstanding toluene decomposition under UV light. For the future study, TiO_2/c-MDF should be capable to degrade chemical under visible-light.
8. TiO_2/c-MDF might be applied as interior finishing material for houses and buildings as well as chemical factories.

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