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Atmospheric monitoring of organochlorine pesticides across some West African countries

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Abstract Most African countries have ratified the Stockholm Convention on persistent organic pollutants (POPs) and are expected to reduce emissions of POPs such as organochlorine pesticides (OCPs) to the atmosphere. Emerging evidence, however, suggests that there are contemporary sources of OCPs in African countries despite the global ban on these products. This study investigated the atmospheric contamination from OCPs in four West African countries—Togo, Benin, Nigeria, and Cameroon—to ascertain the emission levels of OCPs and the characteristic signatures of contamination. Polyurethane foam (PUF) disk passive air samplers (PAS) were deployed in each country for ca. 55 days in 2012 and analyzed for 25 OCPs. Hexachlorocyclohexanes (HCHs) and DDTs constituted the highest burden of atmospheric OCPs in the target countries, at average concentrations of 441 pg m^{-3} (range 23–2718) and 403 pg m^{-3} (range 91–1880), respectively. Mirex had the lowest concentration, ranged between 0.1 and 3.3 pg m^{-3}. The concentration of OCPs in rainy season was higher than in dry season in Cameroon, and presupposed inputs from agriculture during the rainy season. The concentrations of ∑25 OCPs in each country were in the following order: Cameroon > Nigeria > Benin > Togo. There was significant evidence, based on chemical signatures of the contamination that DDT, aldrin, chlordane, and endosulfan were recently applied at certain sites in the respective countries.

Keywords Persistent organic pollutants (POPs) · Organochlorine pesticides (OCPs) · Passive air sampling · West Africa

Introduction

Some persistent organic pollutants (POPs) have been banned from their manufacture, import, and application in the Stockholm Convention, which came into force in May 2004. POPs are defined as persistent, bioaccumulative, toxic chemicals that are prone to long-range transport (LRT). This

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group of chemicals includes organochlorine pesticides (OCPs), such as dichlorodiphenyltrichloroethane (DDT) and hexachlorocyclohexanes (HCHs). Due to their physical-chemical properties and persistence, POPs are distributed throughout the globe and are often detected in remote regions where they were not or have never been used, such as in the Polar Regions.

Environmental contaminations from OCPs are reportedly high in regions with previous heavy applications of these chemicals. For instance, Pozo et al. (2006) reported high concentration of HCH in China due to the past heavy use of HCH in this region. They also reported a wide range of endosulfan concentrations throughout the globe, with high concentrations occurring in tropic regions because it was then in current use in many tropical countries. Time-trend analysis provided evidence that environmental contamination from a particular OCP declines when application is reduced (Jaward et al. 2004). This was clearly demonstrated for HCB, which were consumed largely in Europe in the past, ~200 t per year, but its level in the environment reportedly declined because of reduction of its usage (Jaward et al. 2004).

In the African region, contamination from OCPs has been sparsely documented through some limited measurements in air as part of national, regional, or global monitoring (Harner et al., 2006; Pozo et al. 2006; Batterman et al. 2008; Pozo et al. 2009; Shunthirasingham et al. 2010; Hogarh et al. 2014). Historically, OCPs were widely used as insecticides in the cultivation of cash crops such as cocoa, coffee, oil palm, and cotton in African countries because of its cost effectiveness and broad spectrum activity (Fianko et al. 2011). Also, OCPs were applied in Africa for public health reasons. DDT is presently permitted for malaria control (in countries that have asked for exemption under the Stockholm Convention on POPs), given its effectiveness against the mosquito vector that transmits malaria. Malaria is very endemic in many African countries due to the tropic humid climate and bad hygienic environment. In 2012, the World Health Organization (WHO) estimated malaria prevalence rate of approximately 207 million cases per year, resulting in 627,000 malaria deaths, largely in developing countries and most especially among the rural poor (WHO, 2013). The magnitude of the malaria problem has compelled the restricted application of DDT as a control measure in depressed situations. It is, however, of concern that DDT meant for malaria control possibly could end up in agricultural applications, especially where there is a lax in pesticide control and management.

There is significant threat to human health when pesticides are misapplied and mismanaged. The International Labor Organization (ILO) estimated that there were between two and five million occupational cases of pesticide poisoning a year, with 40,000 fatalities (ILO 1994). In Africa, where a lot of people engage in agriculture, the risk of exposure to toxic pesticides is potentially high because farmers handle and apply pesticides without the necessary precaution and protective measures (Williamson et al. 2008). Over 50,000 metric tons of obsolete pesticides litter the African landscape (Dollimore and Schimpf 2013; World Bank 2013). Nearly every African country grabbles with the problem of obsolete pesticides. Inventories conducted so far suggested that obsolete pesticides range between 50 and 500 metric tons in several countries in Africa including Ghana, Cote d'Ivoire, Togo, Benin, Mali, Liberia, Sierra Leone, Burkina Faso, Cameroon, Central African Republic, Kenya, Zambia, Botswana, and South Africa. Tunisia, Congo, and Sudan were each estimated to have obsolete pesticides within the range of 500–1000 metric tons, while Morocco, Western Sahara, Ethiopia, and Tanzania were each estimated to possess over 1000 metric tons of obsolete and unwanted pesticides (Bernstorff and Stairs 2000). Under the Africa Stockpile Program, about 3310 tons of these obsolete chemicals have been removed (World Bank 2013). Thus, over 45,000 tons of obsolete pesticides have yet to be retrieved. This constitutes a pool of unwanted legacy of pesticides, which potentially could be accessed and used illicitly in farming. It is indeed a matter of concern that banned OCP products are still accessible in some developing countries (Adu-Kumi et al. 2012; NPASP 2012; Manaca et al. 2013). This has necessitated an urgent call for an international concerted effort for the management and control of POPs pesticides (Weber et al. 2013; Weber et al. 2015).

The present study investigated the atmospheric contamination from OCPs in four West African countries—Togo, Benin, Nigeria, and Cameroon. We ascertained the emission levels of OCPs, characterized the chemical signatures of contamination and the distributions of OCPs across the four countries. The differences of OCP occurrence among the countries were also resolved.

Methodology

Sample collection

In this study, the sampling was conducted by deploying polyurethane foam (PUF) disk passive air samplers (PAS). The PUF disk PAS were similar to those used in Hogarh et al. (2012). PUF disks were first pre-cleaned by washing in water and Soxhlet extracted with acetone for 12 h at the National Institute for Agro-Environmental Sciences (NIAES), Tsukuba, Japan. They were then dried under vacuum, wrapped securely in aluminum foil, and then sealed in airtight zipper bags and stored in the dark at room temperature at NIAES, until shipment to West Africa for sampling. Sampling was undertaken in four Western African countries, Togo, Benin, Nigeria, and Cameroon. There were five sites each in Togo, Benin, and Nigeria and sampling was conducted at these sites in the rainy season. In Cameroon,

however, samples were collected from seven sites during both dry and rainy seasons (Fig. 1). The PAS were deployed for eight continuous weeks at each site. Unfortunately, some PAS were lost, one in Benin and two in Togo. At the end of deployment, the PUF disks were harvested in airtight package with adequate protection from photo-exposure and returned to NIAES for chemical analysis.

Analyses of samples

Chemical analyses were carried out completely following the method used in Hogarh et al. (2014). Briefly, the harvested PUFs were Soxhlet extracted with acetone for approximately 12 h (exceeding 50 cycles of circulation). Each extract was concentrated, treated in cleanup applying three different columns—celite, florisil, and graphite—and then analyzed for 25 OCPs using high-resolution gas chromatography/high-resolution mass spectrometry (HRGC/HRMS) (Autospec Ultima-Micromass). The targeted OCPs included hexachlorobenzene; α-, β-, γ-, and σ-HCHs; heptachlor; heptachlor epoxide; *cis*- and *trans*-chlordane; *cis*- and *trans*-nonachlor; oxychlordane; *p,p'*- and *o,p'*-DDT; *p,p'*- and *o,p'*-DDD; *p,p'*- and *o,p'*-DDE; aldrin; dieldrin; endrin; mirex; endosulfan I; endosulfan II; and endosulfan sulfate. Average recoveries for the various OCPs ranged mostly between 70 and 150 %. Peaks were integrated when signals were at least three times greater than background noise. The limit of detection (LOD) and limit of quantification (LOQ) for each compound as estimated from laboratory blanks are provided in the supplementary information (Table S1). When the PUF disks are deployed in the sampling chambers, they give typical sampling rates of ca. 3–4 m^3 air per day (Jaward et al. 2004). In this study, conversion from nanograms/sample to picograms per square meter in air was conducted by using duration of deployment and sampling rate of 3.5 m^3 per day.

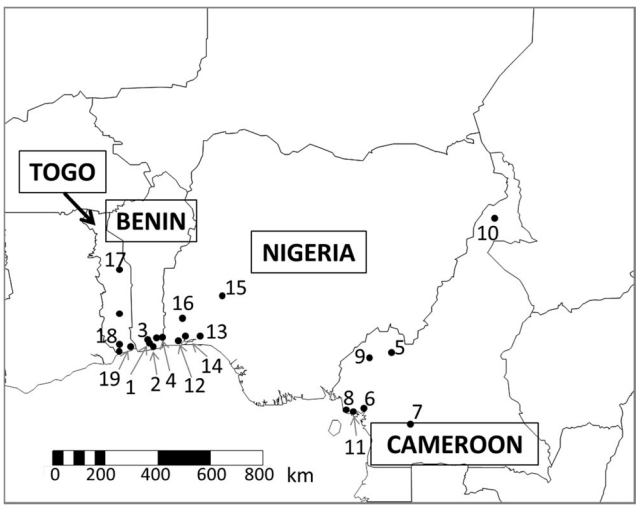

Fig. 1 Map of West Africa and sampling sites. Sampling site names and site numbers in the map in parentheses. Benin: Gbedjromede (*1*), Agblangandan (*2*), Abormey-Calavi (*3*), Porto-Novo (*4*). Cameroon: Yaounde (*4*), Kumbo (*5*), Douala (*6*), Yaounde (*7*), Limbe (*8*), Bamenda (*9*), Maroua (*10*), Buea (*11*). Nigeria: Ojota (*12*), Epe (*13*), Ikorodu (*14*), Ota (*15*), Abeokuta (*16*). Togo: Sokode (*17*), Tsevie (*18*), Vogan (*19*)

Result and discussion

The highest concentrations of ∑25 OCPs were detected in Cameroon, especially in rainy season. There are clear rainy and dry seasons in African countries, and agriculture is mainly operated in rainy season (Table S2). The levels of ∑25 OCPs in each country were in the following order: Cameroon > Nigeria > Benin > Togo. Benin and Togo showed similar compositions of OCPs. This suggests similarities in OCP issues in Togo and Benin, two relatively small countries (in terms of land coverage) bordering each other and with very similar characteristics of socio-cultural and agricultural practices. The details of each OCP are discussed individually in the following sections.

Hexachlorobenzene

Hexachlorobenzene (HCB) is a fungicide introduced in 1945 for seed treatment, particularly against bunt, which affects cereals such as wheat and rye (Batterman et al. 2008). It is also a by-product of the manufacture of industrial chemicals and a known impurity in several pesticide formulations (Batterman et al. 2008). HCB were detected at all sampling sites, the highest concentrations were seen in Cameroon (Table S2). Kumbo, where large farms were located, and Douala, whose economy was the largest in Cameroon, showed similar high concentrations in rainy season, 100 and 97.8 pg m^{-3}, respectively. The mean concentrations of HCB in the rainy and dry seasons in Cameroon were, respectively, 78.3 and 47.0 pg m^{-3}. The difference in HCB concentrations between the two seasons was statistically significant ($p = 0.01$) and pointed to a greater emission of HCB in the rainy season. We assumed that the greater content of HCB in the rainy season emanated from agricultural sources, considering that agriculture is mainly rain-fed in these countries. These concentrations are certainly far above background levels and consistent with evidence of recent local usage reported by Garrison et al. (2014) during the monitoring of POPs in Saharan dust air masses in West Africa.

Hexachlorocyclohexanes (α-, β-, γ-, and δ-HCH)

HCH isomers were detected at all sampling sites, with air concentrations ranging from 23.3 to 2720 pg m^{-3}. γ-HCH contributed most among the four HCH isomers (56–95 % of HCHs, i.e., 18.3–2440 pg m^{-3}) (Fig. 2, Table S2). Levels of ∑HCHs in Cameroon were much higher in rainy season than in dry season (rainy season; 144–2720 pg m^{-3}, dry season; 202–442 pg m^{-3}). The highest concentration was detected in

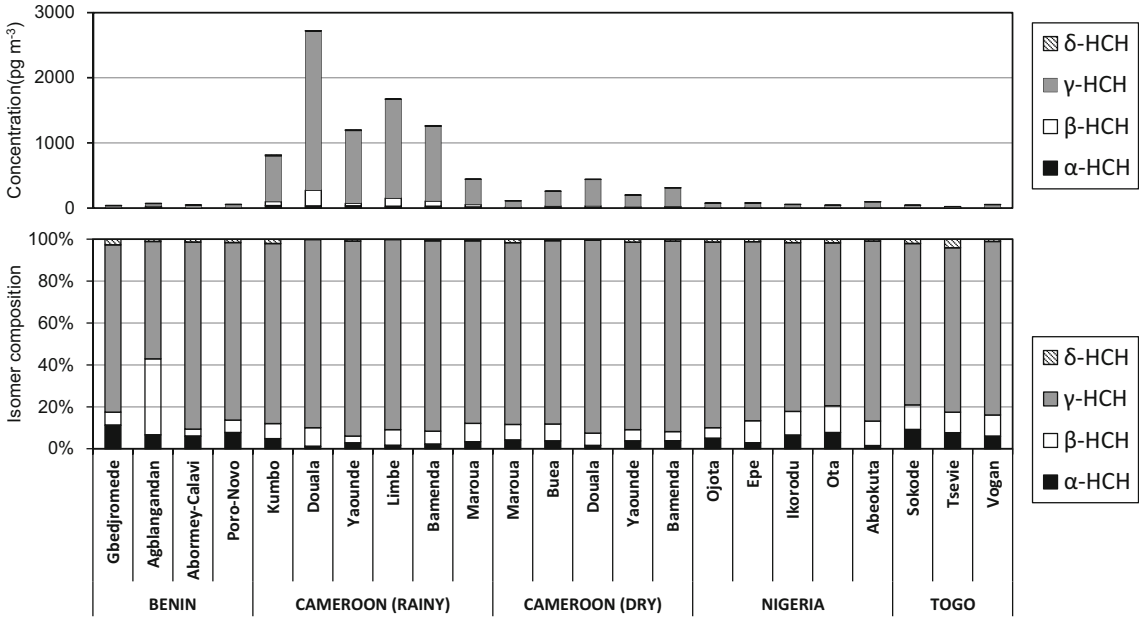

Fig. 2 Atmospheric concentration (*upper*) and isomer profile (*lower*) of HCHs

Douala, whose population was the second largest in Cameroon and economy, the largest. HCHs had been used worldwide as an insecticide since the 1950s. Technical HCH, containing about 60–70 % α-HCH, 5–12 % β-HCH, and 10–15 % γ-HCH, and five other minor isomers, was banned in North America in the 1970s, and in most countries including South Africa some years later. However, applications of technical HCH still continued in a few other countries (Batterman et al. 2008). China and Soviet Union continued to be large consumers of technical HCH until these products were banned in the mid-1980s and 1990s, respectively (Harner et al. 2004). γ-HCH is the main component of lindane, which replaced technical HCH. The relative abundance of the two HCH isomers is often expressed by the α/γ ratio. The α/γ ratio in the technical mixture is around 4–7 (Pozo et al. 2011). The α/γ ratio was in the range of 0.01–0.14 in this study, indicating fresh input of lindane. According to the percentage of γ-HCH observed in this study, farmers in the West African countries must have been using lindane rather than technical HCH (Fig. 2). Use of pure γ-HCH, lindane, continued until quite recently, mainly as a seed dressing for corn and canola; however, most countries phased out γ-HCH by 2004 (Batterman et al. 2008). HCHs can be also emitted from hazardous waste dumps, cable scrapping, and incineration in smelting plants because of the previous use of HCHs as additives in plasticizer production (Bozlaker et al. 2009).

Chlordanes (*trans*- and *cis*-chlordanes; *trans*- and *cis*-nonachlor; and oxychlordane)

Chlordanes were detected at all sampling sites (18.8–447 pg m^{-3}) (Fig. 3, Table S2). Oxychlordane was detected at a few sites with less than 1 pg m^{-3}. Technical chlordane consists mainly of *trans*-chlordane, *cis*-chlordane, and *trans*-nonachlor in the proportion of 1.00:0.77:0.62, respectively, and was used in agriculture, mainly corn, and on lawns and gardens until it was regulated in the 1970s, when its use was limited to termite control (Aigner et al. 1998; Harner et al. 2004;). The ratio *trans*-/*cis*-chlordane is often used to distinguish freshly used chlordane (ratio of *trans*-/*cis*-chlordanes close to the technical value of 1.56) from an aged one. A ratio of *trans*-/*cis*-chlordane less than 1 depicts greater reactivity of *trans*-chlordane in the environment (Pozo et al. 2006). The average ratio of *trans*-/*cis*-chlordane in this study was 1.9, which meant that they were from non-degradation chlordane (Fig. 3).

Heptachlors (heptachlor and heptachlor epoxide)

Heptachlor is an insecticide used for controlling soil insects, termites, grasshoppers, and malaria-carrying mosquitoes (Pozo et al. 2011). Both heptachlor and heptachlor epoxide were detected at all the sampling sites, with air concentrations ranging from 1.4 to 37.4, and 0.5 to 4.9 pg m^{-3}, respectively (Table S2). Heptachlors were detected in relatively high concentrations especially at Bamenda in Cameroon, where big farms were located. Heptachlor concentrations in this study were lower than India, 56–240 and 10–177 pg m^{-3} in two studies (Pozo et al. 2009, Pozo et al. 2011), but were higher than South Africa, non-detected (< 0.1 pg m^{-3}) (Pozo et al. 2009). In Ghana, heptachlor was higher than *trans*- chlordane and they correlated with each other (Hogarh et al. 2014). Heptachlor in this study and South Africa were, however, lower than *trans*-chlordane (Karlsson et al. 2000) (Fig. 4). Technical chlordane contains 7 %

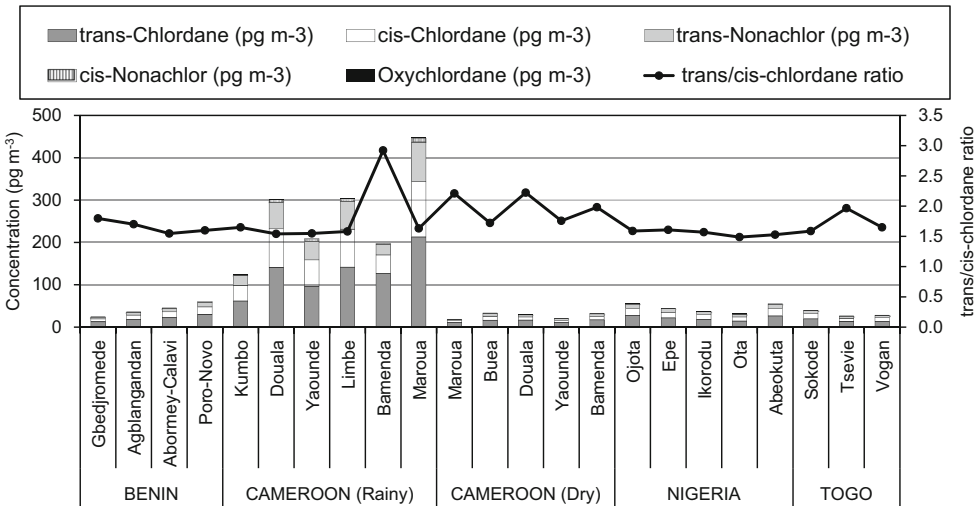

Fig. 3 Atmospheric concentration of chlordane congeners and trans/cis-chlordane ratio

heptachlor, and heptachlor also contains *trans*-chlordane as impurities. Thus, the source of heptachlor in this study as well as the South African study was probably a product of emissions as impurities from chlordane. On the other hand, the *trans*-chlordane reported in the Ghana seemed to be from impurities in heptachlor (Hogarh et al. 2014).

Dichlorodiphenyltrichloroethanes (*p,p'*-DDT, *p,p'*-DDE, *p,p'*-DDD, *o,p'*-DDT, *o,p'*-DDE, and *o,p'*-DDD)

Widely applied as an insecticide in the past, DDT continues to have limited use in the control of the malaria vector (Pozo et al., 2009). In the environment, *p,p'*-DDT (the main component of DDT) is converted to *p,p'*-DDE and DDD. DDTs were detected at all the sampling sites (91–1880 pg m^{-3}) (Table S2). The level of DDTs in Cameroon was greater in the rainy season than dry season. Maroua, the site with the highest DDT concentrations in the rainy season (1880 pg m^{-3}), is located in northern Cameroon, where cotton farms were located. In Maroua, the concentration of DDTs in rainy season was ca. six times greater than that in dry season. *p,p'*-DDE constituted the highest concentration of the DDT isomer profile at several sites (Fig. 5). Often, the relative abundance of parent and metabolite is used to distinguish relatively recent use (DDT/DDE >1) from an aged signature (DDT/DDE <1) (Pozo et al., 2011). The ratio of *p,p'*-DDT/DDE was 0.7 ± 0.4 which meant that DDTs were mainly from aged application. In Maroua, where the concentration of DDTs was high, *p,p'*-DDT/DDE ratio was 0.7 and less than 1, which meant that DDTs in Maroua was aged (Fig. 5). On the other hand, *p,p'*-DDT/DDE ratio in Kumbo was 1.7, more than 1, pointing to relatively fresh applications (Fig. 5). Thus, there was site specific variation regarding past/recent usage of DDT in West African countries, quite consistent with initial findings on DDT usage across the African region (Klánová et al., 2008). The ratio of *o,p'*-DDT/DDE was 2.9 ± 1.9 and indicated fresh applications of dicofol, whose main component is *o,p'*-DDT (Qiu et al. 2005).

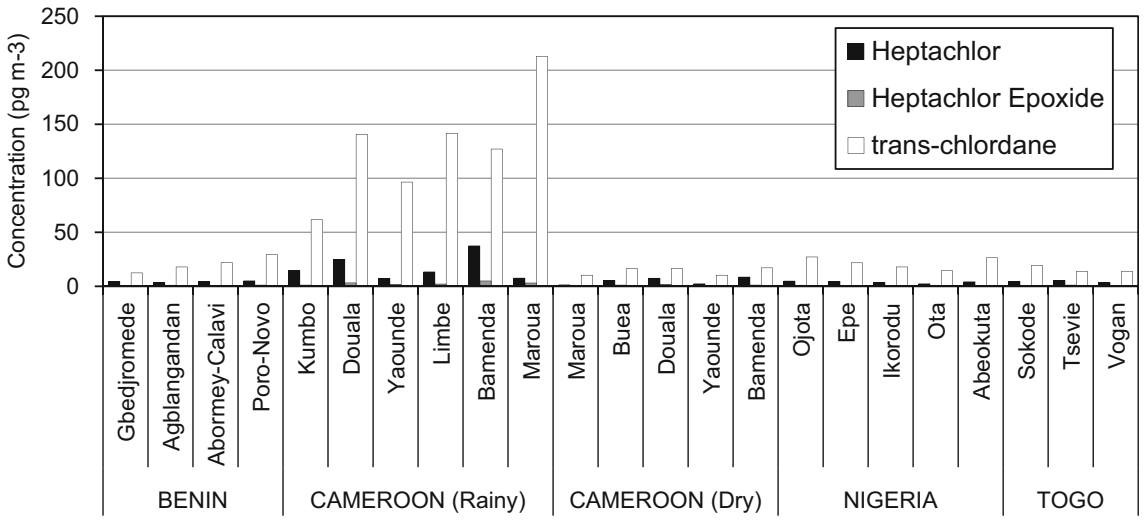

Fig. 4 Atmospheric concentration of heptachlor, heptachlor epoxide, and trans-chlordane

Drins (aldrin, dieldrin, and endrin)

The drins were detected at most of the sampling sites (38–2620 pg m^{-3}) (Table S2). Aldrin was not detected in Agblangandan (<0.19 pg m^{-3}), a residential area in Benin. With only Cameroon samples as exceptions, dieldrin concentrations were higher than those of aldrin. Aldrin was widely used to control soil insects for corn, potatoes, and other crops and to protect wooden structures from termites. It was banned in all countries decades ago (Batterman et al. 2008). Aldrin is readily converted to dieldrin in the environment. The presence of aldrin (27–85 % in drins) in Cameroon suggested ongoing local usage (Fig. 6). Ongoing application of aldrin was also reported in South Africa (Batterman et al. 2008). Especially, high concentration of drins (2620 pg m^{-3}, aldrin; 85 % of drins) was detected in Limbe in Cameroon, where one of Cameroon's leading farms was located growing many kinds of fruits such as banana, coconuts, and oil palm. Dieldrin was also used for the control of soil insects and termites, as well as wood borers, textile pests and tsetse fly (Batterman et al. 2008). Endrin is also a foliar insecticide that was used mainly on field crops such as cotton and grains, and as a rodenticide to control mice and voles. Endrin were detected at all the sampling sites with low concentrations (<10 pg m^{-3}). The levels of endrin in this study, however, were greater than those in South Africa (average 0.06 pg m^{-3}) (Batterman et al. 2008).

Mirex

Mirex concentrations were consistently low (0.1–3.3 pg m^{-3}) (Table S2). Previously reported levels of mirex in the atmosphere in South Africa where mirex had never been registered for use, were similarly low (Batterman et al. 2008; Karlsson et al. 2000). The concentration of mirex in the atmosphere in Ghana was also reportedly low, approximately 0.2 pg m^{-3} (Hogarh et al. 2014). Mirex is a stomach insecticide that has been used primarily to control fire ants in the southeastern USA, leaf cutters in South America, western harvester ants in the USA, and mealy bugs in Hawaii. It also has been used as a fire retardant in plastics, rubber, paint, paper, and electrical products (Batterman et al. 2008).

Endosulfans (endosulfan I, II, and SO_4)

Endosulfan was used globally for agriculture and also for the control of disease vectors (Pozo et al. 2011). It was only recently banned in many countries. The worldwide use of endosulfan reportedly increased over the past few decades with total use estimated to be around 150,000 t (Pozo et al. 2006). In this study, endosulfans were detected at all the sampling sites and ranged from 25.2 to 367 pg m^{-3}. The levels of endosulfans recently reported in neighboring Ghana were within this range (Hogarh et al. 2014). However, extremely high levels of endosulfans (averagely 13,100 pg m^{-3}) have been reported in neighboring Mali (Garrison et al. 2014). It is interesting that countries bordering the coastal regions in West Africa—Ghana, Togo, Benin, Nigeria, and Cameroon, had atmospheric endosulfan concentrations that were approximately two to three orders of magnitude lower than that reported in Mali, which is located closer to the Saharan desert.

Technical grade endosulfan is commercially available as a mixture typically containing >95 % of two diastereoisomers, known as endosulfan I and endosulfan II in ratio from 2:1 to 7:3 depending on the technical mixture (Weber et al. 2010). The endosulfan II isomer is more reactive and an elevated I/II ratio is representative of an aged signature, whereas ratios closer to the starting technical mixture suggest recent application (Pozo et al. 2011). The endosulfan I/II ratio ranged from 0.5 to 3.9 with geometric mean of 1.7, pointing to recent applications of endosulfan in these countries.

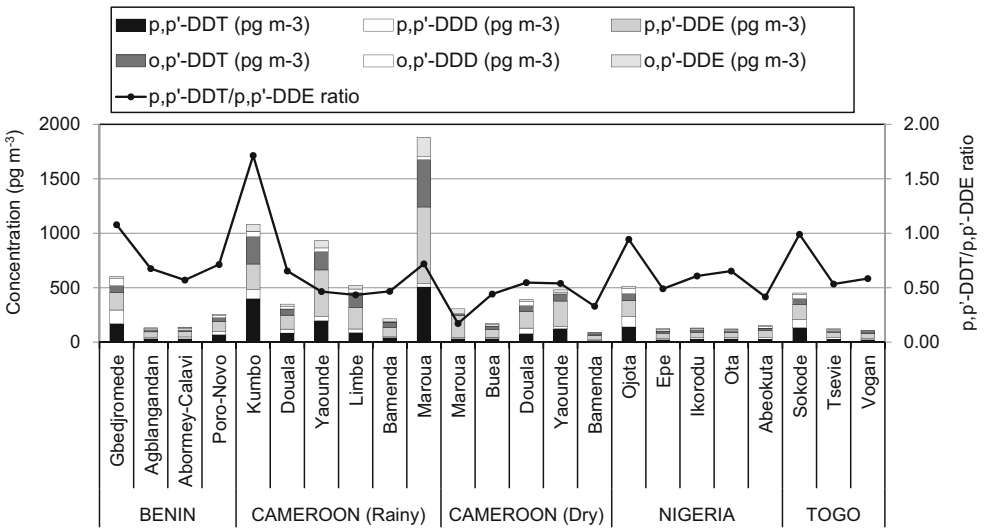

Fig. 5 Atmospheric concentration of DDTs and p,p'-DDT/p,p'-DDE ratio

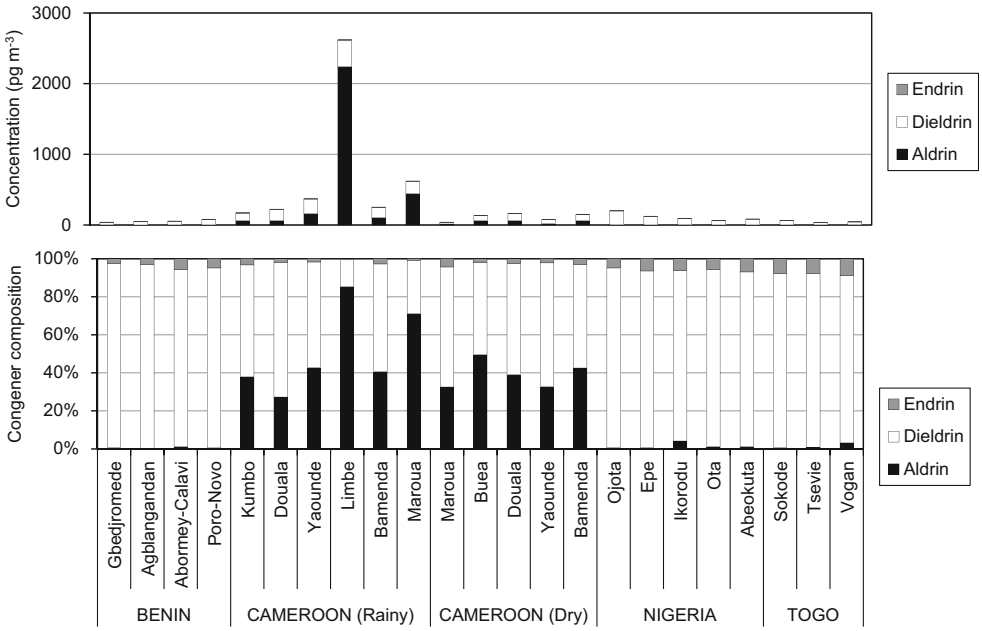

Fig. 6 Atmospheric concentration (*upper*) and congener composition (*lower*) of aldrin, dieldrin, and endrin

Policy implications

This paper presented monitoring results of persistent organochlorine compounds using polyurethane foam disk passive air samplers in four West African countries. Majority of the sites are urban with no direct point sources of OCP emissions. Only two sites—Epe in Nigeria and Tsevie in Togo—are rural and closer to farming communities. However, OCP levels at these rural sites were comparable to other sites in the respective countries. It suggests that atmospheric OCP levels are equilibrating between rural and urban areas in some West African countries. It was clear that some OCPs were or had been used and the levels of OCPs were different in each country due to the different environmental conditions (e.g., different agricultural activities or stage of urbanization). It was revealed that aldrin, for instance, was still being used at certain sites (Limbe and Maroua) in Cameroon, perhaps for agricultural reasons since the levels of this pesticide were relatively greater in the rainy season when local farmers usually plant their crops. Generally, atmospheric OCPs in Cameroon were relatively elevated in the rainy season and potentially linked to agricultural practices since agriculture in West Africa is mostly rainfed. DDT and HCH constituted the highest burden of ambient OCPs. The OCP levels and composition in Benin and Togo were quite similar. Togo and Benin are neighboring countries; they have relatively small land sizes, with similar cultural and economic characteristics. We presumed that the source factors of OCPs were similar in these two countries. The present study underscores a challenge at pesticide management in West African countries. Even though most countries in West Africa have ratified the Stockholm Convention on POPs, the evidence from this and other recent studies (NPASP 2012; Hogarh et al. 2014) suggest that contaminations from POPs such as OCPs are still rife in the sub-region. There may be two reasons to explain this phenomenon: (i) illicit accessibility to these banned products perhaps prevailed in some West African countries and (ii) continuous use of old stocks of OCP products that have not been retrieved. These should be investigated further in pursuit of lowering contaminations from OCPs in the sub-region. Given the absence of regular monitoring of OCPs in West Africa, it is difficult to establish long-term emission trends of these pollutants in the sub-region. We recommend the collection of long-term monitoring data to help ascertain the performance of individual countries at curtailing contaminations from OCPs in West Africa. This would also help clarify specific source factors of OCPs in the sub-region.

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