テトラクロロダイオキシン類光分解の波長依存性

誌名	日本農薬学会誌
ISSN	03851559
著者	腰岡, 政二 山田, 忠男 金沢, 純
巻/号	14巻2号
掲載ページ	p. 181-187
発行年月	1989年5月

J. Pesticide Sci. **14**, 181–187 (1989)

Original Article

Wavelength-dependence of Photodegradation of Tetrachlorodibenzo-*p*-dioxins

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(Received September 8, 1988)

The photodegradation of 2,3,7,8-tetrachlorodibenzo-*p*-dioxin (2,3,7,8-TCDD), 1,3,6,8-TCDD and 1,2,3,4-TCDD in 1,4-dioxane solutions was investigated under xenon lamp irradiation. In a wavelength range of 199.8 nm to 397.9 nm, each of the TCDDs showed two maximal photodegradation peaks after 200-min irradiation. In the 2,3,7,8-TCDD solution, one of the maximal photodegradation peaks was observed at 252.6 nm, and the other in a wavelength range of 292.1 nm to 332.0 nm. In both 1,3,6,8-TCDD and 1,2,3,4-TCDD solutions, one of the maximal photodegradation peaks was at 252.6 nm, and the other at 305.6 nm. Since the energy spectrum of xenon arc is close to that of sunlight, it is likely that sunlight energy intensity affects the photodegradation of 2,3,7,8-TCDD, 1,3,6,8-TCDD and 1,2,3,4-TCDD in the environment to a large extent at 313.6, 305.6 and 305.6 nm, respectively.

INTRODUCTION

Polychlorinated dibenzo-*p*-dioxins (PCDDs) have been recognized as by-products of certain chlorinated phenols and the related compounds.[1] Among chlorinated dioxins, 2,3,7,8-tetrachlorodibenzo-*p*-dioxin (TCDD) is the most toxic, teratogenic, and acnegenic.[1,2]

2,3,7,8-TCDD in methanol solutions photodegrades rapidly when exposed to sunlight, but 2,3,7,8-TCDD thin films on glass and soil plates are stable to sunlight irradiation.[3] On the other hand, 2,3,7,8-TCDD on kieselguhr easily photodegrades under ultraviolet (UV) lamp irradiation.[4] Reductive dechlorination is shown as a major photodegradation reaction of PCDDs[5-10] as well as polychlorinated aromatic compounds.[11-14]

There is a number of studies on the photodegradation of TCDDs, but the photodegradation profiles in a wide range of irradiation wavelengths have not been clarified yet.

In the previous report, we determined the photodegradation products of 2,3,7,8-TCDD, 1,3,6,8-TCDD, and 1,2,3,4-TCDD by GC-MS, and proposed their possible photodegradation pathways.[15] In the present study, we investigate the photodegradation profiles of the above three TCDDs in 1,4-dioxane solutions at various wavelengths under xenon lamp irradiation, aiming to get information on their behaviors in the environment.

MATERIALS AND METHODS

1. *Procedures*

Test compounds 2,3,7,8-tetrachlorodibenzo-*p*-dioxin (2,3,7,8-TCDD) and 1,2,3,4-TCDD were purchased from Cambridge Isotope Laboratories and Gasukuro Kogyo Co. Ltd., respectively. Another test compound, 1,3,6,8-TCDD, was a gift from Mitsui-Toatsu Chemical Inc. Other standard compounds 1,2,4-trichlorodibenzo-*p*-dioxin (1,2,4-TrCDD), 1,6-dichlorodibenzo-*p*-dioxin (1,6-DCDD), 2,3-DCDD, 2,7-DCDD, 1-monochlorodibenzo-*p*-dioxin (1-MCDD), 2-MCDD and dibenzo-*p*-dioxin (DD) were also purchased from Gasukuro Kogyo Co. Ltd.

TCDDs were dissolved in 1,4-dioxane to make 1-ppm solutions. A 3-ml aliquot of each solution was taken into a cubic quartz cell (1×1×5 cm), and irradiated at 14 different wavelengths ranging from 199.8 nm to 397.9 nm with a JASCO CRM-FA xenon lamp irradiator. After irradiated for 16.7 min to 266.7 min, 1 μl of each solution was subjected to GC-MS for the analysis of photodegradation products.

2. Gas Chromatography-Mass Spectrometry

A Hewlett Packard 5890 GC equipped with a splitless injector and a 5970B series mass selective detector were used with a cross-linked methylsilicone capillary column (25 m length, 0.33 μm film thickness, 0.2 mm i.d.). Conditions: head pressure, 1056 g/cm²; carrier gas, He; EM volts, 1800–2400; initial temp., 60°C; initial time, 1 min; final temp., 250°C; program rate, 25°C/min; interface temp., 270°C; injection temp., 250°C.

RESULTS AND DISCUSSION

Tetrachlorodibenzo-*p*-dioxins (TCDDs) in 1-ppm 1,4-dioxane solutions easily degraded while irradiated under a xenon lamp for 16.7 min to 266.7 min, as shown in Table 1. As expected[11-14], an exponential decrease in the concentration of TCDDs generally accompanied an arithmetic increase in irradiation time at the wavelengths between 212.8 nm and 345.0 nm, where the energy intensity was 3.83 to 280.22 mcal/cm²·min (Table 2).

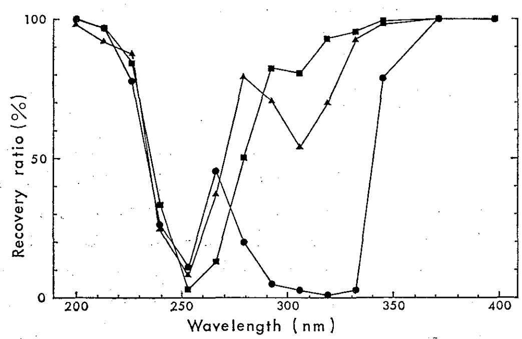

Fig. 1 Recovery ratios of 2,3,7,8-tetrachlorodibenzo-*p*-dioxin (2,3,7,8-TCDD), 1,3,6,8-TCDD and 1,2,3,4-TCDD in 1 ppm 1,4-dioxane solutions after 200-min irradiation by a xenon lamp.

●, 2,3,7,8-TCDD; ▲, 1,3,6,8-TCDD; ■, 1,2,3,4-TCDD.

As shown in Fig. 1, the recovery ratios of TCDDs were strongly affected by the irradiation wavelengths. Each of the recovery ratios of TCDDs in dioxane solutions after 200-min irradiation showed two maximal photodegradation peaks in a wavelength range of 199.8 nm to 397.9 nm. The 2,3,7,8-TCDD solution had one of the maximal photodegradation peaks at 252.6 nm, and the other in a wavelength range of 292.1 nm to 332.0 nm. The degradation ratio at the first peak was slightly smaller than that at the second one. In both 1,3,6,8-TCDD and 1,2,3,4-TCDD solutions, one of the maximal photodegradation peaks was at 252.6 nm, and the other at 305.6 nm. The photodegradation ratios at the first peaks were much larger than those at the second peaks in both solutions. The photodegradation degree at the first maximal peak increased in the order of 2, 3, 7, 8-TCDD<1, 3, 6, 8-TCDD<1, 2, 3, 4-TCDD, while that at the second peak was in the reverse order. The ratios (R_{λ_1/λ_2}) of the photodegradation degree at the first peak (λ_1 nm) to that at the second peak (λ_2 nm) increased in the order of 2,3,7,8-TCDD ($R_{252.6\,nm/318.6\,nm}=0.065$), 1, 3, 6, 8-TCDD ($R_{252.6\,nm/305.6\,nm}=6.82$) and 1, 2, 3, 4-TCDD ($R_{252.6\,nm/305.6\,nm}=28.75$). Pohland and Yang have also reported that the ultraviolet (UV) spectra of 2,3,7,8-TCDD, 1,3,6,8-TCDD, and 1,2,3,4-TCDD in chloroform had two UV absorption peaks at 248 nm (molar absorption coefficient $\varepsilon=2970$) and 310 nm ($\varepsilon'=5590$), at 250 nm ($\varepsilon=5540$) and 305 nm ($\varepsilon'=3340$), and at 257 nm ($\varepsilon=5290$) and 317 nm ($\varepsilon'=2290$), respectively, and that the ε/ε' ratios increased at 0.532, 1.61 and 2.75 in the order of 2,3,7,8-TCDD, 1,3,6,8-TCDD and 1,2,3,4-TCDD.[16] Thus, the order by ε/ε' ratio was the same as that by the degradation ratio found in photodegradation experiments, even though the wavelength of each peak does not exactly correspond each other. This suggests that the photodegradations of TCDDs are strongly affected by their UV absorption.

The disappearance of parental TCDDs was accompanied by the appearance of photodegradation products with a smaller number of chlorine atoms. After 200-min irradiation with a xenon lamp, 2,3,7,8-TCDD formed 2,3,7-TrCDD, 2,7-DCDD, 2,8-DCDD, 2-MCDD and

Table 1 Recovery percentages of tetrachlorodibenzo-*p*-dioxins, 2,3,7,8-TCDD (A), 1,3,6,8-TCDD (B) and 1,2,3,4-TCDD (C) in 1-ppm 1,4-dioxane solutions after xenon lamp irradiation.

Wavelength (nm)	Irradiation time (min)														
	16.7	33.3	50.0	66.7			133.3			200.0			266.7		
	(A)	(A)	(A)	(A)	(B)	(C)	(A)	(B)	(C)	(A)	(B)	(C)	(A)	(B)	(C)
199.8				100.0	100.0	100.0	100.0	100.0	100.0	99.9	98.0	99.9	99.8	96.0	99.3
212.8				99.1	99.8	100.0	98.3	98.2	99.9	96.6	92.9	96.6	95.3	91.3	92.8
226.2				95.3	94.3	95.9	88.5	90.8	92.2	77.4	87.3	83.8	69.2	80.8	77.2
239.2				69.7	59.4	69.3	43.6	40.6	48.5	26.1	24.5	33.2	17.9	17.5	25.0
252.6				44.9	34.3	36.7	22.6	13.1	11.0	10.7	7.9	2.8	8.5	7.0	0.7
265.6				81.5	71.5	54.1	69.7	51.7	26.5	45.3	36.9	12.9	29.1	25.2	6.4
279.1				54.3	93.9	81.0	34.2	84.5	61.7	19.7	78.7	50.1	9.4	71.0	43.6
292.1				31.6	90.9	92.0	8.5	81.3	84.3	4.7	70.3	82.2	3.4	60.3	78.0
305.6	82.0	51.0	22.0	7.5	81.7	97.2	4.2	68.3	84.3	2.5	53.9	80.5	1.7	45.2	77.7
318.6	70.0	41.5	22.0	6.0	87.7	98.6	1.7	78.3	94.4	0.7	69.8	92.7	0.2	59.1	88.3
332.0				23.9	97.5	99.9	7.7	96.9	98.2	2.6	92.2	95.3	1.4	87.7	94.3
345.0				98.3	99.9	100.0	88.5	99.9	99.9	78.6	97.9	99.2	68.4	95.7	96.2
358.4															
371.4				100.0	100.0	100.0	99.9	100.0	100.0	99.9	99.9	99.9	99.8	99.7	99.9
384.9															
397.9				100.0	100.0	100.0	100.0	100.0	100.0	99.9	100.0	100.0	99.9	99.9	99.9

Table 2 Energy intensity of a xenon lamp used for irradiation of tetrachlorodibenzo-*p*-dioxins.

Wavelength (nm)	Energy (mcal/cm²·min)
199.8	2.30
212.8	3.83
226.2	6.71
239.2	13.03
252.6	29.31
265.6	57.88
279.1	76.66
292.1	99.64
305.6	144.30
318.6	191.61
332.0	246.53
345.0	280.22
358.4	256.81
371.4	285.70
384.9	276.40
397.9	284.52

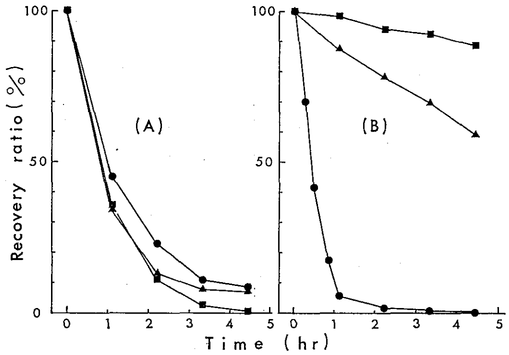

Fig. 2 Recovery ratios of 2,3,7,8-tetrachlorodibenzo-*p*-dioxin (2,3,7,8-TCDD), 1,3,6,8-TCDD and 1,2,3,4-TCDD in 1 ppm 1,4-dioxane solutions under xenon lamp irradiation at wavelengths of 252.6 nm (A) and 318.6 nm (B).

●, 2,3,7,8-TCDD; ▲, 1,3,6,8-TCDD; ■, 1,2,3,4-TCDD.

DD, 1,3,6,8-TCDD formed 1,3,6-TrCDD, 1,3-DCDD, 1,6-DCDD, 1-MCDD, 2-MCDD and DD, and 1,2,3,4-TCDD formed 1,2,3-TrCDD, 1,2,4-TrCDD, 1,2-DCDD, 1,3-DCDD, 1,4-DCDD, 2,3-DCDD, 1-MCDD, 2-MCDD and DD, as shown in Table 3. We have reported the identification of these photodegradation products in the previous paper.[15] They were mainly formed in the vicinity of the former maximal degradation peaks, and their relative intensities tended to decrease in the order of TrCDDs>DCDDs>MCDDs>DD on the GC-MS. At wavelengths where the recovered relative intensities of the parental TCDDs were as low as 0.007 at 318.6 nm in the 2,3,7,8-TCDD experiment and as 0.028 at 252.6 nm in the 1,2,3,4-TCDD experiment, the recovered relative intensities of the photodegradation products were also very low or unobtainable. This means that not only the parent TCDDs but their photodegradation products undergo drastic photodegradation at such wavelengths.

Typical photodegradation results of TCDDs in dioxane solutions at the wavelengths of 252.6 nm and 318.6 nm are shown in Fig. 2. At the wavelength of 252.6 nm, at which the first maximal photodegradation peak was observed, 1,2,3,4-TCDD almost completely degraded after 266.7-min irradiation, and 1,3,6,8-TCDD and 2,3,7,8-TCDD also degraded to less than 10% of the initial concentrations after 226.7-min irradiation. In contrast, at the wavelength of 318.6 nm, at which the second maximal degradation peak was observed, 2,3,7,8-TCDD almost completely decomposed after 266.7-min irradiation, but 1,3,6,8-TCDD and 1,2,3,4-TCDD only decomposed to 59% and 88% of the initial concentrations, respectively, even after 266.7-min irradiation. As TCDDs exponentially decreased with irradiation time, the photodegradation rate can be expressed by the following equation,

$$(-dp/dt)_\lambda = \varepsilon_\lambda \cdot E_\lambda \cdot P \cdot \phi_\lambda \qquad (1)$$

where λ is an irradiation wavelength, ε_λ is the molar absorptivity at wavelength λ nm, E_λ is the energy intensity of light absorption at wavelength λ nm, P is the TCDD concentration and ϕ_λ is the quantum yield at wavelength λ nm for reaction. Since the ε_λ and the ϕ_λ are constants for irradiated compounds, $\varepsilon_\lambda \cdot \phi_\lambda$ is rewritten by using photodegradation constant K_λ. Then the Eq. (1) becomes as follows.

$$(-dp/dt) = K_\lambda \cdot E_\lambda \cdot P \qquad (2)$$

Consequently, the TCDD concentration (P_T) after T-min irradiation at wavelength λ nm, where the absorbed energy intensity of light is E_λ cal/cm²·min, is expressed by the following

Table 3 Recovery ratios (shown in relative intensity) of parental TCDDs and their photodegradation products in 1-ppm 1,4-dioxane solutions after 200-min of xenon lamp irradiation at various wavelengths.

Compound	Wavelength (nm)													
	199.8	212.8	226.2	239.2	252.6	265.6	279.1	292.1	305.6	318.6	332.0	345.0	371.4	397.9
2,3,7,8-TCDD (**Ia**)	0.999	0.966	0.774	0.261	0.107	0.453	0.197	0.047	0.025	0.007	0.026	0.786	0.999	0.999
2,3,7-TrCDD (**IIa**)	0.022	0.041	0.152	0.312	0.308	0.242	0.045	0.017	tr[a]	tr	0.017	0.027	0.017	0.011
2,8-DCDD (**IIIa**)	—[b]	—	0.009	0.047	0.063	0.010	tr	—	—	—	—	—	—	—
2,7-DCDD (**IIIb**)	—	—	0.014	0.088	0.116	0.011	0.005	—	—	—	—	—	—	—
2-MCDD (**IVa**)	—	—	—	0.054	0.046	0.002	0.005	tr	tr	—	0.004	—	—	—
DD (**V**)	—	—	0.003	0.012	0.018	0.002	0.004	0.013	0.012	—	—	—	—	—
1,3,6,8-TCDD (**Ib**)	0.098	0.929	0.873	0.245	0.079	0.369	0.787	0.703	0.539	0.698	0.922	0.979	0.999	1.000
1,3,6-TrCDD (**IIb**)	0.010	0.022	0.107	0.382	0.471	0.503	0.034	0.015	0.001	0.001	0.003	0.005	0.002	—
1,6-DCDD (**IIIc**)	—	—	0.012	0.221	1.099	0.101	0.023	tr	tr	—	—	—	—	—
1,3-DCDD (**IIId**)	—	—	0.004	0.036	0.118	0.016	0.005	tr	tr	—	—	—	—	—
1-MCDD (**IVb**)	—	—	—	0.093	0.217	tr	0.004	—	—	—	—	—	—	—
2-MCDD (**IVa**)	—	—	—	—	0.128	tr	0.003	—	—	—	—	—	—	—
DD (**V**)	—	—	—	0.014	0.135	tr	0.002	—	—	—	—	—	—	—
1,2,3,4-TCDD (**Ic**)	0.999	0.966	0.838	0.332	0.028	0.129	0.501	0.822	0.805	0.927	0.953	0.992	0.999	1.000
1,2,3-TrCDD (**IIc**)	0.009	0.019	0.073	0.076	0.015	0.100	0.084	0.015	0.010	0.007	0.010	0.007	tr	—
1,2,4-TrCDD (**IId**)	0.004	0.021	0.063	0.090	0.015	0.149	0.114	0.058	0.065	0.021	0.014	0.006	tr	—
1,2-/1,4-DCDD (**IIIe**)	—	—	0.005	0.022	0.012	0.023	0.007	0.003	0.002	—	—	—	—	—
2,3-DCDD (**IIIf**)	—	—	0.015	0.082	0.052	0.050	0.004	0.004	0.002	—	—	—	—	—
1,2-/1,3-DCDD (**IIIg**)	—	—	0.009	0.070	0.049	0.081	0.018	0.006	0.006	—	—	—	—	—
1,3-DCDD (**IIId**)	—	—	0.012	0.028	0.018	0.084	0.027	0.008	0.008	—	—	—	—	—
1-MCDD (**IVb**)	—	—	—	0.036	0.043	0.014	tr	—	—	—	—	—	—	—
2-MCDD (**IVa**)	—	—	—	0.029	0.030	0.009	tr	—	—	—	—	—	—	—
DD (**V**)	—	—	0.005	0.050	0.029	0.008	0.013	0.007	0.011	—	—	—	—	—

[a] tr: trace. [b] —: not detected.

Table 4 Photodegradation equations and half-life times of tetrachlorodibenzo-*p*-dioxins (TCDDs) at the maximal degradation peaks under xenon lamp irradiation.

Compounds	Maximal photodegradation wavelength	Equation	Half-life time (min)
2,3,7,8-TCDD	252.6 nm	$P=0.8827 \cdot P_0 \cdot e^{-0.009546T}$ ($R=0.98685$)[a]	72.6
	318.6 nm	$P=0.6728 \cdot P_0 \cdot e^{-0.02331T}$ ($R=0.97637$)	29.7
1,3,6,8-TCDD	252.6 nm	$P=1.171 \cdot P_0 \cdot e^{-0.01018T}$ ($R=0.95856$)	68.1
	305.6 nm	$P=1.001 \cdot P_0 \cdot e^{-0.003006T}$ ($R=0.99912$)	225.5
1,2,3,4-TCDD	252.6 nm	$P=1.164 \cdot P_0 \cdot e^{-0.01874T}$ ($R=0.99792$)	37.0
	305.6 nm	$P=1.005 \cdot P_0 \cdot e^{-0.001040T}$ ($R=0.99690$)	666.7

[a] Correlation coefficient.

equation,

$$P_T = C \cdot P_0 \cdot \exp\,(-K_\lambda \cdot E_\lambda \cdot T) \qquad (3)$$

where P_0 is an initial concentration of TCDD, C is the equation constant. Based on above Eq. (3), the photodegradation equations of TCDDs at the maximal photodegradation peaks were obtained from the data listed in Tables 1 and 2. The equations of TCDDs and their half-life times at the maximal photodegradation peaks are shown in Table 4.

According to Crosby *et al.*[17] 286.3 nm is the shortest wavelength ever recorded on the earth surface, and solar energy below about 295 nm can be negligible. Since the energy spectrum of xenon arc is close to that of sunlight,[18] it is expected that the TCDDs, especially 2,3,7,8-TCDD, easily decomposes in the environment, because one of the maximal photodegradation peaks of 2,3,7,8-TCDD solution was at 318.6 nm, and one of the maximal photodegradation peaks of both 1,3,6,8-TCDD and 1,2,3,4-TCDD solutions was at 305.6 nm above 295 nm. The photodegradation of TCDDs depends on the total energy intensity absorbed, but the photodegradation of 2,3,7,8-TCDD, 1,3,6,8-TCDD and 1,2,3,4-TCDD in the environment will be strongly affected by sunlight energy intensity at 318.6 nm, 305.6 nm and 305.6 nm, respectively. The half-life times ($T_{1/2}$ min) of 2,3,7,8-TCDD, 1,3,6,8-TCDD, and 1,2,3,4-TCDD in 1-ppm 1,4-dioxane solutions at such wavelengths in the environment can be expressed by the following equations, based on the equations shown in Table 4,

$$T_{1/2}(2,3,7,8\text{-TCDD}) = 5.70/E_{318.6\,\mathrm{nm}} \qquad (4)$$

$$T_{1/2}(1,3,6,8\text{-TCDD}) = 32.54/E_{305.6\,\mathrm{nm}} \qquad (5)$$

and

$$T_{1/2}(1,2,3,4\text{-TCDD}) = 96.28/E_{305.6\,\mathrm{nm}} \qquad (6)$$

where $E_{318.6\,\mathrm{nm}}$ and $E_{305.6\,\mathrm{nm}}$ are the energy intensities (cal/cm^2·min) at the wavelengths of 318.6 and 305.6 nm, respectively.

ACKNOWLEDGMENTS

We acknowledge with thanks the authentic 1,3,6,8-TCDD received from Mitsui-Toatsu Chemical Inc. This research was partially supported by a grant for Environment Preservation Research from the Environment Agency of Japan.

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要　　約

テトラクロロダイオキシン類光分解の波長依存性

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ジオキサン溶液中のテトラクロロダイオキシン類(TCDD)，2,3,7,8-TCDD，1,3,6,8-TCDD，1,2,3,4-TCDD，はキセノンランプ照射下，容易に光分解を受け199.8 nm から 397.9 nm の範囲内でそれぞれ二つの光分解極大ピークを示した．2,3,7,8-TCDD では長波長側極大波長 (318.6 nm) での光分解度は低波長側極大波長 (252.6 nm) での光分解度より若干上回っていたが，1,3,6,8-TCDD および 1,2,3,4-TCDD については低波長側極大波長 (252.6 nm) での光分解度は長波長側極大波長 (305.6 nm) での光分解度よりきわめて大きかった．これらの化合物は太陽光が影響を及ぼすと考えられる295 nm 以上の波長域において光分解極大ピークを持つことから，環境中での分解はこの極大波長近辺でおもに起こるものと推察された．