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REVIEW ARTICLE

Contamination and remediation of phthalic acid esters in agricultural soils in China: a review

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Abstract Phthalic acid esters have been used as plasticizers in numerous products and classified as endocrine-disrupting compounds. As China is one of the largest consumers of phthalic acid esters, some human activities may lead to the accumulation of phthalic acid esters in soil and result in contamination. Therefore, it is necessary for us to understand the current contamination status and to identify appropriate remediation technologies. Here, we reviewed the potential sources, distribution, and contamination status of phthalic acid esters in soil. We then described the ecological effect and human risk of phthalic acid esters and finally provided technologies to remediate phthalic acid esters. We found that (1) the application of plastic agricultural films, municipal biosolids, agricultural chemicals, and wastewater irrigation have been identified as the main sources for phthalic acid ester contamination in agricultural soil; (2) the distribution of phthalic acid esters in soils is determined by factors such as anthropogenic behaviors, soil type, properties of phthalic acid esters, seasonal variation, etc.; (3) the concentrations of phthalic acid esters in soil in most regions of China are exceeding the recommended values of soil cleanup guidelines used by the US Environmental Protection Agency (US EPA), causing phthalic acid ester in soils to contaminate vegetables; (4) phthalic acid esters are toxic to soil microbes and enzymes; and (5) phthalic acid ester-contaminated soil can be remedied by degradation, phytoremediation, and adsorption.

Keywords Contamination source · Distribution · Phthalic acid esters · Remediation · Soil contamination · Toxicity

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1 Introduction

Phthalic acid esters (Fig. 1) are a class of refractory organic plasticizer compounds that are widely used in numerous products, such as medical equipment, upholstery, gaskets, composite moldings, piping, plastic roofing systems, rain wear, electrical wire insulation, and plastic film for food packaging and agricultural uses (Fig. 2). They also serve to provide paints with special coating properties (Horn et al. 2004; Abdel daiem et al. 2012). Since the addition of phthalic acid esters to plastics was to improve the flexibility of the plastic, thus, a low phthalic acid ester concentration would lead to hard products and a high phthalic acid ester concentration would produce soft and flexible plastic products, for example, medical devices and tubing containing 20 to 40 % diethylhexyl phthalate (Sathyanarayana 2008), and in some case as high as 50 % (Fatoki and Vernon 1990). The global production of phthalic acid esters is approximately 6.0 million tons year^{-1} (Xie et al. 2007). In China, phthalic acid esters account for 90 % of the plasticizer usage in polyvinyl chloride production. The current consumption of phthalic acid esters in China is more than 0.87 million tons year^{-1} and is predicted to increase (Teil et al. 2006). Phthalic acid esters are readily discharged from many plastic applications to the surrounding environment because they are not chemically bonded to plastic polymers (Li et al. 2004). These are some of the reasons that cause

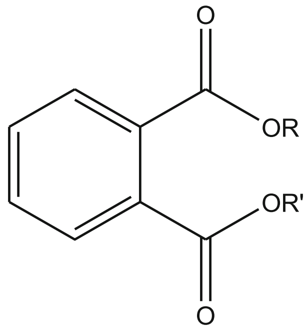

Fig. 1 The molecular structure of phthalic acid esters consisting of a benzene ring with two adjacent (-*ortho*) carboxylic acid side groups. The *R* and *R′* are general placeholders and the groups they presented are usually the same; if *R* and *R′* signify methyl, then the molecule is dimethyl phthalate, and if *R* and *R′* signify ethyl, the molecule is diethyl phthalate, etc.

phthalic acid ester contamination in water, sediment, and soil (Zeng et al. 2008; Dargnat et al. 2009; Liu et al. 2014). Moreover, phthalic acid esters are semi-volatile compounds (Mo et al. 2008) and their half-lives in soil, which relate to their structure characteristics and environment, range usually from <1 week to several months or longer in anaerobic or cold environments depending on their molecular structure characteristics and environmental conditions (Stales et al. 1997). The US Environmental Protection Agency has identified various congeners of phthalic acid esters including dimethyl phthalate, diethyl phthalate, di-*n*-butyl phthalate, dioctyl phthalate, butylbenzyl phthalate, and diethylhexyl phthalate as priority pollutants. Phthalic acid esters are endocrine-disrupting compounds, which have been shown to reduce the diversity of microbial communities and decline crop quality (Kapanen et al. 2007). Human exposure to phthalic acid esters could potentially have some bad effects on the reproductive, hepatic, and renal systems (Hauser and Calafat 2005; Swan 2008). There have been increasing concerns about uncertainties regarding phthalic acid ester exposure and the risks that phthalic acid esters may pose to human health and the environment.

In this paper, we provided information about the phthalic acid ester contamination in Chinese agricultural soils; the potential risk of phthalic acid esters on soil ecosystems, the human food chain, and human health; and the effectiveness of various techniques developed for the remediation of soils contaminated with phthalic acid ester.

2 Source of phthalic acid esters in agricultural soils

In soils, the main anthropogenic sources of phthalic acid esters originate from agricultural films (Wang et al. 2013a); agricultural chemicals, such as pesticides and fertilizers (Wang et al. 2013b; Guo and Kannan 2012); waste water irrigation and biosolids fertilization (Cai et al. 2007), and industrial emissions (Weschler et al. 2008; Zhu et al. 2010) which was one of the phthalic acid ester sources but not the main route for agricultural soil. Phthalic acid esters from these sources are further distributed in the environment through various biogeochemical cycling processes supported by the soil (Fig. 3). Besides degradation by microorganisms and uptake by plants, the phthalic acid esters in soil can also enter to the atmosphere through evaporation and migrate into the groundwater and surface water by rain, etc.

2.1 Agricultural plastic films and greenhouse

Greenhouse vegetable production in China is rapidly expanding, and the area covered by agricultural film has become the largest in the world. In contrast to the huge economic benefits, greenhouse cultivation has the disadvantage of phthalic acid ester pollution because phthalic acid esters are the major components of plastic films used for greenhouse and soil mulching cover. The concentrations of dibutyl phthalate and diethylhexyl phthalate in plastic greenhouse soils, for example, were 2.5–3 times higher than that of corresponding soils not covered by plastic greenhouses (Wang et al. 2002, 2011). In addition, Kong et al. (2012) found that phthalic acid ester concentrations in film-covered soils were 74 and 208 % higher than those for farmland and vegetable soils where no film was used. Since phthalic acid esters are only physically bound to the plastic structure, they can be

Fig. 2 Commonly used agricultural plastic products in China. The *left photo* is a plastic greenhouse and the *right one* is about the mulch film used on crop land. Since phthalic acid esters are only physically bound into the plastic products, they can be readily released from the greenhouse and mulch film by heat or solvents, thereby resulting in their accumulation in soil

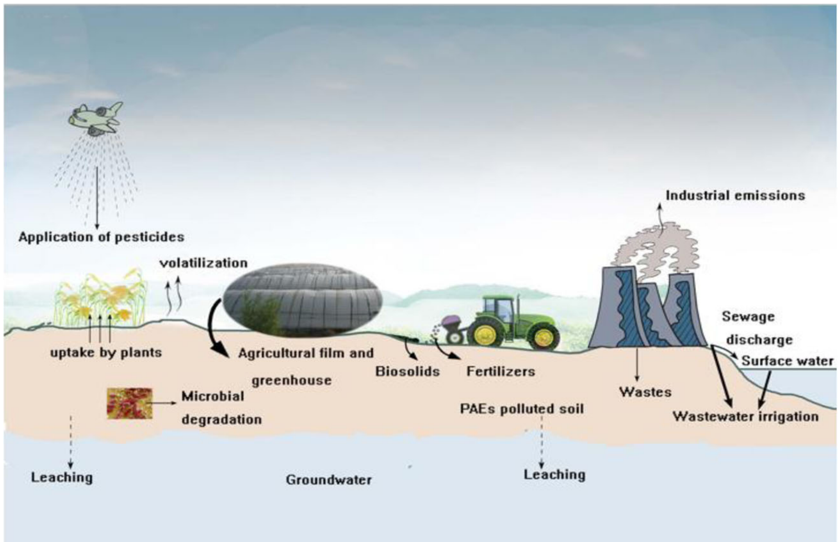

Fig. 3 Sources of phthalic acid esters (PAEs) in agricultural soils and their environmental fate. The application of agricultural film and greenhouse, biosolids, agricultural chemicals (fertilizers, pesticides, etc.), and wastewater irrigation will lead to the phthalic acid ester contamination in soil. The industrial emissions including sewage and solid waste to soils will also contaminate the soil with phthalic acid esters if an industry emits phthalic acid esters. Phthalic acid esters in soil can be degraded by microorganisms, absorbed by plants, volatilized to the atmosphere, and/or leached to the groundwater, which will result in further contamination of water, air, food, etc.

leached from plastic into the environment (Li et al. 2004). Therefore, the usage of shed and mulching films would result in high concentrations of phthalic acid esters in atmosphere and soils. The pollution level of phthalic acid esters caused by greenhouse and mulching films is influenced by greenhouse characteristics, film usage rates, and durability.

Chen et al. (2011) showed that soil applied with black plastic mulch for long periods contained elevated phthalic acid ester levels. They speculated that black plastic mulch can absorb heat more readily than light-colored plastic mulch, resulting in the elevation of the temperature of the plastic. This would decrease the bonding strength between phthalic acid esters and polyvinyl chloride and thus release phthalic acid esters into the environment. The thicker film has already been reported to increase levels of diethylhexyl phthalate in soil and vegetables, whereby thicker films can emit more diethylhexyl phthalate than thin ones (Fu and Du 2011). In addition, the volume of air in a greenhouse was inversely proportional to the concentrations of diethylhexyl phthalate in the greenhouse atmosphere (Yu et al. 2012). Since the concentration of diethylhexyl phthalate in the atmosphere correlates to its concentration in vegetables, the vegetables grown in low greenhouses were able to absorb more diethylhexyl phthalate from the atmosphere than those grown in tall greenhouses. Furthermore, irrespectively of dimensions, new plastics emit more phthalic acid esters than aged ones, thus causing phthalic acid ester uptake in vegetables to decrease with greenhouse and mulch age (Fu and Du, 2011) even though phthalic acid esters did accumulate in the soil (Chen et al. 2011).

Besides the usage of plastic films, the disposal methods of spent plastic films were also highly correlated with the regional variation of the soil phthalic acid ester concentrations in China (Gao and Zhou 2013). The durability of the main types of plastic films currently used in China is low, such as polyvinyl chloride and polyethylene, so they are readily broken and difficult to reuse. Not only does this increase film usage rate but also the amount of film that is spent on the soil. This spent film can destroy soil aggregates, which in turn will decrease soil aeration and water permeability. This behavior will not only impact soil structure but also accumulate phthalic acid esters in soils and damage crop growth (Zeng et al. 2013b). Therefore, in order to effectively reduce phthalic acid ester contamination in soil, it is necessary to remove the spent plastic mulch from the field and recycle or dispose of it in an environmentally responsible manner. In addition, the plastic film used for agriculture should be thin, light in color, and durable so that less phthalic acid esters will be released into the environment. Development of suitable plastic films that do not contain phthalic acid esters would further reduce phthalic acid ester contamination of the soil. Currently, some films without phthalic acid esters are available, such as polyethylene or polytetrafluoroethylene, but the short service life or high cost makes them difficult for large scale-applications in agriculture. Therefore, there is a need to develop new types of film that have a long service life, are non-toxic, and more environmentally friendly. This would prevent further contamination and would protect the ecological environment and human health (Zeng et al. 2013a).

2.2 Wastewater irrigation

Wastewater reuse in agriculture is a widespread practice in developing countries, especially in urban areas where there are water shortage and poverty (Kunhikrishnan et al. 2014). Wastewater streams provide an important source of water and nutrients to improve crop production. For example, irrigation of sewage effluent yielded larger winter crops than irrigation with well water, because sewage also supplied nitrogen, phosphorus, potassium, and organic carbon to the soil (Singh et al. 2012). However, land application of untreated/treated wastewaters containing phthalic acid esters may over time result in the accumulation of phthalic acid esters in the receiving soils (Table 1). Irrigation with wastewater may not only result in soil contamination through accumulation but also affect the food quality and food safety through the uptake of contaminants by crops (Calderón-Preciado et al. 2011). Therefore, it is important that irrigation systems with treated wastewater are designed with the risk of phthalic acid ester pollution in mind. Alternatively, advanced pretreatments or the adoption of water-saving irrigation technologies are strategies that would decrease the risk of phthalic acid ester polluting soils.

2.3 Municipal biosolids

Application of municipal biosolids, or treated sewage sludge, to agricultural land can supply nutrients and organic matter to soils (Zuloaga et al. 2012). For example, in the city of Buenos Aires, about 29–45 % of biosolids carbon was still present 1 year after application, indicating the slow-release nature of biosolids-applied nutrients and organic matter (Torri and Alberti 2012). This will save the need to buy chemical fertilizers resulting in cost saving and preventing excessive nutrient leaching from the soil caused by the application of chemical fertilizers. However, phthalic acid esters also tend to concentrate in biosolids because they have a low solubility in wastewater (Abad et al. 2005). Application of municipal biosolids to agricultural land could thus lead to phthalic acid ester accumulation in the soil and phthalic acid ester uptake in plants (Table 1). This pathway would cause phthalic acid esters to enter into the human food chain, with a potential risk for human health (Grøn et al. 2001). Consequently in recent years, the application of biosolids to agricultural land and the phthalic acid ester concentrations in biosolids have received more attention. The phthalic acid ester concentration in biosolids from 11 cities in China ranged from 10 to 114 mg kg^{-1} (Mo et al. 2001). In Taiwanese biosolids, the mean concentration of dibutyl phthalate, diethylhexyl phthalate, and butylbenzyl phthalate was 718, 41, and 8 mg kg^{-1} dry weight, respectively (Ma and Lin 2011). When a biosolids containing 37 mg kg^{-1} of dibutyl phthalate and 116 mg kg^{-1} of diethylhexyl phthalate were land applied at a rate of 5 ton ha^{-1}, the concentrations in harvested barley were two and five times higher than barley grown in control soils without sludge addition (Kirchmann and Tengsved 1991). The European Commission has suggested that for agricultural use, the diethylhexyl phthalate concentration in sewage sludge should be limited to 100 mg kg^{-1} dry weight (Bagó et al. 2005). Although some researchers showed that the application of biosolids did not increase the phthalic acid ester concentrations in soil (Rhind et al. 2002; Petersen et al. 2003), the effect of biosolids application on soil concentrations cannot be neglected, because it may involve risks with other potentially harmful concentrations such as pathogens and heavy metals. Therefore, the effect of biosolids will need to be investigated further, and biosolids application rates will need to be controlled in order to manage potential pollution risks.

2.4 Application of agricultural chemicals

Fertilizers and pesticides are commonly used in modern agriculture to enhance agricultural production and protect crops. The widespread use of pesticides in modern agriculture is of

Table 1 Selected references describing the soil accumulation of phthalic acid esters (PAEs) applied with wastewater and biosolids

Location	Origin of PAEs	Soil response to wastewater or biosolids	Reference
Kanpur, India	Tannery effluent	Wastewater-irrigated soil revealed the presence of PAEs	Masood and Malik (2013); Zubair Alam et al. (2010)
Taiyuan, China	Sewage and industrial wastewater	Soil showed elevated PAE concentration	Du et al. (2010)
Guangzhou, China	Sewage and industrial wastewater	Long-term application of wastewater irrigation and sludge application, diethylhexyl phthalate greatly exceeded the recommended allowable soil PAE concentrations	Zeng et al. (2008)
Beijing, China	Municipal biosolids	The concentrations of diethylhexyl phthalate in 91 % biosolids samples met the limit (100 mg kg^{-1}, dry weight) proposed by the Europe Union	Cai et al. (2007)
Guangzhou, China	Municipal biosolids	Application of biosolids resulted in higher soil concentrations of PAEs	Cai et al. (2008a)
Glil Yam, Israel	Municipal effluents	Relatively high levels of PAEs were detected in soil	Muszkat et al. (1993)

increasing concern because, among others, they contain phthalic acid esters (Johnsen et al. 2001). These phthalic acid esters do not only leach from packaging into fertilizers and pesticides, they are also used as a solvent in many pesticides (Wang et al. 2013b; Mo et al. 2005). Application of large quantities of fertilizers and pesticides in agriculture may therefore lead to the accumulation of phthalic acid esters in soil (Liu et al. 2010a; Zeng et al. 2013a; Yang et al. 2013).

In China, 22 widely used fertilizers were found to contain phthalic acid esters ranging from 1.2 to 2795 μg kg^{-1} (Mo et al. 2008). The phthalic acid ester concentration in soils was found to increase with the level of fertilizer input (Zorníková et al. 2011). Cai et al. (2003) showed that the total phthalic acid ester concentration in *Ipomoea aquatica* shoots was higher when it had been grown in potted paddy soils fertilized with chemical fertilizers than when it had been grown without fertilizers. Furthermore, vegetable and orchard fields contained higher levels of phthalic acid esters than rice and cotton fields. This difference in phthalic acid ester levels may be attributed to a more intense fertilizer regime in the vegetable and orchard fields compared to the rice and cotton fields (Cai et al. 2006; Liu et al. 2010a). Phthalic acid ester uptake in plants was found to be positively correlated with phthalic acid ester concentrations in soil (Yin et al. 2003). These findings indicated that the potential of phthalic acid ester pollution needs to be considered when these agriculture chemicals are applied to soils.

3 Distribution of phthalic acid esters in soil

There are various pathways in which phthalic acid esters can be redistributed in the soil. These pathways are related to transverse, vertical, and seasonal changes in phthalic acid ester distribution. Transverse distribution is usually negatively related to the geographic distance from a pollution source, although this may change according to properties of the phthalic acid esters, such as molecular weight or water solubility (Gómez-Hens and Aguilar Caballos 2003). The soil type, anthropogenic behavior, and land use have been shown to affect the vertical distribution through the degradation and transport of phthalic acid esters, while the rainfall and temperature are likely to affect the distribution through degradation and leaching of phthalic acid esters in the profile of soils (Vikelsøe et al. 2002; Wang et al. 2013b). Seasonal distribution in phthalic acid esters results from seasonal changes in agricultural inputs as well as temperature and rainfall (Wu et al. 2011a). However, these distributions are not independent from each other (Fig. 4). For example, the phthalic acid ester concentrations were found to be usually higher near industrial and commercial centers but occasionally they were higher at a greater distance from industrial and commercial centers due to rainfall and seasonal changes (Liu et al. 2009). Therefore, it is necessary to investigate all factors that affect phthalic acid ester distribution in soils in order to provide suitable remediation pathways for phthalic acid ester-polluted soils.

3.1 Transverse distribution of phthalic acid esters in soil

The transverse distribution of phthalic acid esters is usually site specific and generally inversely proportional to the distance from industrial and commercial sources (Zeng et al. 2008, 2009). For example, the phthalic acid ester concentrations in urban soils were found to be higher than those in rural soils, which was due to the intense commercial activities and greater phthalic acid ester discharge from plastic materials in urban regions than rural regions (Sun et al. 2013; Xia et al. 2011b). Furthermore, transverse phthalic acid ester distribution was also impacted by the phthalic acid ester mobility in the soil (Liu et al. 2009, 2010b); for example, elevated concentrations of diethylhexyl phthalate and dibutyl phthalate were found at a further distance rather than at the disposal center (Liu et al. 2009). This may have been caused by an increase in the water solubility of phthalic acid esters when soluble humic materials are present in the soil, which could have decreased the apparent degree of soil sorption (Gómez-Hens and Aguilar Caballos 2003). Overall, industrial and human activity in relation to transverse transport generally increased phthalic acid ester concentrations in the soil, while the phthalic acid ester mobility in relation to transverse transport generally decreased by the sorption of soluble soil humic materials.

3.2 Vertical distribution of phthalic acid esters in soil

The distribution of phthalic acid esters across the entire soil profile could be due to a range of physical, chemical, and microbiological factors as well as other factors such as the cultivation types, land use, the type of phthalic acid ester compound, and the level of phthalic acid esters in the surface soil (Wang et al. 2013b). Most of diethylhexyl phthalate and dibutyl phthalate occurred in the top 20 cm of the soil, and their concentration generally decreased with soil depth (Gao and Zhou 2013), although some anthropogenic activities, such as tillage, may have caused phthalic acid ester concentration in subsurface soils (20–40-cm depth) to be similar to that in surface soils (0–20-cm depth) (Wang et al. 2013b).

Leaching of phthalic acid esters depends on soil type, seasonal water movement through the soil, and thus local weather conditions. The movement of diethylhexyl phthalate through the soil, for example, ranged from 0.1 to 1.6 m annually depending on soil type and sampling time of the year (Vikelsøe et al. 2002; Liu et al. 2010a). Vertical distribution was also influenced by adsorption and degradation processes in the soil. The diethylhexyl phthalate, di-*n*-butyl

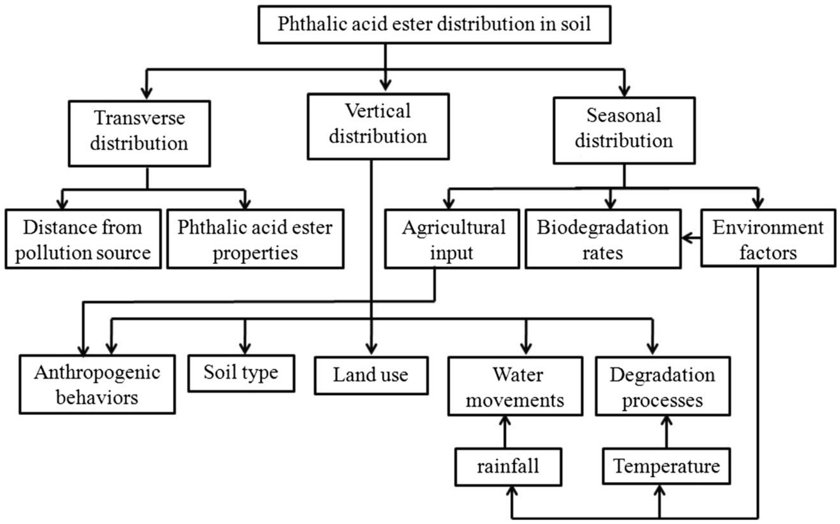

Fig. 4 Factors affecting the distribution of phthalic acid esters in soils where they can be redistributed via transverse, vertical, and seasonal changes. The phthalic acid ester concentration usually decreased with the distance from a pollution source; however, the properties of phthalic acid esters (such as molecular weight, water solubility, etc.) would affect their transverse distribution in soil. The anthropogenic behavior, soil type, and land use will change the phthalic acid ester distribution vertically. The environment factor such as rainfall and temperature can affect the degradation processes and movement of phthalic acid esters in soil, which will result in different seasonal and vertical distributions

phthalate, and di-*i*-butyl phthalate were found in deeper soil layers compared to diethyl phthalate, which was only found in the top 50 cm of soil. This difference in distribution with depth indicated that diethyl phthalate could be easier degraded in surface soil than diethylhexyl phthalate and di-*i*-butyl phthalate and therefore was not leached deep into the soil (Liu et al. 2010a). The phthalic acid ester degradation processes can further be influenced by the presence of oxygen in the soil. Under aerobic conditions, phthalic acid esters in particular diethyl phthalate, dipentyl phthalate, dibutyl phthalate, and butylbenzyl phthalate are readily degraded, but under anaerobic conditions which prevail in rice fields and deep soils, phthalic acid esters such as diethyl phthalate are not readily degraded (Yuan et al. 2002). In a 1-year-old rice field, phthalic acid ester concentrations were significantly greater than those in a long-term bean field, and the concentrations of diethylhexyl phthalate and dibutyl phthalate were significantly greater in the deeper soils compared to those in shallow soils (Wang et al. 2013b). Overall, the vertical distribution of phthalic acid ester levels depended upon soil cultivation, soil adsorption, and type of degradation processes that occurred.

3.3 Seasonal variation

The level of phthalic acid esters in soil was found to be higher and compositions more complicated in winter than in summer. This was due to either less agricultural input or greater biodegradation rates in summer (Liu et al. 2010a). Wu et al. (2011a) found that the dibutyl phthalate degradation rate increased rapidly as temperature increased from 25 to 30 °C. Therefore, lower soil temperatures are likely to increase the persistence of phthalic acid esters in soil. The seasonal variation in rainfall is also likely to have an impact on phthalic acid ester leaching. In the rainy season, water leaching through the soil will transport phthalic acid esters down the soil profile and into the groundwater (Liu et al. 2010a). Overall, variations in temperature and rainfall will be the major cause of the seasonal variability in phthalic acid ester concentration in the soils.

4 Status of phthalic acid ester-contaminated soil in China

4.1 Accumulation of phthalic acid esters in soil

Owing to the rapid industrial emissions and the application of shed and mulching film, biosolids, manures, composts, pesticides, fertilizers, and wastewater irrigation, phthalic acid esters have accumulated in soils across China (Table 2). In particular, dibutyl phthalate and diethylhexyl, the most dominant phthalic acid ester compounds found in soils, are listed in Table 2.

The most serious soil pollution documented in Table 2 occurred in Lianxing, a center for vegetable production in Guangdong Province. The high phthalic acid ester concentrations in those soils were likely caused by its proximity to a main traffic trunk, the scale of the operation, and the irrigation of wastewater containing phthalic acid esters (Cai et al. 2005). The second most polluted soil was found in Shouguang, a town in Shandong Province, which is also a prominent vegetable cultivation area in China. The excessive use of agricultural chemicals and plastic film has lead to considerable accumulation of phthalic acid esters like diethylhexyl

Table 2 Concentration of dibutyl phthalate (DBP) and diethylhexyl phthalate (DEHP) in soils

Location	Source	DBP (mg kg^{-1})	DEHP (mg kg^{-1})	References
Harbin (Heilongjiang)	Greenhouse field (black soil)	2.8–14.6	0.4–4.2	Xu et al. (2008)
Handan (Hebei)	Greenhouse field (fluvoaquic soil)	3.2–29.4	1.2–8.0	Xu et al. (2008)
Beijing	Urban soil	3.8	6.5	Li et al. (2006b)
Shouguang (Shandong)	Vegetable greenhouse	2.3–20.5	1.9–25.2	Wang (2007)
Jinan (Shandong)	Proximity to plastic industries	2.6–4.6	3.0–5.2	Meng et al. (1996)
	Proximity to vegetable greenhouse	0.7–3.6	0.6–3.5	
Hangzhou (Zhejiang)	Vegetable greenhouse	0.1–0.4	0.8–2.2	Chen et al. (2011)
Taizhou (Zhejiang)	Proximity to e-waste recycling site	0.4–5.1	0.2–14.1	Liu et al. (2009)
Dongguan (Guangdong)	Vegetable soil	0.3–0.9	0.3–4.0	Zhang et al. (2009)
Lianxing (Guangdong)	Vegetable soil	17.2–20.6	12.8–19.7	Cai et al. (2005)
US Environmental Protection Agency guidelines	–	0.08	0.44	Liu et al. (2009)

phthalate and dibutyl phthalate in those soils. The production of both diethylhexyl phthalate and dibutyl phthalate represents a high percentage of the whole phthalic acid ester production (Xu et al. 2008). In addition, the abundance of diethylhexyl phthalate may be explained by its low water solubility, which decreased its migration rate (Ejlertsson et al. 1997) and bioavailability as the result of being adsorbed on the sediments (Katayama et al. 2010). All of these factors contributed to long retention times and elevated phthalic acid ester concentrations in the soil. The phthalic acid ester concentrations in soils from test sites in most regions of China were far greater than the recommended values in soil cleanup guidelines used by the US Environmental Protection Agency (Table 2). Contamination by phthalic acid ester compounds has already generated considerable public concern and should no longer be neglected in China. In order to select effective soil remediation methods for phthalic acid ester pollution, it is necessary to understand the main causes of phthalic acid ester pollution that include the application of agricultural chemicals, utilization of film for vegetable production, wastewater irrigation, and proximity to the industrial pollution source (e.g., plastics industries). All of these could lead to serious phthalic acid ester pollution in soils, in particular in soils that grow vegetable produce.

4.2 Effect and accumulation of phthalic acid esters in vegetables

According to the statistics of United Nation's Food and Agriculture Organization (FAO), in 2008, 43 % of the land area in China was sown and used for production of vegetables. The annual vegetable production in China was 565 million tons. This accounted for 49 % of the worldwide production in 2007 and created huge economic benefits for China (Zhang 2007). However, not only the quantity but also the quality of vegetables is particularly important. Both quantity and quality can be affected by phthalic acid esters in the soils. Phthalic acid esters have been shown to inhibit the root length, shoot length, and biomass (fresh weight) of rapes (*Brassica chinensis* L.) after treatment with dibutyl phthalate (Ma et al. 2013). In addition, Chinese cabbage (*Brassica rapa* var. *chinensis*) leaves showed a white discoloring due to chlorosis and necrosis after exposure to 30 mg L^{-1} of dibutyl phthalate for 42 days. Also, as a result of dibutyl phthalate exposure, etiolation occurred on the whole leaf of the Chinese cabbage (Liao et al. 2009). The dibutyl phthalate was found to affect the proteome formation as well as the physiology and the morphology of Chinese cabbage during growth. A decrease in biomass and the accumulation of dibutyl phthalate in Chinese cabbage indicated that plants were able to absorb dibutyl phthalate from the soil via the roots (Liao et al. 2006).

In *Capsicum*, the dibutyl phthalate concentration was negatively correlated with vitamin C and capsaicin content while the dibutyl phthalate concentration in the shoots was positively correlated to that in the roots. This suggested that the dibutyl phthalate uptake by *Capsicum* was responsible for a decrease in the *Capsicum* quality (Yin et al. 2003). Similarly, lab and field experiments that applied diethylhexyl phthalate and dibutyl phthalate to other vegetables also showed a negative correlation between diethylhexyl phthalate or dibutyl phthalate concentration and the vegetable quality. The vegetables in order of the most to the least sensitive to diethylhexyl phthalate and dibutyl phthalate were as follows: cauliflower> spinach>radish>chili pepper (An et al. 1999; Yin et al. 2003).

The accumulation of phthalic acid esters in vegetables listed in Table 3 varied with vegetable species, and the diethylhexyl phthalate concentration in wax gourd was significantly higher than that in other vegetables (Table 3). Wang et al. (2010b) showed that plants with greater lipid content were able to accumulate more diethylhexyl phthalate than other plants. This could explain why wax gourd was more effective at accumulating phthalic acid esters than the other

Table 3 Concentrations of phthalic acid esters (PAEs) such as dibutyl phthalate (DBP) and diethylhexyl phthalate (DEHP) in the edible part of vegetables

Vegetable type	Type of PAEs	Concentration (mg kg^{-1})	Location	Reference
Wax gourd	DEHP	560.0–1480.0 (dry weight)	Hangzhou	Fang (2009)
Potherb mustard	DEHP	34.0±2.5 (dry weight)	Hangzhou	Fu and Du (2011)
Lettuce	Sum of 6 PAEs identified by the US Environmental Protection Agency	6.9 (dry weight)	Dongguan	Zhang et al. (2009)
Rape	DEHP	43.2 (dry weight)	Hangzhou	Wang et al. (2010b)
Rape	DEHP	2.3–6.3 (wet weight)	Taizhou	Liu et al. (2010b)
Cucumber	DEHP	0.4–0.7 (wet weight)	Shouguang	Wang (2007)
Cucumber	DBP	0.2 (dry weight)	Dongguan	Zhang et al. (2009)
Green onion	DEHP	0.5 (dry weight)	Guangdong	Li et al. (2010)
Chinese cabbage	DEHP	0.3–3.2 (dry weight)	Guangdong	Li et al. (2010)
Tolerable daily intake	DEHP	37 μg kg^{-1} body weight day^{-1}	European Union	
			Scientific Committee	CSTEE (1998)
Reference dose	DEHP	20 μg kg^{-1} body weight day^{-1}	US Environmental Protection Agency	Koch et al. (2003)

vegetables (Fang 2009), and maize was the least effective at accumulating phthalic acid esters (Wang 2009). These results suggest that some plants like maize are more suitable for planting in phthalic acid ester-polluted fields than others like wax gourd.

Besides the type of vegetable, the accumulation level in vegetables is also influenced by the location and properties of phthalic acid esters. To be specific, vegetables planted near industrial districts and vegetables grown in greenhouses or mulch film usually contained higher levels of phthalic acid esters than others. The diethylhexyl phthalate concentration of rape in Table 3 proved that the proximity to an industrial area manufacturing plastics (Wang et al. 2010a) or e-waste recycle sites (Liu et al. 2010b) caused elevated phthalic acid ester level in the soils. Some phthalic acid esters were absorbed into the plants through the roots and then distributed throughout the plant. For example, dibutyl phthalate, which is less lipophilic and more water soluble than diethylhexyl phthalate, migrated more readily from the roots to the stems and leaves than the more lipophilic diethylhexyl phthalate (Chiou et al. 2001). The small molecular weight makes dibutyl phthalate more likely to flow out from the soil and pollute the environment than diethylhexyl phthalate (Xu et al. 2008). The translocation of diethylhexyl phthalate from the roots to the shoots was minimal even though Fang (2009) showed that the diethylhexyl phthalate concentration in wax gourd was positively correlated with that in soil. Any diethylhexyl phthalate located in the above-ground biomass of a plant could therefore have been derived from the atmosphere. Besides this, the low water solubility of diethylhexyl phthalate contributes to its persistence in the soil (Ejlertsson et al. 1997) and therefore its higher accumulation in vegetables than other phthalic acid esters.

Human health risk assessments have indicated that the consumption of phthalic acid ester-contaminated vegetables presents a particularly high exposure risk for all ages of the population (Kong et al. 2012). Based on an average vegetable consumption of 0.5 kg day^{-1} per adult, the consumption of most vegetables listed in Table 3 would exceed the acceptable daily intake of phthalic acid esters compared to the tolerable daily intake (37 μg kg^{-1}) recommended by the European Union Scientific Committee (CSTEE 1998) and the reference dose (20 μg kg^{-1}) recommended by the US Environmental Protection Agency (Koch et al. 2003). It is suggested that vegetables grown in contaminated soils could be considered as potentially contaminated (Li et al. 2006a). Therefore, to minimize phthalic acid ester accumulation in vegetables, the use of film and agricultural chemicals containing phthalic acid esters should be avoided and vegetables should not be grown in industrial areas. In addition, only vegetables that accumulate phthalic acid esters weakly should be grown in phthalic acid ester-polluted soils. However, more information is needed about phthalic acid ester uptake in vegetables to determine the actual level of phthalic acid esters that humans are exposed to through their diet.

5 Ecological effect and human risk of phthalic acid esters

5.1 Effect of phthalic acid esters on soil ecosystems

Soil microbial communities play a crucial role in nutrient cycling, maintenance of soil structure, detoxification of noxious chemicals, and control of plant pests (Elsgaard et al. 2001, Filip 2002). Upon soil exposure to phthalic acid esters, however, there are some environmental impacts on soil health. For example, Kapanen et al. (2007) investigated the influence of diethyl phthalate esters on the soil environment at concentrations of 0.01, 0.1, 1, 10, and 100 g kg^{-1}. At concentrations greater than 1 g kg^{-1}, diethyl phthalate reduced the diversity of a microbial community to only ten major species and reduced culturable bacteria numbers in a heterotrophic plate count by 47 % and pseudomonad species by 62 % within 1 day of exposure. At those concentrations, diethyl phthalate accumulated in hydrophobic regions of the microbial cytoplasmic membrane and disrupted the membrane fluidity (Cartwright et al. 2000). Furthermore, there is evidence that phthalic acid esters decrease the microbial metabolic activity by reducing soil basal respiration and catalase activity (Guo et al. 2010; Xie et al. 2009). Besides the effect of phthalic acid esters on microbial communities, in contaminated soils, phthalic acid esters also affected urease, phosphatase, catalase enzyme activities, and soil invertebrates such as earthworms (Chen et al. 2004). After long-term exposure to four phthalic acid esters at different concentrations (1, 10, and 100 μg g^{-1} soil), the phthalic acid esters in increasing order of toxicity for soil microbes and enzymes were diethylhexyl phthalate<dioctyl phthalate<dimethyl phthalate<dibutyl phthalate (Chen et al. 2013).

Overall, the diversity and activity of soil microbial communities are important indicators of soil quality (Schloter et al. 2003). The toxicity of phthalic acid esters to microorganisms could impact on soil quality and may affect the crop yield and quality.

5.2 Effect of phthalic acid esters on human health

Phthalic acid esters have been classified as priority pollutants and endocrine-disrupting compounds by the US Environmental Protection Agency, the State Environmental Protection Administration (SEPA) of China, and the European Commission (Keith and Telliard 1979). Humans are exposed to phthalic acid esters from food that may be contaminated during the crop's growth, processing, storage and packaging, as well as exposure to air (Kavlock et al. 2002). Phthalic acid esters have also been detected in human urine and milk samples (Brock et al. 2002; Zhu et al. 2006). This is a cause for concern because phthalic acid esters could accumulate in the human body and cause potential health threats to an exposed individual and their subsequent progeny. The maximum exposure limit and tolerable daily intake are proposed in Table 3. Beyond this tolerable dosage, there will be a risk of mutagenic action on the reproductive system and the embryonic development, and anti-androgenic effects in humans (Swan 2008) by reducing fetal testicular testosterone production (Howdeshell et al. 2008). Diethylhexyl phthalate and dibutyl phthalate have hepatic and renal effects at high doses and cause hepatocellular carcinoma, anovulation, and decreased fetal growth (Hauser and Calafat, 2005). There are also some effects in relation to phthalate exposure on respiratory function, metabolism, and thyroid function of humans (Hoppin et al. 2004; Stahlhut et al. 2007; Meeker et al. 2007).

6 Remedial techniques for phthalic acid ester-polluted soils

From the above discussion, it is clear that the phthalic acid esters in soil will not only have negative effects on soil properties but also accumulate in food, causing human exposure to phthalic acid esters. Therefore, phthalic acid esters may pose a major risk to ecosystems and human health. In order to avoid the threat, it will be important to reduce and eliminate phthalic acid ester emission sources and remedy the phthalic acid ester pollution that is already present in the environment. A range of remedial techniques including physical, chemical, and biological treatments will be introduced in detail in the following sections.

6.1 Removal by microbial degradation

Soil microorganisms have been reported to degrade and mineralize phthalic acid esters. The degradation rates decrease with increasing molecular weights of the phthalic acid esters (Wang et al. 2000; O'Grady et al. 1985; Chang et al. 2007). Some of the phthalic acid esters with relatively high molecular weights, such as diethylhexyl phthalate, dioctyl phthalate, and didecyl phthalate (DDP), are considered recalcitrant because the low water solubility of these phthalic acid esters could limit degradation (Ejlertsson et al. 1997). Remediation technologies often use aspects of microbial degradation to remove phthalic acid esters from the environment (Table 4).

The phthalic acid ester degradation may be enhanced through inoculation of soil with selective isolates of bacterial strains that are efficient in degradation (Chao and Cheng 2007; Prasad and Suresh 2012). Wu et al. (2011b) reported that the addition of pure bacterial strains, *Gordonia* spp. for example, did reduce the negative effect of dibutyl phthalate on indigenous soil bacteria, thereby reducing the time required to completely degrade dibutyl phthalate in the soil from 14 to 9 days. By co-inoculation of the bacteria *Bacillus* sp. and *Gordona* sp. with the fungus *Acaulospora laevis*, the degradation of diethylhexyl phthalate in the soil was enhanced more than when those isolates were inoculated alone. This indicated a synergistic effect on diethylhexyl phthalate degradation when the three strains were inoculated together (Qin et al. 2008).

An alternative to in situ remediation was the use of a relatively new removal technology that uses bioslurry reactors. Bioslurry reactors utilize a liquid slurry environment to

Table 4 Selected references describing the degradation rate of dibutyl phthalate (DBP), diethyl phthalate (DEP), dimethyl (DMP), and diethylhexyl phthalate (DEHP) in soils

Degradation methods	Degradation bacterial strains or amendments in soil	Type of phthalic acid esters	Remediation effects	Reference
Bacterial strains	*Gordonia* spp.	DBP	Reduce the degradation time to from 14 to 9 days	Wu et al. (2011b)
Bacterial strains	*Agrobacterium* spp.	DBP	Degradation half-life was about 10.4 h (<200 mg L^{-1})	Wu et al. (2011a)
Bacterial strains	*Variovorax* spp.	DMP DEP DBP	>99 % of 300 mg L^{-1} within 30 h	Prasad and Suresh (2012)
Co-inoculation	*Bacillus* sp., *Gordona* sp., and the fungus *Acaulospora laevis*	DEHP	Remediation effect better than isolate bacterial strain inoculated alone	Qin et al. (2008)
Bioslurry reactors	Sludge	DEHP	99 % removal in 49 days	de Moura Carrara et al. (2011)
Bioslurry reactor	Isolated bacteria of *P. aeruginosa* and effluent treatment plant microflora	DEHP	More than 90 % degradation was observed within 12 days	Shailaja et al. (2008)
Bioreactor	Sludge	DBP	Decrease the degradation half-lives from 1.4 to 1.1 days	Yuan et al. (2011)
		DEHP	Decrease the degradation half-lives from 6.9 to 5.0 days	
Bioreactor	Chunghsing compost	DBP	Decrease the degradation half-lives from 2.4 to 1.1 days	Chang et al. (2009)
		DEHP	Decrease the degradation half-lives from 5.0 to 3.9 days	
Bioreactor	Sludge and bacterial strain	DEHP	Decrease the degradation half-lives from 8.6 to 4.2 days	Chang et al. (2007)

degrade hazardous organic compounds present in solid, liquid, or sorbed forms to non-toxic simple end products such as carbon dioxide and water (Mohan et al. 2004). Bioslurry reactors are highly efficient in degrading phthalic acid esters likely due to their ability to control culturing conditions and to provide nutrients that support microbial growth (Di Gennaro et al. 2005; Yuan et al. 2011). Furthermore, in the presence of compost, sludge, and bacterial strains, the degradation rate of phthalic acid esters was further enhanced (Table 4) when particle sizes of the amendments were smaller (Semple et al. 2001; Chang et al. 2009; Yuan et al. 2011).

Microbial degradation is considered to be one of the major routes of phthalic acid ester removal from the environment. A number of investigations have successfully demonstrated in situ microbial degradation for a range of phthalic acid esters under aerobic and abiotic conditions in soil, natural waters, and wastewaters (He et al. 2012). However, microorganisms do not completely degrade and remove phthalic acid esters from soil or aqueous solution (Zhang et al. 2007). Furthermore, for a bioslurry reactor, soils would need to be excavated, which limits the remediation capacity and the practical application of this technique. Nevertheless, in some cases, excavation and treatment of soil in bioslurry reactors may be advantageous.

6.2 Removal by phytoremediation

Phytoremediation is defined as the use of plants to remove or degrade polluted substrates. Phytoremediation and phytostabilization techniques can reduce or eliminate contaminants from the environment and reduce human exposure to contaminants (Cunningham et al. 1995; Bolan et al. 2011). Previous studies have shown that certain plant species can enhance the dissipation of soil organic contaminants. For example, 96 % of 2,4,6-trinitrotoluene (TNT) was removed from soil by cultivating maize (*Zea mays* L.) (Van Dillewijn et al. 2007), and 87 % of total petroleum hydrocarbons (TPHs) were removed from soil with *Avicennia schaueriana* (Moreira et al. 2013). The concentration of phthalic acid esters in soil was reduced by 87 % when alfalfa (*Medicago sativa* L.) was cultivated in monoculture. Alfalfa cultivation in combination with *Eupanacra splendens* was able to remove 91 % of phthalic acid esters and alfalfa intercropped with *E. splendens*, and *Sedum plumbizincicola* was able to remove 89 % of phthalic acid esters from soil (Ma et al. 2012a). Therefore, combined plant cultivations should be considered to improve remedial efficiencies when considering phytoremediation of contaminated soils.

Another phytoremediation technique is rhizoremediation. This is a microbe-assisted phytoremediation. These rhizosphere microbes can occur naturally or can be encouraged by introducing specific microbes into the rhizosphere (Gerhardt et al. 2009). Introduction of specific mycorrhiza can significantly decrease levels of soil pollution (Teng et al. 2011). For example, Xu et al. (2010) have demonstrated that alfalfa inoculated with *Rhizobium* decreased polychlorinated biphenyl concentration in the soil by 43 % compared to 36 % when in the absence of inoculation.

Not much is known about the phytoremediation of phthalic acid esters besides the work carried out by Ma et al. (2012a, b) and Cai et al. (2008b). In order to understand the complex interaction between soil, microorganisms, and roots with regard to the fate of organic pollutants such as phthalic acid esters, further research work is required about the phytoremediation of phthalic acid esters covering the effects of plant species, intercropping method, and rhizoremediation with some bacteria on phthalic acid ester degradation.

Overall, plant species and their growth requirements are important to consider when designing phytoremediation strategies for contaminated soils and sediments (Wang and Chi, 2012; Robinson et al. 2009). Phytoremediation is relatively inexpensive and is an environmentally friendly approach. However, the plant growth may be inhibited under high concentrations of contaminants (Harvey et al. 2002), which will limit the remediation effects. Plants used for phytoremediation are generally species with a small biomass and little economic value. Furthermore, once contaminants are transferred from the soil to the plant, the contaminant levels in the plant need to be evaluated and an environmentally safe use for the removed plant crops will need to be found.

6.3 Reduction of bioavailability by adsorption

Adsorption of organic compounds by soil organic matter is a key mechanism that controls their transport, environmental fate, and bioavailability in soils (Smernik 2009; Park et al. 2011). The adsorption process can be employed as a useful control technique that aims to reduce mobility and therefore bioavailability of pollutants in soils or as a technique that aims to remove pollutants such as phthalic acid esters from the aqueous environment. Materials known to adsorb phthalic acid esters include activated carbon, chitosan, β-cyclodextrin, and various types of organic matter (Julinová and Slavík, 2012); while these adsorbents are not commonly used in soils than in aqueous environment, only Xia et al. (2011a) found that biochar application can enhance phthalic acid ester adsorption in soils. However, these adsorbents were already proven to be effective. The adsorption of polycyclic aromatic hydrocarbons onto pure charcoal was about 10–1000 times stronger than the adsorption onto organic carbon in soils and sediments (Accardi-Dey and Gschwend 2002). Soils amended with biochar were also found to be particularly effective at enhancing soil adsorption of organic contaminants and thus reduce the bioavailability, leaching risk, and plant uptake of those contaminants (Kookana et al. 2011; Sheng et al. 2005; Wang et al. 2010a; Zhang et al. 2010; Zhang et al. 2012; Sopeña et al. 2012). Therefore, biochar and other types of organic matter are useful materials to consider when designing phthalic acid ester immobilization technologies for the reduction of phthalic acid ester bioavailability (Zhang et al. 2013).

7 Conclusions

Phthalic acid ester pollution in agricultural and urban soils is widespread in China through the use of plastic greenhouses, plastic film mulching, land application of agricultural chemicals, wastewater and biosolids, as well as the use of plastics in general modern-day living. The presence of phthalic acid esters in soils is affecting the soil quality because phthalic acid ester pollution affects microbial activity, microbial diversity, enzyme activity (e.g., urease, phosphatase, catalase), and soil invertebrates such as earthworms, as well as the yield and quality of agricultural crops. The current level of research is not sufficient to understand all mechanisms and implications of the widespread phthalic acid ester distribution in soils. It is therefore important to improve the understanding of pollution pathways for phthalic acid esters in soils and the connection between the original pollution source and their environmental fate. In association with better knowledge about pollution pathways, new technologies need to be developed to remove or immobilize phthalic acid esters in soils. Finally, for food and environmental safety reasons, it is important to develop land use-specific phthalic acid ester pollution control standards for agricultural products. These standards can be based on the US Environmental Protection Agency or European Union standards but need to be adapted to Chinese conditions. Therefore, establishing a management policy specific to Chinese conditions will help establish reliable monitoring methods and evaluation criteria for the remediation of phthalic acid ester pollution in Chinese soils. This will ultimately help to protect the environment.

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