



Byproduct Analysis from Gamma Radiolysis of 1,2,4-Trichlorobenzene in Benzene

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Abstract – In this study, we investigated the degradation properties of trichlorobenzene (TCB) as a model compound for hexachlorobenzene (HCB), utilizing benzene as the solvent, with the purpose of understanding the efficiency and pathways of gamma radiolysis in the remediation of persistent organic pollutants (POPs). Gamma radiation from a ⁶⁰Co source was employed, and mainly qualitative changes in the sample solutions were analyzed using gas chromatography-mass spectrometry (GC-MS). Our findings demonstrate a 99,1% conversion of TCB, resulting in the formation of various less chlorinated benzenes (CBs) and other chlorinated and non-chlorinated organic compounds. Specifically, 39 distinct compounds were identified in the TCB + benzene system based on their mass spectra. By-products such as, 3,5-dichlorobiphenyl, 4,4'-dichloro-1,1'-biphenyl, and 3,4-dichloro-1,1'-biphenyl (PCBs) exhibit significant carcinogenic hazards. These findings underscore the potential health risks associated with the radiolytic degradation process of POPs in benzene. The G value for TCB degradation in benzene exhibits 5,47 molecules/100 eV at an absorbed dose of 3,0 kGy. However, the G value subsequently declines to 0,19 molecules/100 eV with higher doses, a trend that reflects not only the saturation of the reactive species' capacity but also the diminishing concentration of TCB, limiting further degradation despite increased radiation exposure.

Keywords: trichlorobenzene, dechlorination, gamma radiolysis, chlorinated benzenes, POPs.

Технологии ликвидации источников химической опасности

Анализ побочных продуктов гамма-радиолиза 1,2,4-трихлорбензола в бензоле

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Аннотация – В этой работе исследовалась деградация трихлорбензола (ТХБ) как модельного соединения для гексахлорбензола (ГХБ), с использованием бензола в качестве растворителя, с целью понимания эффективности гамма-радиолиза при очистке от стойких органических загрязнителей (СОЗ). Использовалось гамма-излучение от источника ^{60}Co . Качественные изменения в растворах образцов анализировались с помощью газовой хроматографии-масс-спектрометрии (ГХ-МС). Наши результаты демонстрируют 99,1%-ную конверсию ТХБ, что приводит к образованию различных менее хлорированных бензолов (ХБ) и других хлорированных и нехлорированных органических соединений. В частности, в системе ТХБ + бензол на основе их масс-спектров было идентифицировано 39 различных соединений. Побочные продукты, такие как 3,5-дихлорбифенил, 4,4'-дихлор-1,1'-бифенил и 3,4-дихлор-1,1'-бифенил (ПХБ), проявляют значительную канцерогенную опасность. Эти результаты подчеркивают потенциальные риски для здоровья, связанные с процессом радиолитической деградации СОЗ в бензоле. Значение G для деградации ТХБ в бензоле показывает 5,47 молекул/100 эВ при поглощенной дозе 3,0 кГр. Однако значение G впоследствии снижается до 0,19 молекул/100 эВ при более высоких дозах, тенденция, которая отражает не только насыщение емкости реагирующих веществ, но и уменьшение концентрации ТХБ, что ограничивает дальнейшую деградацию, несмотря на возросшее воздействие радиации.

Ключевые слова: трихлорбензол, дехлорирование, гамма-радиолиз, хлорированные бензолы, СОЗ.

INTRODUCTION

Chlorinated organic compounds are a group of toxic chemicals widely utilized in various industries and agriculture [1]. HCB, a well-known member of these compounds, is recognized as a POP under the Stockholm Convention. Due to its hazardous nature, HCB use in agriculture has been prohibited [2]. However, HCB can still be generated as a byproduct during the manufacture of other chlorinated compounds [3]. Analyses of surface waters and soils globally reveal the presence of chlorinated pesticides at varying concentrations, raising concerns about their persistence in the environment [4]. By considering their long-term persistence in the environment, potential for bioaccumulation, and severe health risks, including carcinogenicity and endocrine disruption the degradation of POPs is crucial. Effective degradation of POPs not only mitigates environmental and health risks but would also reduce economic burdens associated with pollution cleanup and healthcare costs.

To remove these compounds from environment, various physical-chemical methods have been employed, including incineration, phytoremediation, ozone-based processes, microbial degradation, electrochemical and electrocatalytic degradation, photochemical and photocatalytic degradation, and catalysis [5–15]. While these methods are effective to some extent, they have drawbacks. Incineration can release more toxic compounds or gases, such as dioxins and furans, into the atmosphere, posing additional health and environmental risks [16]. Phytoremediation, while environmentally friendly, is often slow and limited by the bioavailability of pollutants and may be ineffective for highly contaminated sites and certain POPs [17]. Ozone-based processes can be costly and require careful control to prevent the formation of harmful by-products like bromates and aldehydes [18]. Microbial degradation can be

slow and is often hindered by the presence of other toxic compounds, with its effectiveness dependent on specific environmental conditions [19]. Electrochemical and electrocatalytic degradation methods can be energy-intensive and may require expensive electrodes and catalysts, with a risk of generating secondary pollutants [20]. Photochemical and photocatalytic degradation processes often require UV light or other sources of radiation, which can be costly and energy-intensive, with efficiency limited by natural sunlight and potential formation of harmful intermediates [21]. Catalytic processes, while effective in breaking down POPs, can be expensive due to the cost of catalysts, which may become deactivated over time and require regeneration or replacement [22]. These drawbacks highlight the need for developing more efficient, cost-effective, and environmentally friendly methods for the remediation of POPs.

Ionizing radiation presents a promising alternative for the destruction of toxic compounds by breaking down their molecular structure. Several studies have investigated the use of radiation for environmental treatment, highlighting its advantages such as a single-step process, applicability without specific conditions, and the absence of required additives or catalysts [22–26].

The current study focuses on the full conversion pathway of TCB in benzene under gamma radiation. Benzene was selected due to its non-polar nature and ability to dissolve TCB, facilitating a homogeneous distribution and enhancing interaction with gamma radiation. Additionally, benzene's common use in industrial applications makes these findings relevant for practical remediation efforts.

In our previous study, we examined the radiolytic degradation of TCB in some organic solvents, including benzene using gamma radiation [26]. We investigated the kinetic properties of TCB dechlorination and identified primary degradation pathways. The results demonstrated that methanol facilitated efficient TCB degradation with minimal harmful by-products. In contrast, benzene led to a broader range of by-products, including hazardous PCBs.

Building on these findings, the current study provides a more detailed analysis of the by-products formed during the gamma radiolysis of TCB in benzene. While the initial research identified the presence of toxic by-products, this study aims to fill the gap by extensively characterizing the degradation products and evaluating their potential environmental and health impacts. This wider analysis is crucial for several reasons:

- Understanding the full spectrum of by-products and their toxicity is essential for assessing the environmental safety of using gamma radiolysis for POPs degradation in benzene.
- Detailed toxicity profiles will aid in compliance with environmental regulations and guidelines, ensuring that the degradation process does not inadvertently create new environmental hazards.
- By identifying the most hazardous by-products, we can optimize the radiolysis process to minimize their formation, potentially by modifying reaction conditions or exploring alternative solvents.

Overall, this study aims to enhance our understanding of the gamma radiolysis of TCB in benzene, providing valuable insights for the development of safer and

more effective POPs remediation strategies. The findings underscore the potential of gamma radiolysis as a promising method for the remediation of POPs, highlighting the importance of solvent selection to ensure safe and effective treatment. The evaluation provided in this study aims to inform better practices and guidelines for the safe and effective remediation of POPs through gamma radiolysis.

EXPERIMENTAL

The methodologies employed in this study are based on and extend the approaches utilized in the previous research on the radiolytic degradation of TCB [26].

Materials. 1,2,4-TCB with a purity greater than 99% was procured from MERCK. Gradient-grade benzene was obtained from LiChrosolv. Standard solutions containing 1,4 g/L of TCB were prepared for apparatus calibration and γ -irradiation.

Irradiation Procedure. Samples of TCB and benzene mixtures were prepared and placed in 2 mL glass bottles with Teflon screw caps. Irradiation was carried out at atmospheric pressure and normal temperature using the MRX- γ -25 apparatus equipped with a ^{60}Co source. The dose rate was determined using Fricke dosimetry, which utilizes ferrous sulfate, and was measured to be 1,67 Gy/s. Samples were irradiated at doses ranging from 0 to 457 kGy, corresponding to an irradiation timespan from 0 seconds to approximately 75,85 hours. Fifteen samples were prepared for irradiation, each exposed to specific doses in 0-457,0 kGy. Each experiment was conducted three times to ensure reproducibility.

GC-MS Analysis. The analysis of irradiated samples was performed using Gas Chromatography-Mass Spectrometry (GC-MS) with a Shimadzu QP2010 SE GC-MS. Molecules were separated on an Rxi-5ms low-polarity phase, diphenyl dimethyl polysiloxane capillary column (30 m $\times$ 0,25 mm I.D., 0,25 μm film thickness). A temperature gradient was applied, starting at 40°C (held for 2 minutes) and increasing to 250°C (held for 2 minutes) at a rate of 10°C per minute. Helium served as the mobile phase at a flow rate of 1 mL/min. Injection was carried out using a split/splitless injector maintained at 280°C in split mode with a split ratio of 20:1.

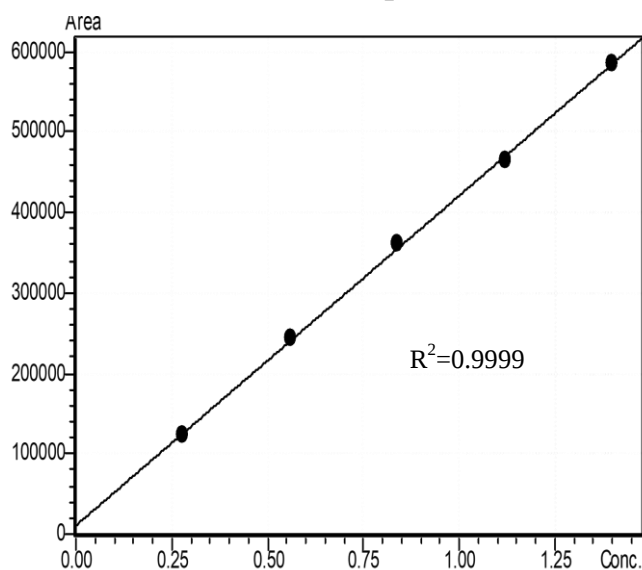


Fig. 1. Calibration curve for TCB quantification: Area of the peak = $f([\text{TCB}])$

Qualitative detection and quantification were performed using electronic ionization mass spectrometry (single quadrupole) in full scan and Single Ion Monitoring (SIM) modes. The calibration curve for TCB quantification was plotted as the area of the peak versus the concentration of TCB ([TCB]). The selected masses for SIM were: 74 m/z, 109 m/z, and 145 m/z for TCB. The source and transfer line temperatures were set at 210°C and 280°C, respectively. Separation of TCB, along with its chlorinated and non-chlorinated radiolysis products, was achieved within 27 minutes.

RESULTS AND DISCUSSION

The byproduct diversity of gamma radiolysis of TCB in benzene is extensive. A range of compounds, from small molecules to large polycyclic compounds such as benzene and terphenyls, have been identified using GC-MS. The chromatogram of irradiated TCB in benzene at only 48,0 kGy, shown in Fig. 2, demonstrates the successful separation on the adsorbent phase in the column.

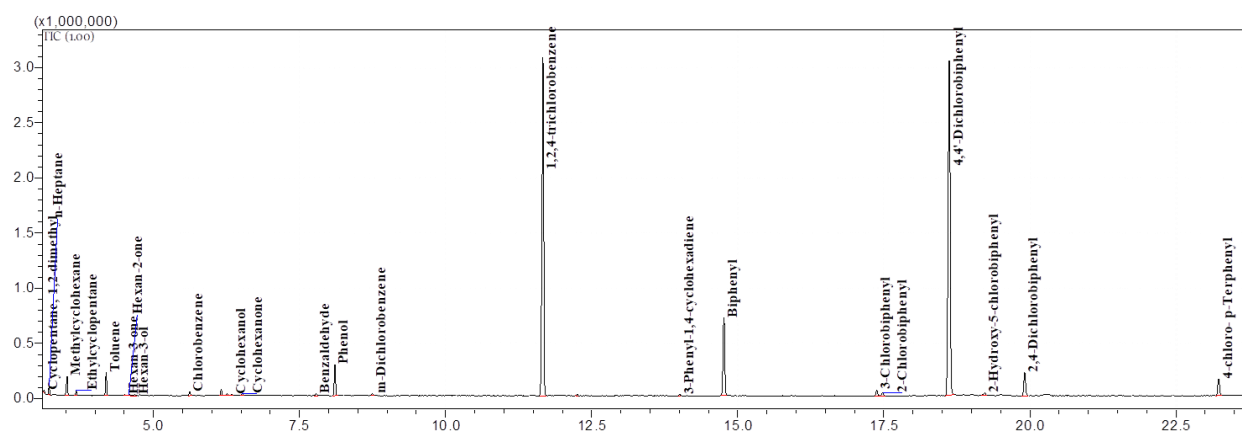


Fig. 2. The chromatogram of gamma irradiate sample of TCB + benzene at 48 kGy.

As shown in Fig. 3, 99,1% of TCB conversion has been achieved at 457,0 kGy dose, and the radiation chemical yield at 0 – 457,0 kGy, changes from 5,47 to 0,19 molecule 100 eV⁻¹.

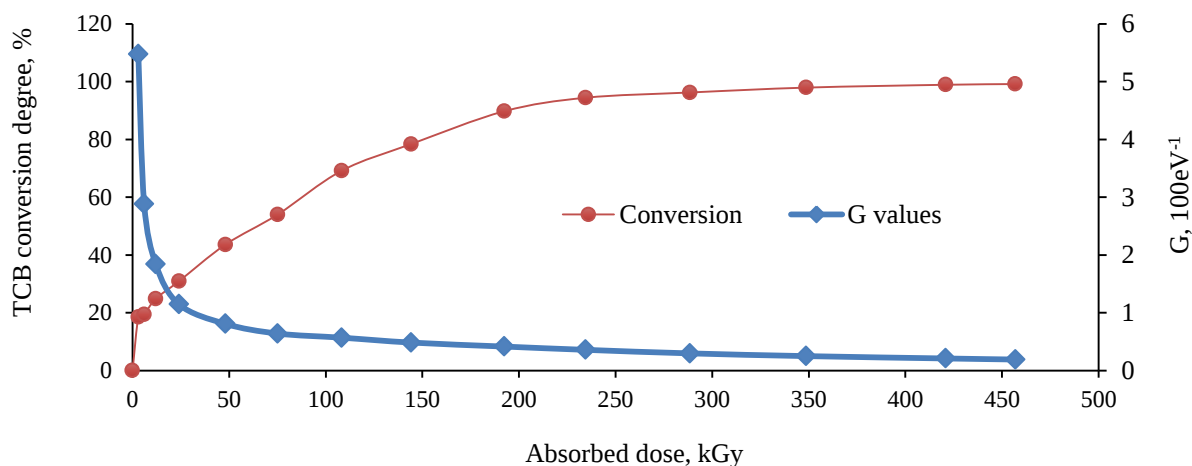


Fig. 3. The dependence of TCB degradation degree and G value of TCB degradation on absorbed dose.

As the absorbed dose increases at the beginning, the radiation chemical yield shows high value, reflecting an increase in the concentration of reactive species generated within the benzene medium [27]. These reactive species, produced by the interaction of ionizing radiation with benzene, significantly enhance the TCB degradation rate. 5,47 molecule 100 eV⁻¹ at 3,0 kGy occurs due to the initial high concentration of TCB. However, as the dose continues to increase, there is a notable decrease in the G values, dropping to 2,89 at 6,0 kGy and progressively declining to 0,19 molecule 100 eV⁻¹ at 457,0 kGy. This trend suggests that although higher doses continue to produce reactive species, the rate of TCB degradation does not increase proportionally. Beyond a certain point, the efficiency of degradation decreases, likely due to the depletion of available TCB, the potential recombination of reactive species, or the stabilization of intermediate products.

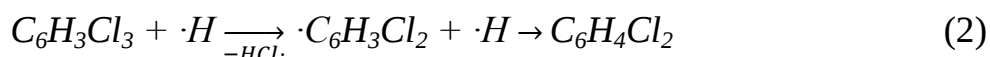
Predicted reaction mechanisms

The identified product molecules from the radiolysis of TCB in benzene can be classified into nine groups as in Fig. 4: *derivatives of cyclohexane; mononuclear aromatic derivatives; aliphatic hydrocarbons; oxygenated derivatives; chlorinated aliphatic derivatives; chlorinated terphenyls; polycyclic aromatic hydrocarbons; chlorinated CBs and polychlorinated aromatic compounds*. Among these, CBs are particularly important as they are byproducts of TCB's dechlorination. However, the formation of other potentially more toxic radiolysis products also necessitates further study to fully understand their implications.

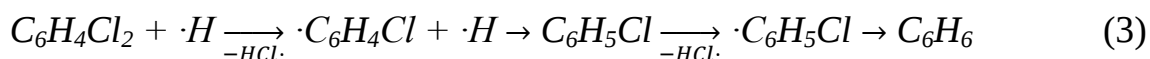
It is predicted that, the first interaction goes on the solvent molecule to form active species in the solution. Benzene molecules separate into 2 radicals under gamma rays in the first steps of irradiation as shown in the reaction (1) [26, 27, 28]:



It is expected that, hydrogen radicals attack to TCB molecules to form the chlorinated radical as intermediate species then dichlorobenzene (DCB) (compound number 12, Fig. 4) by elimination of $\cdot Cl$ radicals in reaction (2) [29]:



With continuation of this trend as in reaction (3), the compounds number 13–15 (Fig. 4), molecules of DCB dechlorinate into monochlorobenzene (MCB) and finally to benzene:



These sequential reactions show stepwise dechlorination of TCB happens under gamma rays in benzene medium. The formation of other materials, especially toxic compounds decreases the interest of the use of benzene for chlorinated organic pesticide degradation in it.

Oxygenated derivatives including hydroxyl, $-OH$ and carbonyl, $=CO$ groups containing compounds in Fig. 4, could be formed due to the presence of dissolved oxygen in TCB + benzene solution. Oxidative characteristics of oxygen in the samples causes oxidation of TCB and benzene molecules and their degradation

products such as cyclohexane derivatives of 3-ethylcyclohexanol, cyclohexanol, cyclohexanone.

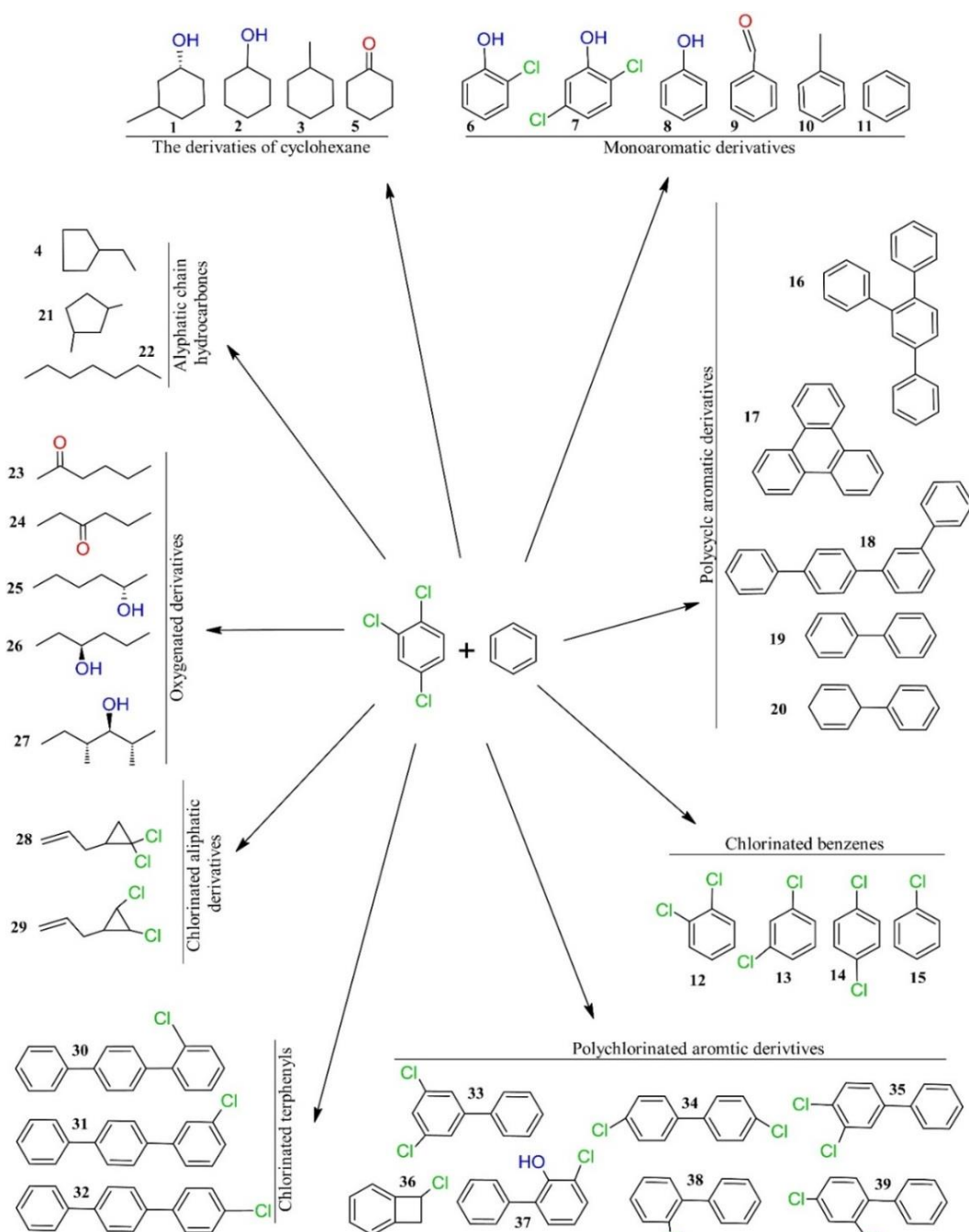


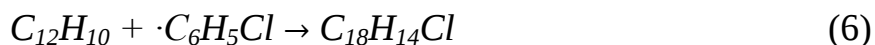
Fig. 4. The main product groups of gamma radiolysis of TCB + benzene identified in GC-MS in 0-457 kGy irradiated samples.

Monoaromatic derivatives including compounds number 6–7 and 8–10 in form from the dehalogenation product of TCB and the benzene respectively. The oxidation and reduction products 4 and 21–29 (Fig. 4) are the destruction products of aromatic rings, proofs that gamma rays are able to destruct strong cyclic compounds, such as benzene [30].

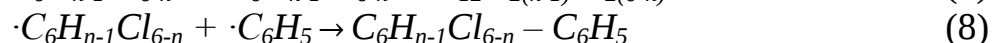
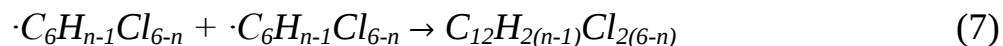
Polycyclic aromatic derivatives numbered 16, 18–19 (Fig. 4) are the products of the addition of $\cdot\text{C}_6\text{H}_5$ radicals with different combinations to form biphenyl (4) and isomers of tetracene (5) molecules [28]:



The isomer compounds 30–32 (Fig. 4), chlorinated terphenyls are predicted to be formed with the reaction (6) between $C_{12}H_{10}$ and MCB:



PCB congeners 33–39, are the products of chlorinated phenyl radicals (7) and phenyl radicals with chlorinated phenyl radicals (8):



Toxicology and Environmental Implications of By-products

The findings from this research on the gamma radiolysis of TCB in benzene illustrate the complexities and environmental considerations of using radiation to degrade POPs. Although 99.1% conversion of TCB in benzene showcases the efficacy of gamma radiolysis, it also highlights critical environmental trade-offs.

Our results demonstrated that while TCB is about fully degraded in benzene, the process produces a wide spectrum of by-products, including both chlorinated and non-chlorinated organic compounds as shown in Table 1. The formation of PCBs and chlorinated terphenyls as degradation by-products is particularly concerning.

These compounds are known for their persistence and bioaccumulation potential, and their presence during the degradation process poses significant environmental and health risks, potentially undermining the benefits of TCB removal.

Of particular concern are the PCB by-products identified as carcinogenic (Group 1) by the International Agency for Research on Cancer [31]. Many of the compounds summarized in Table 1 are known irritants, which can cause skin and eye irritation. Highly toxic and corrosive substances such as 2-chlorophenol and 2,5-dichlorophenol also emerged as by-products. The presence of these compounds highlights the potential acute and chronic health effects associated with exposure, including respiratory issues, skin burns, and systemic toxicity.

Our analysis of the chemical reactions occurring during the radiolysis process revealed intricate pathways involving the initial generation of reactive species from benzene. These species engage in a series of dechlorination reactions, effectively breaking down TCB but also leading to the formation of various other chemical entities mentioned above.

The environmental implications of these findings are significant. POPs identified in this study can bioaccumulate in the food chain, leading to long-term ecological and health impacts [32]. Furthermore, the considerable toxicity and carcinogenic potential of PCBs, as classified under the Stockholm Convention on POPs, emphasize the importance of carefully optimizing the conditions under which gamma radiolysis is conducted.

To mitigate these risks, it is essential to refine the conditions of gamma radiolysis to minimize the production of hazardous by-products. This includes selecting the most suitable solvents and adjusting treatment parameters such as dose

rate and irradiation time. Additionally, continuous monitoring and comprehensive risk assessments are vital to ensure that the degradation process does not inadvertently create new environmental hazards.

Table 1. Toxic background of gamma radiolysis products of TCB + benzene identified in 0–457 kGy

No	Name of the compound	CAS number	Maximum m/z in mass spectrum	Toxicity profile	Reference
1	3-Methylcyclohexanol	591-23-1	114	Harmful if inhaled	33
2	Cyclohexanol	108-93-0	82	Harmful if inhaled, Irritant	34
3	Methylcyclohexane	108-87-2	98	Irritant	35
4	Ethyl cyclopentane	1678-91-7	98	Not Classified (Limited data)	36
5	Cyclohexanone	108-94-1	83	Irritant, harmful if inhaled	37
6	2-Chlorophenol	95-57-8	128	Inhalation Toxicity	38
7	2,5-Dichlorophenol	583-78-8	162	Toxic, irritant	39
8	Phenol	108-95-2	94	Highly toxic	40
9	Benzaldehyde	100-52-7	106	Harmful if swallowed, irritant	41
10	Toluene	108-88-3	91	Toxic	42
11	Benzene ¹	71-43-2	78	-	-
12	1,2-DCB	95-50-1	146	Harmful if swallowed, irritant	43
13	1,3-DCB	541-73-1	146	Harmful if swallowed, irritant	44
14	1,4-DCB	106-46-7	146	Possible carcinogen (Group 2B)	45
15	MCB	108-90-7	112	Harmful if inhaled, irritant	46
16	1,2,4-Triphenylbenzene	1165-53-3	228	Not Classified (Limited data)	47
17	Triphenylene	217-59-4	228	Not Classified (Limited data)	48
18	1,1':3',1'':4'',1'''-Quaterphenyl	1165-57-7	306	Not Classified (Limited data)	-
19	1,1'-Biphenyl	92-52-4	154	Irritant	49
20	3-Phenyl-1,4-cyclohexadiene	4794-05-2	156	Not Classified (Limited data)	-
21	1,3-dimethylcyclopentane	2453-00-1	98	Irritant	50
22	n-Heptane	142-82-5	100	Irritant	51
23	Hexan-2-one	591-78-6	100	Harmful if swallowed, irritant	52
24	Hexan-3-one	589-38-8	100	Harmful if swallowed, irritant	53
25	Hexan-2-ol	626-93-7	102	Harmful if swallowed, irritant	54
26	Hexan-3-ol	623-37-0	102	Harmful if swallowed, irritant	55

¹ Benzene, being the solvent in the reaction mixture, has not been qualitatively analyzed as a product of the dechlorination of TCB in benzene. It is assumed to remain unchanged in quantity during the gamma irradiation process due to its role as the solvent.

Continuation of table 1					
27	2,4-Dimethyl-3-hexanol	13432-25-2	130	Not Classified (Limited data)	56
28	1,1-dichloro-2-propenylcyclopropane	17725-76-7	150	Not Classified (Limited data)	-
29	1,2-dichloro-3-propenyl cyclopropane	-	150	Not Classified (Limited data)	-
30	2-chloro-p-Terphenyl	21711-49-9	264	Not Classified (Limited data)	-
31	3-chloro-p-Terphenyl	1762-86-3	264	Not Classified (Limited data)	-
32	4-chloro-p-Terphenyl	1762-83-0	264	Not Classified (Limited data)	57
33	3,5-Dichlorobiphenyl	34883-41-5	222	Carcinogenicity Group 1	58
34	4,4'-dichloro-1,1'-Biphenyl	2050-68-2	222	Carcinogenicity Group 1	59
35	3,4-dichloro-1,1'-Biphenyl	2974-92-7	222	Carcinogenicity Group 1	59
36	7-chlorobicyclo[4.2.0]octa-1,3,5-triene	61599-88-0	138	Not Classified (Limited data)	-
37	3-chloro-2-ol-1,1'-Biphenyl	85-97-2	204	Carcinogenicity Group 1	59
38	2-chloro-1,1'-Biphenyl	2051-60-7	188	Carcinogenicity Group 1	59
39	2,4-dichloro-1,1'-Biphenyl	33284-50-3	222	Carcinogenicity Group 1	59

CONCLUSIONS

This study investigated the degradation of TCB using gamma irradiation in benzene, demonstrating 99.1% conversion of TCB into various less chlorinated and non-chlorinated organic compounds. However, the process also resulted in the formation of hazardous by-products such as PCBs and chlorinated terphenyls. These findings highlight the significant environmental and health risks associated with using benzene as a solvent for the degradation of POPs through gamma radiolysis. Given the persistent and bioaccumulative nature of PCBs and their adverse health impacts, benzene is not recommended as a solvent for this purpose.

The study emphasizes the critical need for selecting appropriate solvents to prevent the formation of secondary toxic pollutants during the degradation of chlorinated organic compounds.

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