

# Lindane, kepone and pentachlorobenzene: chloropesticides banned by Stockholm convention

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**Abstract** Persistent organic pollutants are a serious problem to the environment due to their toxicity to both fauna and flora. Extremely resistant to biodegradation and prone to transfer through long distances via atmosphere can contaminate almost any place in the planet. They tend to bioaccumulate in fat tissue due to their lipophilicity and seriously affect poisoned organism's nervous, hepatic, reproductive or hormonal system. Since 2009, due to the Stockholm convention on persistent organic pollutants production and utilisation of certain halogenated pesticides has been prohibited. This group includes hexachlorocyclohexane, chlordecone (kepone) and pentachlorobenzene. All of these chloropesticides pose a serious threat to environment, and careful control of their production and release to the environment is required. This paper is a review of physical and chemical properties as well as sources in environment, impact on animal organisms, methods of degradation of most broadly used chlorinated persistent organic pollutants and suggestions concerning their utilisation.

**Keywords** Persistent organic pollutants · Kepone · Lindane · Pentachlorobenzene · Chloropesticides · Health safety and environment

## Introduction

Persistent organic pollutants (POP) are compounds of high resistance to degradation in environment in natural processes, being by chance highly toxic and dangerous to animals, including humans (Petriello et al. 2014; Usman et al. 2014). Vast majority of them were used as pesticides by the time they were identified as toxin and found to be accumulated in fats (Lee et al. 2010). Marked as dangerous for human health and biosphere, their production and utilisation was regulated few times, with the most actual being Stockholm convention on persistent organic pollutants, signed in 2001. EU countries as one of the parties of United Nations accepted the Stockholm convention and obliged following the procedures indicated in protocol on persistent organic pollutants, including the Amendments Adopted by the Parties on 18 December 2009 (ECE/EB.AIR/104). The protocol regulate or ban several substances present in many biocides, as their bioaccumulation and easy propagation should not be left beyond control (Stockholm Convention on Persistent Organic Pollutants 2001). These substances were divided into two groups, depending on their threat to environment:

1. Substances whose production and utilisation is strictly banned (with some exceptions),
2. Substances which can be utilised and produced under special circumstances (Tables 1, 2).

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**Table 1** Substances included in Stockholm convention (prohibition)

Name	CAS number	Prohibited to use since (year)
Aldrin	309-00-2	2004
Chlordane	57-74-9	2004
Endrin	72-20-8	2004
Dieldrin	60-57-1	2004
Heptachlor	72-44-8	2004
HCB	118-74-1	2004
Mirex	2385-85-5	2004
Toxaphene	8001-35-2	2004
PCB*	200 + compounds	2004
HBB	36355-01-8	2010
Bromodiphenyl ethers*	Several compounds	2010
HCH— $\alpha$ , $\beta$ and $\gamma$ isomers	319-84-6; 319-85-7; 58-89-9 respectively	2010
Kepone	145-50-0	2010
PeCB	608-93-5	2010

\* Under certain conditions can be produced and utilised

**Table 2** Substances included in Stockholm convention (restricted use)

Name	CAS number	Restricted use since (year)
DDT	50-29-3	2004
PFOS	1763-23-1	2010
PFOSF	307-35-7	2010

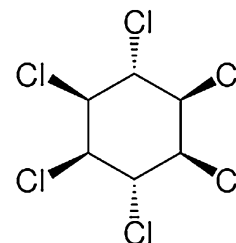
Characteristics of the chloro-derivatives of selected POPs, i.e. hexachlorocyclohexane isomers, kepone and pentachlorobenzene as well as routes of their biodegradation, are reviewed in this paper. The literature survey was conducted in February and March 2017 at Lodz University of Technology, Poland.

## Properties and production

### Hexachlorocyclohexane

Hexachlorocyclohexane (HCH), existing in eight isomers, is an organic molecule, halogenic derivative of a common cycloalkane. The names of its isomers are based on the location of the chlorine atoms in the structure. From all of them, only three isomers have significant influence in the topic of POPs— $\alpha$ -,  $\beta$ - and, most important one,  $\gamma$ -hexachlorocyclohexane, known as lindane (trade name).

Under normal conditions, it is a white solid with a colourless vapour, possessing a slight musty odour. It is neither flammable nor can autoignite. It dissolves very

**Fig. 1** Lindane structure

poorly in water (0.0073 g/L) but quite well in organic solvents (from 6.4 g/100 g of ethanol to 28.9 g/100 g of benzene) (U.S. Department of Health and Human Services 2002; Environmental Protection Agency 2010a, b). Its chemical structure is shown in Fig. 1.

The other two isomers:  $\alpha$  and  $\beta$ , are produced usually during the synthesis of lindane (Environmental Protection Agency 2010a, b). They have different physical properties. Although both have crystalline structure with brownish to white colour (Environmental Protection Agency 2010a, b) they differ in melting temperature (about 160 °C for  $\alpha$ -isomer and 315 °C for  $\beta$ -isomer) and solubility—both are almost insoluble in water but soluble in organic solvents (6.2 g/100 g for  $\alpha$ - and 1.8 g/100 g for  $\beta$ -in ethers). All data considering their physical properties are shown in Table 3.

Hexachlorocyclohexane is not a natural substance. It is synthesised during photochlorination of benzene, at least for technical grade purposes (U.S. Department of Health and Human Services 2002). To achieve a higher concentration of lindane, it is required to perform methanol or acetic acid treatment, followed by fractional crystallisation.

**Table 3** Properties of most important hexachlorocyclohexane isomers

Property	$\alpha$ -HCH	$\beta$ -HCH	$\gamma$ -HCH
Molecular mass	290.83	290.83	290.83
State	Crystalline solid	Crystalline solid	Crystalline solid
Colour	Brownish-white	Brownish-white	White
Melting point (°C)	159-160	314-315	112.5
Solubility (H <sub>2</sub> O) (ppm)	10	5	17
Solubility (organic solvents)	1.8 g/100 g ethanol 6.2 g/100 g ether	1.1 g/100 g ethanol 1.8 g/100 g ether	6.4 g/100 g ethanol 20.8 g/100 g ether

Its commercial production is allowed neither in the USA (2007) (Environmental Protection Agency 2006) nor in Europe (2009) (Lee et al. 2010). Nevertheless, before restricted use hundreds thousands of tonnes were released into the environment solely in Europe (European Parliament's Policy Department for Citizens' Rights and Constitutional Affairs).

The international HCH & Pesticide Association claims that global lindane use in 1950–2000 was around 600,000 tonnes, of which 450,000 had agricultural application, including 290,000 tonnes in Europe. It used to be spread on food crops or animals in USA and Europe in the highest amount (Usman et al. 2014) and was broadly used in China in 1970s and 1980s, where application of technical mixtures of HCH isomers lead to pollution with concentrations up to 30 ng/g of soil in Zhangzhou City (Yang et al. 2012) or 1000 ng/g in Tianjin Area (Gong et al. 2004).

### Kepone

Chlordecone (kepone) is an organic chloropesticide primarily used for banana root borer control in the central and south America main bananas exporters (Kadhel et al. 2014). As an effective insecticide, used in extremely high amounts, it was also used against leaf-eating insects, cockroaches and ants (Environmental Protection Agency 2010a, b). Until the mid 1980s, kepone was mainly manufactured in USA, by the time its high toxicity was discovered after some incidents (Faroon et al. 1995). One of the examples is James River estuary, where the kepone was produced in an amount of more than 457,000 kg by the time the health problems of workers were detected in 1975. It was found that more than 90,000 kg of kepone was released into the atmosphere. The soil adjacent to the manufacturing plant was contaminated in the range of 2–6  $\mu$ g of kepone per gram (Luellen et al. 2006). Similar situation is found in Caribbean, where chlordecone was used in the amounts of 3 kg per hectare per year in 1970s

and 1980s, leading to the pollution of 45% of agriculture area in Martinique, 8% of which is considered to be contaminated with more than 1 mg/kg of dry matter of Kepone (Fournier et al. 2017), providing threat to inhabitants (Clostre et al. 2017). Between 90 and 99% was exported to other continents (Europe, South America, Asia) afterwards. Before 1975 it is estimated that about 1.6 millions of kilograms of kepone were produced (US Department of Health and Human Services). Its production was finally banned in 2010 (Stockholm Convention on Persistent Organic Pollutants 2001), although there are still some suppliers in Europe or USA.

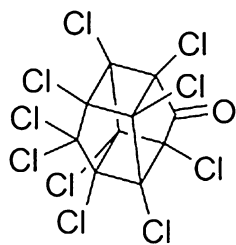
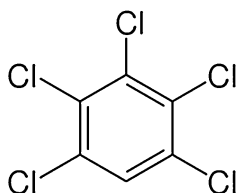
Kepone is a colourless or tan-coloured, odourless substance of crystal structure. It is well soluble in organic solvents like alcohols, acetone or ketones while practically insoluble in water (2.70 mg/L) (Faroon et al. 1995, Tsai 2010). It has good thermal stability (up to 350 °C) and quite high boiling point (434 °C) (Faroon et al. 1995, Tsai 2010). Its structure is shown in Fig. 2.

As mentioned before, kepone is neither longer produced nor commercially available. Before ban of production, it was synthesised through the reaction of hexachlorocyclopentadiene with sulphur trioxide (Cannon et al. 1978).

### Pentachlorobenzene

Pentachlorobenzene (PeCB) in the contrary to Kepone and HCH was not used primarily as a pesticide. On industrial scale it used to be a component added to polychlorinated biphenyls (PCBs), in order to reduce their viscosity affecting heat transfer (King et al. 2003; Sobiecka et al. 2009). Although production and utilisation of PeCB was banned in 2010, its huge amounts are found in old electrical equipment. They are thought to be a main source of PeCB release to environment nowadays, along with by-products released during many chemical or metallurgical processes. Significant amounts of PeCB are also already present in environment due to being a component of mix-



**Fig. 2** Kepone structure**Fig. 3** Pentachlorobenzene structure

tures of chloropesticides (King et al. 2003; Carrizo et al. 2008).

PeCB is a crystalline, white-coloured or colourless substance. It dissolves poorly in water (about 6.8 mg/L) (Shen and Wania 2005) while being decently soluble in organic solvents (insoluble in ethanol, slightly soluble in ethers, benzene, chloroform) (Carrizo et al. 2008; Shen and Wania 2005). It possesses a decent thermal stability (86 °C melting point, about 277 °C boiling point) (Haynes 2010–2011; Fig. 3).

The production of PeCB was prohibited in 2010. Nevertheless, current emissions of PeCB are estimated to be about 121 tonnes per year, mainly from biomass burning and solid waste combustion (Bailey et al. 2009). As it is a by-product or substrate of numerous reactions, there is a

need of substitution and elimination of this POP from unintentional production (Stockholm Convention on Persistent Organic Pollutants 2001; Bailey et al. 2009).

Current concentrations of PeCB differ depending on region. Annual average concentration in atmosphere across the USA and Canada after year 2000 is reported to be equal to about 45 and 33  $\mu\text{g}/\text{m}^3$  in Sweden; The estimated inflow of PeCB with water to the arctic sea until the end of previous millennium equalled about 400 kg per year, and to Humber estuary about 0.8 kg, respectively (Bailey et al. 2009; Meharg et al. 2000; Table 4).

### Sources and influence of selected POPs to environment

The main problem connected with persistent organic pollutants is their extreme resistance to biodegradation and bioaccumulation. All of them show high lipophilicity what facilitates their persistence in the environment (Tsai 2010). By the time they were ceased to be used as pesticides, huge amounts of them were produced and spread, but the exact amount of POP produced and synthesised is not possible to estimate due to numerous legal and non-legal producers. Nevertheless, there are three main sources of POPs in environment:

- POPs that remained in the environment after their utilisation,
- POPs that are still released into the environment due to chemical transformations of substrates needed for industry (metallurgy, etc.),

**Table 4** Comparison of physical properties of selected POPs

Feature	Lindane	Kepone	Pentachlorobenzene
Molecular weight (g)	290.83	490.63	250.32
Density ( $\text{g}/\text{cm}^3$ )	1.89 <sup>a</sup>	1.6	1.8 <sup>b</sup>
Melting point (°C)	112.5	349	86.1 <sup>a</sup>
Boiling point (°C)	Decomposes at 323.4 <sup>c</sup>	Decomposes at 349	275 <sup>d</sup>
Solubility in H <sub>2</sub> O (g/L)	7.3 <sup>c</sup>	2.7	$6.8 \times 10^{-5}$
Solubility in org. solv. (g/100 g)	6.4–20.8 (ethanol–ether) <sup>c</sup>	Unknown	Unknown
Common use	Insecticide	Insecticide, fungicide	PCB additive, substrate for cyclic chloro-derivatives of hexane

<sup>a</sup> Sigma-Aldrich substance data sheet (Online)

<sup>b</sup> IPCS substance data sheet (Online) <http://www.inchem.org/documents/icsc/icsc/eics0531.htm>

<sup>c</sup> Gestis substance database [http://gestis-en.itrust.de/nxt/gateway.dll/gestis\\_en/026380.xml?f=templates\\$fn=default.htm\\$3.0](http://gestis-en.itrust.de/nxt/gateway.dll/gestis_en/026380.xml?f=templates$fn=default.htm$3.0)

<sup>d</sup> Haynes, W.M. (ed.) CRC Handbook of Chemistry and Physics. 91st ed. Boca Raton, FL: CRC Press Inc., 2010–2011, p. 3–412



- POPs that remain present in equipment and may leak (e.g. PeCB in old electric devices).

The main reason of some of the POPs abundance in the environment, despite prohibition of their production or in the areas where they were never used, derives from their physical properties. Once disposed into the environment, they redistributed into several zones by vast number of processes like adsorption on particles, leaching, volatilisation, etc., what lead to pollution of whole ecosystem (Pereira et al. 2008). They were deliberately used on soils for years as pesticides by farmers, being by chance also sources of pollution also to the atmosphere or seas and oceans (Valle et al. 2005). The latter are the reason of POPs circulation in the biosphere, as pollutants may remain in oceanic-mixed layer and can back to atmosphere by dynamic air–water coupling, as described by Jaward et al. 2004. Volatilisation in hot areas and further deposition in colder ones has made lindane a worldwide problem (Buccini 2003), similarly to chlordecone, widely used on food crops in tropical countries and Europe, along with an insecticide mirex, providing kepone as an intermediate during its decomposition (Fernández-Bayo et al. 2013; Fonger 1995). The studies of bioaccumulation of chlorpesticides indicated their tendency to entry the plants mainly via air-plant route, especially for highly lipophilic contaminants like lindane, either by partitioning between air-suspended particles or volatilisation from soil and adsorption on leaves (Pereira et al. 2008). Despite being able to limit their application and prohibition of production, these compounds are still released into the environment as by-products due to chemical transformations of various halogen derivatives of benzene ring containing compounds or were used as components of the mixtures applied in electric industry (e.g. condensers cooled with PCB mixed with PeCB). Therefore, not only is it important to find effective and efficient ways of biodegradation of POPs, but also to prevent further contamination of the environment.

Unfortunately, it was not obvious from the beginning that the POPs had a potential harmful effect on environment and human, what was proved afterwards in many investigations. Wide utilisation for a long time against insects and fungi resulted in chronic contamination of the soil and water, severely affecting the organisms living in the contaminated habitat. It is obvious that the concentration of these selected POPs varies, depending on the country, utilisation history or current legislation. Never-

theless, traces of mentioned POPs, due to their atmospheric dispersion, can be found actually worldwide (Jimenez et al. 2015; Sharma et al. 2014; Nadal et al. 2015; Brunström and Halldin 2000; Pinzone et al. 2015; Pozo et al. 2013).

There are many publications concerning the effect and bioaccumulation of selected POPs on environment. Their lipophilicity results in massive accumulation in crops, where they were use. Persistent organic pollutants easily accumulate in marine organisms, especially those that possess significant fat content (Kelly and Gobas 2003; Deribe et al. 2011). The accumulated POPs can later on go upward the food chain due to predators. Lindane may change the fish behaviour as a neurotoxicant, causing darting, hyperexcitation and nudging when exposed to concentration of 10 µg/L of water (Croom et al. 2015; Nandan and Nimila 2012) with a concomitant decrease of blood quality and reduced ATPase activity (Croom et al. 2015; Nandan and Nimila 2012; Valle et al. 2005; Pesce et al. 2008). Kepone can cause lateral curvature of the spine causing scoliosis (Couch et al. 1977; Hansen et al. 1977) and extinction of small organisms like crustaceans or amphipods, when concentration exceeds 100 µg/L of water (Fournier et al. 2017). It is extremely toxic for some species like Donaldson trout or sheepshead minnow with a LC<sub>50</sub> of 56.9 and 21.4 mg/L, respectively (Vallero and Letcher 2013). Bioaccumulation in birds (either coastal or inland ones) is also observed, especially on the places where HCH or kepone were sprayed (Hong et al. 2014). It was proved that these pesticides can alter the behaviour of the animals, making them either neglect caring of the offspring, decrease awareness against predators or provoke feminization (Zala and Penn 2004). Considering mammals, lindane negatively affects ovulation rate and offspring quality in rabbits (Tiemann 2008) and degeneration of hepatocytes and kidney injuries in rats (Padma et al. 2011). It is also neurotoxicant and is provokes seizures at the concentration of 1.9 µg/mL of blood (Croom et al. 2015). Kepone on the other hand is cancerogenic and hepatotoxic in mice and rats (Tabet et al. 2016; Fournier et al. 2017) and affects also their reproductive capabilities at high chlordecone concentrations (Brown et al. 1991).

### Effect on human's health

Without a doubt, POPs negatively affect human health. Exposure to these substances is very easy—each of them can enter to any organism with contaminated food or meat,



HCH- $\gamma$ , additionally, can be inhaled. Lindane is known as a highly immunotoxic and neurotoxic poison. Not only does it decrease the number of lymphocytes, by chance promoting oncogenesis, but also, in case of acute poisoning, it causes nausea, headache and loss of consciousness. It is also expected to provoke seizures at the concentration of about 7  $\mu\text{g}/\text{mL}$  of blood (Croom et al. 2015). Kepone, on the other hand, is believed to be hepatotoxic due to mentioned experiments on mice (McCarty and Borgert 2006; Belfiore et al. 2007) and highly toxic to nervous and reproductive system on humans (Boucher et al. 2013; Grandjean and Landrigan 2014). Also, it may slow down psychomotor abilities in the infant life and provoke preterm birth (Belfiore et al. 2007; Dallaire et al. 2012; Boucher et al. 2013; Kadhel et al. 2014), and cause cancer (Sirica et al. 1989). The chronic health-based guidance value for chlordecone was set (for France) to no more than 0.5  $\mu\text{g}/\text{kg}$  body weight per day (Seurin et al. 2012). PeCB is highly toxic for pancreas (Gregoraszczyk et al. 2012) and a mild immunotoxicant (Schielen et al. 1996). Worth mentioning is that still long-term consequences of these POPs exposure are quite hard to assay. Due to research done about the group of compounds similar to selected ones—polychlorinated biphenyls—it is possible that they may influence reproductive system via dysregulation of CYP19 gene, influencing many biochemical processes based on oestrogen hormone (Li 2007; Mrema et al. 2013), provoke neuroabnormalities and can be chronically toxic for immune system (Grandjean and Landrigan 2014).

### Degradation of the lindane, pentachlorobenzene and kepone

As mentioned before, the biological degradation of persistent organic chemicals is a slow process. Due to their polyhalogenic character and toxic features they are almost invulnerable to microorganisms. Nowadays, majority of the POPs are either combusted (what is not recommended due to toxic wastes released into the atmosphere) or degraded chemically. Many ways of degradation of selected POPs were investigated. For lindane (and other isomers of HCH) one of the newest method is one employing iron (II) activated peroxydisulfate. Once supported by UV radiation it provides nearly perfect decarbonisation and dechlorination (Khan et al. 2015) due to strong role of  $\text{SO}_4^{2-}$  radical in slightly acidic environment (pH about 5.8) (Khan et al.

2016). Other methods include photodegradation with photo-Fenton reaction (Nitoi et al. 2013; Antonaraki et al. 2010; Senthilnathan and Philip 2010) assuring total organic carbon removal within 2 h and some biodegradation processes including oxidation by fungi (Guillén-Jiménez et al. 2012; Rigas et al. 2010) or bacteria using it as a carbon source (Pesce and Wunderlin 2004; Saez et al. 2012, 2014). But not only simple microorganisms can cope with them—also demosponges were found to be capable of its biodegradation (Aresta et al. 2015). Nature can also cope with lindane presence in atmosphere by gas-phase degradation with  $\text{OH}^-$  radicals in troposphere with no concomitant formation of other POPs (Vera et al. 2015).

Chlordecone, on the other hand, is much harder to be decomposed than lindane. In natural conditions, in soil, it is said that it can only be degraded by leaching (Buccini 2003). Nevertheless, its bioavailability to flora may be diminished by using sequestration, as kepone molecules can be trapped inside mesopores in allophane clays, especially when adding the compost (Woignier et al. 2013). Unfortunately, due to its structure, kepone is extremely hard to be degraded and there is limited evidence it may be biodecomposed. However, recent findings indicate that chlordecone is degraded in small level by microorganisms in tropical soil; however, they have not been discovered yet (Fernández-Bayo et al. 2013). Nevertheless, population that may be exposed to this POP should rely on minimal consumption of potentially contaminated crops and on water filtered with active carbon filters (Durimel et al. 2013). A very efficient method of kepone removal was established by Rana et al. (2017), with adsorbent consisting of iron oxyhydroxide/iron oxide nanoparticles and activated carbon, capable of adsorbing up to 79.8  $\mu\text{g}/\text{mg}$  of kepone.

PeCB, in contrary to kepone, is prone to be biodegraded. However, it requires probably anaerobic conditions, as there are no convincing proofs of its decomposition when it comes to aerobic microorganisms (Environmental Protection Agency 2010a, b). Biological dechlorination of PeCB was reported by many authors (Brahushi et al. 2004; Pavlostathis and Prytula 2000); nevertheless, it may persist in both soil and water for many years. Chemical degradation of pentachlorobenzene bases on dehalogenation processes, either with hydroxyl radicals in atmosphere (Atkinson 1990), cobalt salen-catalysed reduction (Gach et al. 2008), activated carbon modified with nitric acid, accompanied by temperature of 350 °C in a packed-bed reactor (Takaoka

et al. 2007) and commercially available products like flue gas with  $V_2O_5$ - $WO_3/TiO_2$  catalyst (Xu et al. 2012).

Nevertheless, when only it is possible, the microbial decomposition should be applied, as they provide the most eco-friendly and cheap process. But not only the microorganisms should be able to decompose selected POPs, but also be able to withstand their toxicity. One of the examples of microorganisms resistant to toxic compounds is species belonging to white rot fungi. *Phanerochaete chrysosporium*, *Trametes versicolor* and *Pleurotus ostreatus* are known producers of low-specific enzymes and are applied in biodegradation of nitroaromatic pollutants like 2,4,6-trinitrotoluene (TNT) or 3,5-dinitrophenol (DNP) (Madaj et al. 2016). Moreover, they are relatively easy to cultivate, which also make them attractive for chosen chloroorganic persistent organic pollutants biodegradation.

## Conclusion

Chloropesticides are persistent organic pollutants that pose a serious threat to the ecosystems where they were spread. Resistant to biodegradation, prone to be transferred through long distances via atmosphere can contaminate areas where they were not used. High toxicity of all PeCB, HCHs and Kepone causes that these compounds can influence both environment and human health, leading to multisystem problems, from liver damage to infertility and disability of children. It is required to minimise consumption of putatively contaminated crops by people, both by raising the awareness of POPs influence to health and biosphere and avoiding consumption of crops from contaminated areas. The polluted regions, especially those where substrates for food production come from, should undergo remediation processes or at least purification processes like application of flora capable of efficient POPs eliminating from soil before any food production. Another way that is not excluding soil from farming is to use crops of limited uptake of contaminants. One of the examples is application of crops categorised by Clostre et al. (2017). Research indicated that for chlordecone content of 0.1  $\mu\text{g}/\text{kg}$  of soil there is no need to change cropping system, for higher values only low-uptake crops like banana, cabbage or papaya should be farmed as products like cucumber, lettuce or radish can exceed the norms of 20  $\mu\text{g}/\text{kg}$  of fresh product. Considering lindane, the method developed by Khan et al. (2016) to remove it from water seems most efficient, as it

provides reasonable total organic carbon removal within hours. However, this method may be too expensive to be applied in poor regions, alike those proposed for PeCB degradation.

Research concerning decomposition and decrease of their production is necessary. Current technologies are either inefficient or very expensive what practically excludes their application in poor regions. As huge amounts of halogenated organic pollutants were produced within the years and released as well as some are still stored, there is a need to develop proper treatment technologies, enabling elimination of these compounds from contaminated biosphere along with their residues kept in warehouses. The best way would be development of advanced microbiological treatments, as they are much less expensive and more like to be applied in developing countries. Therefore, much more effort should be put in investigation concerning biodegradation.

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