

Contamination of Lakes in Broknes Peninsula, East Antarctica through the Pesticides and PAHs

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Received: 22 February 2019;

Accepted: 29 March 2019;

Published online: 21 May 2019;

AJC-19417

Present study was designed to evaluate the concentration of pollutants such as polycyclic aromatic hydrocarbons and some pesticides in the lake water samples, collected from five selected locations of Broknes peninsula during 34th Indian Scientific Expedition to Antarctica (ISEA) in austral summer of 2014 to 2015. A total of 15 lake water samples were collected from the different five lakes. In these samples, 34 pesticides and 16 polycyclic aromatic hydrocarbons were estimated. Lake water samples were processed using a liquid-liquid method solvent extraction, cleaned-up and concentrated. The concentrated sample then injected into GC-MS/MS along with the standards and then quantitative analysis was performed. Pesticides residue levels found in lake water samples varied from 10.33 pg/mL to 70.00 pg/mL and polycyclic aromatic hydrocarbons levels were observed from 10.33 pg/mL to 50 pg/mL.

Keywords: Larsemann Hill, East Antarctica, Broknes peninsula, Pesticides, Polycyclic aromatic hydrocarbons.

INTRODUCTION

The Larsemann Hill (69°12'- 69°28'S, 76°00'- 76°30'E) is approximately 50 km² oasis on the Ingrid Christensen Coast of Princess Elizabeth land in East Antarctica. Hodgson *et al.* [1] stated that it is the second largest among four major ice-free areas along East Antarctica's coastline. More than 150 lakes on different islands and peninsulas are present on Larsemann Hill area which is located halfway between the Vestfold Hills (410 km²) and the Amery Ice Shelf on the south-eastern coast of Prydz Bay.

According to Bharti and Niyogi [2], Stornes and Broknes are two major peninsulas, 130 nearshore islands, and four minor peninsulas are situated on the ice-free area of Larsemann Hill. The Broknes peninsula is further divided into western and eastern components by Nella Fjord. It is one of the very few coastal areas of Antarctica that remained partially ice-free through the last glaciation and sediments deposited there contain continuous biological and paleoclimate record dating back some 130,000 years. A total of 26 million km³ of the ice sheet is present in East Antarctica, which is about 83 % of the total volume of ice in Antarctica [3].

The lakes are a significant feature of the earth's landscape which is the source of precious water and provides valuable habitats to animals and plants. Gillieson [4] stated that the lakes around the Larsemann Hills are newly formed. The melted ice gets accumulated in depression forming freshwater lakes and streams of varying sizes. Moreover, during local summer the polar ices melt and supply fresh water to the existing lakes. Sometimes the melting becomes large forming streams over the oasis regions and adds a significant quantity of water into the lakes. However due to low temperature the water is frozen and locked into the vast polar ice caps which make Antarctica the largest stock of fresh water on earth.

Some water bodies are free from ice in the summer months when their temperature increases hastily and reach +8 °C, while in the rest months water bodies are covered with 2 m of ice [5,6]. However, the impacts are not evenly spread because the footprints of human on Antarctica are small with the comparison of the total size of the continent. Legrand and Mayewski [7] stated that the human activities such as tourism, scientific work and logistic support affect the atmosphere of the polar region. The environmental condition of continent is changing rapidly due to increased human activities and the impact of climate change.

These pollutants are semi-volatile, toxic organic compounds which show resistance from chemical, photolytic and biological degradation. These pollutants are ubiquitous and globally anthropogenic in nature [8]. These compounds persist in the environment for a long time. They can be unintentionally produced from industrial activities. Due to the long-range transport mechanism, these pollutants migrate across the earth [9]. These pollutants transport in across the world through the air, water and migratory species and after traveling a long distance, they are deposited far from their original place [10,11].

Several researchers have reported the pesticides and polycyclic aromatic hydrocarbons (PAHs) in lake water [2,12], marine atmosphere/air [13-15], soil/sediment [16-19], snow [12], mosses [16,18,20], lichen [16,19,20], krill [21], penguin [22-25], skua blood [23,24]. A detailed review of the presence of persistent organic pollutants in abiotic and biotic components studied nationally and internationally [26].

In this study, we have evaluated the occurrence of pollutants in the lake water samples of Broknes peninsula, and this is one of the very few studies analyzing so many different pollutants such as pesticides and PAHs in this area. This research can make

a significant contribution to the monitoring of different pollutants in Antarctica.

EXPERIMENTAL

Study area: Broknes peninsula of Larsemann Hills, East Antarctica was selected as a sampling site for the study of contaminants. The location map of the study area is shown in Fig. 1.

Sampling: The sampling of lake water samples was carried out in the month of December-February of 2014-2015. A total of 15 samples from five different lakes were collected randomly. Before taking samples from each site, dark amber color sterile polyethylene bottles were rinsed twice with the lake water. Triplicate samples of 1.0 L each were collected from the lakes. Samples were collected from easily accessible inner areas of each lake.

All lake water samples were immediately stored in an ice chest with ice at 4 °C and transported to the laboratory after completion of the expedition for analysis of pollutants such as pesticides and PAHs. The sampling sites and sample details (Table-1) at Broknes peninsula are given in Fig. 2.



Fig. 1. Location map of the study area in Larsemann Hills (right), East Antarctica marked on the continental map of Antarctica (left)

TABLE-1
SAMPLE DETAILS WITH GEOGRAPHIC COORDINATES, DATE OF COLLECTION, SAMPLE QUANTITY, AND THE NAME OF THE LAKES FROM BROKNES PENINSULA, EAST ANTARCTICA COLLECTED DURING IN AUSTRAL SUMMER OF 2014 TO 2015

Lake name	Latitude	Longitude	Replicate	Date of sampling	Quantity of sample
P1 lake	69° 23' 49.54" S	76° 23' 17.43" E	P1-1	17-02-2015	1 L
			P1-2	17-02-2015	
			P1-3	17-02-2015	
P2 lake	69° 24' 09.005" S	76° 23' 15.00" E	P2-1	18-02-2015	1 L
			P2-2	18-02-2015	
			P2-3	18-02-2015	
P3 lake	69° 23' 32.17" S	76° 22' 17.78" E	P3-1	19-02-2015	1 L
			P3-2	19-02-2015	
			P3-3	19-02-2015	
P4 lake	69° 23' 22.19" S	76° 22' 55.82" E	P4-1	20-02-2015	1 L
			P4-2	20-02-2015	
			P4-3	20-02-2015	
Reid lake	69° 23' 8.83" S	76° 22' 42.06" E	Reid lake-1	21-02-2015	1 L
			Reid lake-2	21-02-2015	
			Reid lake-3	21-02-2015	

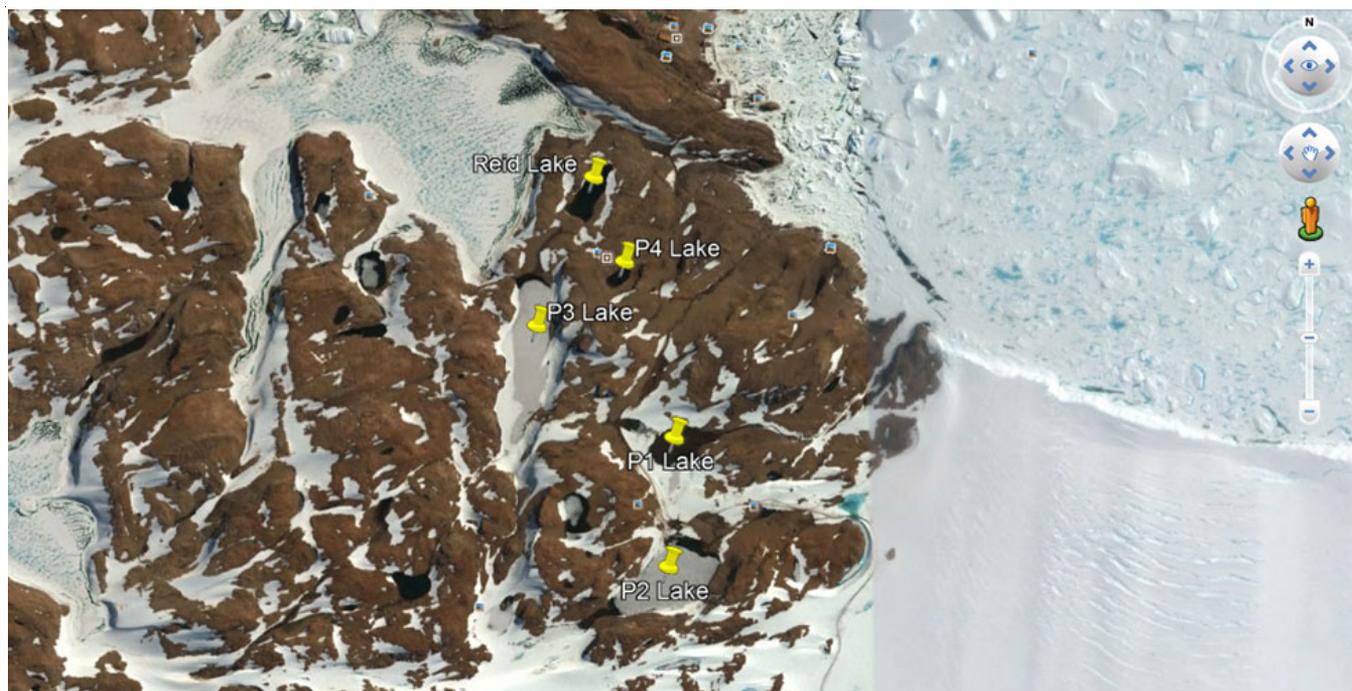


Fig. 2. Location of sampling sites mark with pinpoints on Broknes peninsula, East Antarctica

Pesticides and PAHs standards used were from Sigma-Aldrich. Methylene chloride and *n*-hexane were from Merck. Silica gel (100-200 mesh size) and anhydrous sodium sulphate were procured from Fisher Scientific. Before use, silica gel was activated at 130 °C for at least 16 h and anhydrous sodium sulphate was baked at 400 °C for 4 h.

Extraction process: Prior to extraction, lake water sample (1 L) was filtered through a Whatman No. 41 filter paper (1.0 µm). After filtration, the sample was kept at 4 °C until analysis. Pollutants were extracted from the lake water samples by the method USEPA 3510B [27]. The sample was taken into a 2000 mL separatory funnel and added 20 g sodium chloride then shakes well to dissolve it. The sample was extracted three times with a portion of 50 mL of methylene chloride and the separatory funnel was clapped for 20 min with periodic venting to release excess pressure then keep to allow the phase separation. Take the lower organic layer of methylene chloride and filter into a 250 mL of round bottom flask through Whatman No. 41 filter paper which contains 5 g of activated sodium sulphate. Then the upper aqueous layer was discarded. The total volume of collected methylene chloride was then evaporated by rotary vacuum evaporator at 45 °C upto ~ 2 mL and then it is ready for a clean-up.

Clean-up process: The final residue of the sample was cleaned up by the method USEPA 3630C [28]. Deactivated silica gel (10 g) and sodium sulphate (3 g) were transferred into a 10 mm of glass chromatographic column after that the column was preconditioned with 50 mL of methylene chloride and *n*-hexane (1:1, v/v). The final residue (2 mL) was then transferred onto the column and add 50 mL portion of hexane onto the column. The column flow was eluted into a round bottom flask (250 mL) as fraction one. Then the same process was followed for the three times and collected the elute in the same flask. All the fractions of each sample were concentrated

to dryness by using rotary evaporator at 45 °C then the sample was redissolved into 1 mL of hexane and transferred in GC sample vial for analysis of these pollutants from GC-MS/MS.

Instrumental analysis: The instrumental analysis was performed by using a GC-MS/MS system (Agilent, Model No. 7000C). The final residue of each sample (1 µL) was injected into the system. The chromatographic separation was done on an HP-5MS, 30 m capillary column with 0.25 mm internal diameter and 0.25 µm film thicknesses. Helium was the carrier gas with a constant flow of 1 mL/min and makeup gas was nitrogen. The oven temperature programme as follows: initial temperature was set at 70 °C for 2 min and ramped at 25 °C/min to 150 °C and then ramped at 3 °C/min to 200 °C and then ramped at 8 °C/min to 280 °C for 10 min. The total time for GC cycle was 41.867 min. The injector setting was a pulsed splitless mode with a temperature of 280 °C at a pressure of 27.5 psi. Purge flow to split vent was 30 mL/min at 0.75 min. The detector temperature was 280 °C and the transfer line temperature was 280 °C and the source temperature was 300 °C. The temperature of quadrupole was 180 °C for Q1 and Q2. The collision gas was nitrogen with a flow rate of 1.5 mL/min and quenching gas was helium with a flow rate of 2.25 mL/min.

Quantitative analysis: All the target compounds were quantified relative to the surrogate standards using peak areas by GC-MS/MS system. Each sample was analyzed in triplicate and the mean value was noted. For every set of the samples; one blank for quality control was processed. The sample recovery for pesticides and PAHs was found in the range of 40-100 %, which fulfilled the recovery limit of the method USEPA-1699 [29].

RESULTS AND DISCUSSION

The finding of the research indicates that the lakes of Broknes peninsula have been contaminated with traces of pollutants, where the pollutants concentration ranges varied

from 10.33 pg/mL to 70 pg/mL. This gives a clear trend of pesticides and PAHs residue level in the lake water of Broknes peninsula as illustrated in Figs. 3 and 4 with bar diagram and error bars. Pyrene and DDT are exceptionally higher in samples of P3 and P4 lakes, respectively.

Pesticides: Pesticides concentrations were found in the range of 10.33 pg/mL to 70.00 pg/mL in all five different lakes. *p,p'*-DDT was the dominant pollutant with the concentration from 20.00 ± 1.15 pg/mL to 70.00 ± 0.58 pg/mL, followed by endosulfan I 10.33 ± 0.33 pg/mL to 60.33 ± 0.88 pg/mL, malaoxon 10.33 ± 0.33 pg/mL to 50.33 ± 0.33 pg/mL, phorate sulfoxide 10.33 ± 0.33 pg/mL to 50.00 ± 1.15 pg/mL, phorate sulfone 10.33 ± 0.33 pg/mL to 50.00 ± 0.58 pg/mL, ethion 20.00 ± 0.58 pg/mL to 49.67 ± 0.88 pg/mL, alachlor 10.33 ± 0.33 pg/mL to 40.00 ± 0.58 pg/mL, malathion 10.33 ± 0.33 pg/mL to 30.33 ± 0.88 pg/mL, chlorpyrifos 10.33 ± 0.33 pg/mL to 30.00 ± 0.58 pg/mL, monocrotophos 10.67 ± 0.67 pg/mL to 30.33 ± 0.33 pg/mL, paraoxon-methyl 10.33 ± 0.33 pg/mL to 30.33 ± 0.33 pg/mL, atrazine 10.33 ± 0.33 pg/mL to 21.00 ± 0.58 pg/mL, isoproturon 10.33 ± 0.33 pg/mL to 20.67 ± 0.33 pg/mL in P1, P2, P3, P4 and Reid lake. The rest of all compounds were found below the detection limit (10.00 pg/mL) in all five different lakes (Table-2).

DDT was detected in varied concentrations while the usage of DDT was banned in several developed countries since 1972. However, it has been still in use in some countries especially to control the vectors of malaria [30,31]. Several scientists have also reported the presence of DDT in air, soil, water, sediment, snow, lichen and mosses samples from 5 to ~15000 pg/g or pg/L [16,18,32] including food chains of the biotic components of Antarctic ecosystem [26]. According to UNEP [33], production of 6300t of DDTs was done by India alone in 2003, therefore, it is not surprising to find the traces of DDTs in the Antarctic ecosystem. Due to global warming, DDT is released from stockpiles and also through migrating seabirds [34].

Corsolini and Focardi [35] studied the continental migration of persistent pollutants, which is due to the glacier melting while according to Geisz *et al.* [30], it is due to recent climate changes. Current findings of DDT in Antarctic lakes re-confirms the continental migration of pollutants. Corsolini [36] reported the concentrations of HCB and DDT in Emerald rockcod (*Trematomus bernacchi*) which was collected from Terra Nova Bay, Ross Sea and found to be decreased from the late 1980s until 1995 and then increases again in 2000-2002. The global use and the emission of DDT have declined drastically from the late 1960s [37]. Since, the nature of DDT is lipophilic and

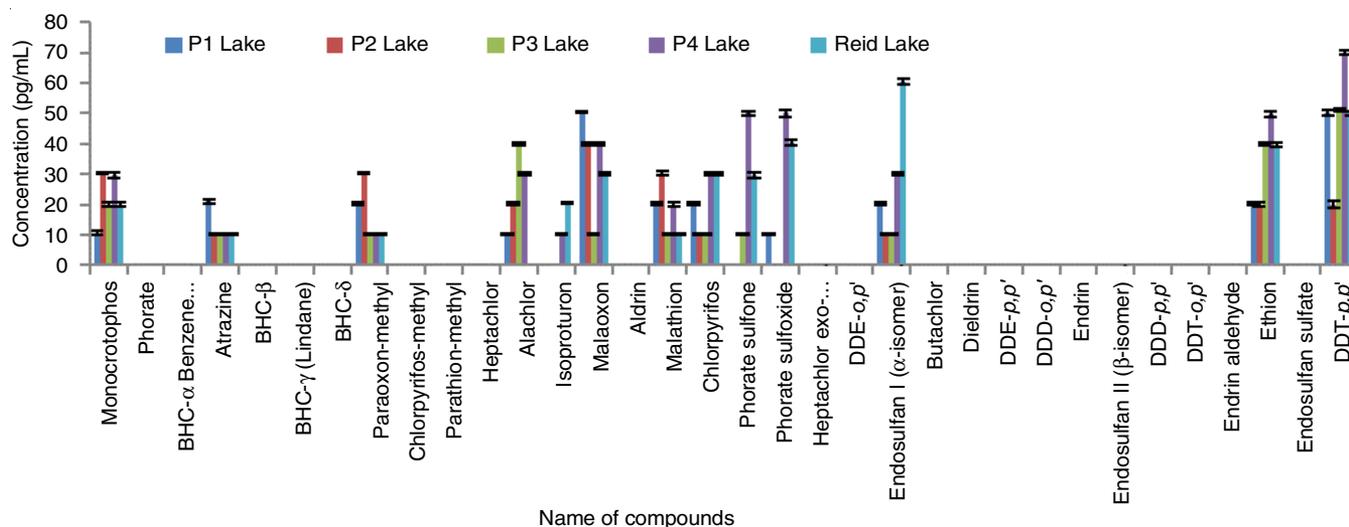


Fig. 3. Occurrence of different pesticides in lakes of Broknes peninsula

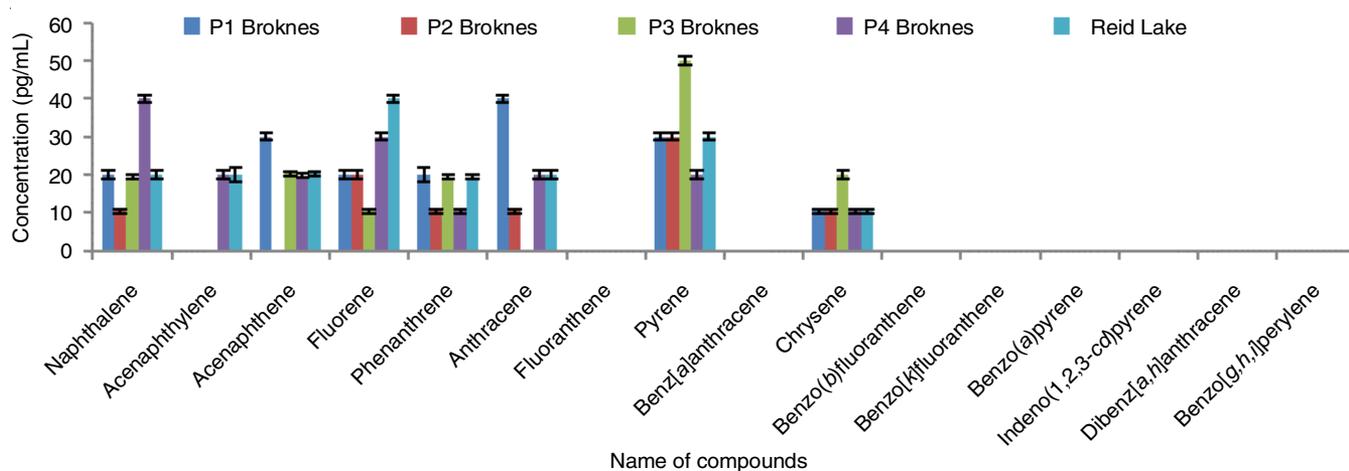


Fig. 4. Occurrence of different PAHs in lakes of Broknes peninsula

TABLE-2
CONCENTRATION OF DIFFERENT PESTICIDES IN LAKE WATER SAMPLES OF BROKNES PENINSULA

Compound name	P1 lake (pg/mL)	P2 lake (pg/mL)	P3 lake (pg/mL)	P4 lake (pg/mL)	Reid lake (pg/mL)
Monocrotophos	10.67	30.33	20.00	29.67	20.00
Phorate	< 10.00	< 10.00	< 10.00	< 10.00	< 10.00
BHC- α	< 10.00	< 10.00	< 10.00	< 10.00	< 10.00
Atrazine	21.00	10.33	10.33	10.33	10.33
BHC- β	< 10.00	< 10.00	< 10.00	< 10.00	< 10.00
BHC- γ (Lindane)	< 10.00	< 10.00	< 10.00	< 10.00	< 10.00
BHC- δ	< 10.00	< 10.00	< 10.00	< 10.00	< 10.00
Paraoxon-methyl	20.33	30.33	10.33	10.33	10.33
Chlorpyrifos-methyl	< 10.00	< 10.00	< 10.00	< 10.00	< 10.00
Parathion-methyl	< 10.00	< 10.00	< 10.00	< 10.00	< 10.00
Heptachlor	< 10.00	< 10.00	< 10.00	< 10.00	< 10.00
Alachlor	10.33	20.33	40.00	30.00	< 10.00
Isoproturon	< 10.00	< 10.00	< 10.00	10.33	20.67
Malaaxon	50.33	40.00	10.33	40.00	30.00
Aldrin	< 10.00	< 10.00	< 10.00	< 10.00	< 10.00
Malathion	20.33	30.33	10.33	20.00	10.33
Chlorpyrifos	20.33	10.33	10.33	30.00	30.00
Phorate sulfone	< 10.00	< 10.00	10.33	50.00	29.67
Phorate sulfoxide	10.33	< 10.00	< 10.00	50.00	40.33
Heptachlor exo-epoxide	< 10.00	< 10.00	< 10.00	< 10.00	< 10.00
DDE- <i>o,p'</i>	< 10.00	< 10.00	< 10.00	< 10.00	< 10.00
Endosulfan I (α -isomer)	20.33	10.33	10.33	30.00	60.33
Butachlor	< 10.00	< 10.00	< 10.00	< 10.00	< 10.00
Dieldrin	< 10.00	< 10.00	< 10.00	< 10.00	< 10.00
DDE- <i>p,p'</i>	< 10.00	< 10.00	< 10.00	< 10.00	< 10.00
DDD- <i>o,p'</i>	< 10.00	< 10.00	< 10.00	< 10.00	< 10.00
Endrin	< 10.00	< 10.00	< 10.00	< 10.00	< 10.00
Endosulfan II (β -isomer)	< 10.00	< 10.00	< 10.00	< 10.00	< 10.00
DDD- <i>p,p'</i>	< 10.00	< 10.00	< 10.00	< 10.00	< 10.00
DDT- <i>o,p'</i>	< 10.00	< 10.00	< 10.00	< 10.00	< 10.00
Endrin aldehyde	< 10.00	< 10.00	< 10.00	< 10.00	< 10.00
Ethion	20.33	20.00	40.00	49.67	39.67
Endosulfan sulfate	< 10.00	< 10.00	< 10.00	< 10.00	< 10.00
DDT- <i>p,p'</i>	50.33	20.00	51.00	70.00	50.00

BHC = Benzene hexachloride, DDD = Dichlorodiphenyldichloroethane, DDT = Dichlorodiphenyltrichloroethane, DDE = Dichlorodiphenyldichloroethylene

starts accumulating in the fatty tissue of lower organisms like krills, small fishes, *etc.* therefore DDT transfers from lower trophic level to higher trophic level of food chain [21,22,24,25, 38].

Present investigations have also reported a higher levels of endosulfan beside DDT. Apart from these two, malaaxon, phorate sulfoxide, phorate sulfone, ethion, alachlor, malathion, *etc.* have also been reported in trace amounts. The presence of isomers or metabolites of some pesticides which have been reported in Antarctica, are due to the actions of microorganisms [39,40]. α -HCH and γ -HCH are more volatile compounds than β -HCH, which leads to move in the air and travel a long distance [41]. HCH is transported by air and accumulated in higher amount in fish-eating seabirds rather than *p,p'*-DDE [42]. Goerke *et al.* [43] reported the highest concentration of *p,p'*-DDE in top predators like Southern elephant seal (*Mirounga leonina*) and Weddell seal (*Leptonychotes weddellii*). However, our findings have not reported the presence of any of the isomers of HCH and DDT in the investigated Antarctic lakes.

Like DDT, other pollutants have also reported in higher trophic level organism like HCH in resident Adelie Penguins and HCB in migrating species of snow petrel and south polar skua [19,22, 23,25,44]. HCB was the most commonly detected pollutants

in the marine sediments sample [17]. Norstrom [45] and Wania [46] have described the biomagnification and metabolized character of HCB and its presence in Antarctica.

Polycyclic aromatic hydrocarbons: PAHs concentrations were found in the range of 10.33 pg/mL to 50.00 pg/mL in all five different lakes. Pyrene was the dominant pollutant with the concentration of 20.00 ± 1.00 pg/mL to 50.00 ± 1.00 pg/mL, followed by anthracene 10.33 ± 0.58 pg/mL to 40.00 ± 1.00 pg/mL, naphthalene 10.33 ± 0.58 pg/mL to 40.00 ± 1.00 pg/mL, fluorene 10.33 ± 0.58 pg/mL to 40.00 ± 1.00 pg/mL, acenaphthene 19.67 ± 0.58 pg/mL to 30.00 ± 1.00 pg/mL, acenaphthylene 20.00 ± 1.00 pg/mL, phenanthrene 10.33 ± 0.58 pg/mL to 20.00 ± 2.00 pg/mL, chrysene 10.33 ± 0.58 pg/mL to 20.00 ± 1.00 pg/mL in P1,P2, P3, P4 and Reid lake. The rest of all compounds were found below the detection limit (10.00 pg/mL) in all five different lakes (Table-3).

In current findings, a higher level of anthracene followed by pyrene is also observed. Apart from these two, naphthalene, fluorene, fluoranthene, acenaphthene, acenaphthylene, phenanthrene, chrysene, *etc.* have been reported in trace amounts. According to Schwarzenbach *et al.* [47] lipids are measured as the major accumulation compartments in biota for lipophilic organochlorine pesticides (OCPs).

TABLE-3
CONCENTRATION OF DIFFERENT PAHs IN LAKE WATER SAMPLES OF BROKNES PENINSULA

Compound name	P1 lake (pg/mL)	P2 lake (pg/mL)	P3 lake (pg/mL)	P4 lake (pg/mL)	Reid lake (pg/mL)
Naphthalene	20.00	10.33	19.33	40.00	20.00
Acenaphthylene	< 10.00	< 10.00	< 10.00	20.00	20.00
Acenaphthene	30.00	< 10.00	20.33	19.67	20.33
Fluorene	20.00	20.00	10.33	30.00	40.00
Phenanthrene	20.00	10.33	19.33	10.33	19.33
Anthracene	40.00	10.33	< 10.00	20.00	20.00
Fluoranthene	< 10.00	< 10.00	< 10.00	< 10.00	< 10.00
Pyrene	30.00	30.00	50.00	20.00	30.00
Benz[<i>a</i>]anthracene	< 10.00	< 10.00	< 10.00	< 10.00	< 10.00
Chrysene	10.33	10.33	20.00	10.33	10.33
Benzo(<i>b</i>)fluoranthene	< 10.00	< 10.00	< 10.00	< 10.00	< 10.00
Benzo[<i>k</i>]fluoranthene	< 10.00	< 10.00	< 10.00	< 10.00	< 10.00
Benzo(<i>a</i>)pyrene	< 10.00	< 10.00	< 10.00	< 10.00	< 10.00
Indeno(1,2,3- <i>cd</i>)pyrene	< 10.00	< 10.00	< 10.00	< 10.00	< 10.00
Dibenz[<i>a,h</i>]anthracene	< 10.00	< 10.00	< 10.00	< 10.00	< 10.00
Benzo[<i>g,h,i</i>]perylene	< 10.00	< 10.00	< 10.00	< 10.00	< 10.00

The presence of lower mass PAHs and HCB in the lake water samples indicates the long-range atmospheric transport of the contamination in East Antarctica which is the most feasible cause of contamination. The presence of a high level of contaminants in the matrices which were collected from Broknnes peninsula also supports the long-range transport mechanism of contaminants to Antarctica.

Conclusion

The results of present study reported the pollutant's presence such as pesticides and polycyclic aromatic hydrocarbons (PAHs) in lakes of Broknnes peninsula, East Antarctica which confirms the presence of pollutants as reported earlier in other Antarctic regions. The application of sophisticated instrument with lower detection limit has significantly helped in the understanding of contaminants estimation from terrestrial and marine ecosystem. In different regions of the continent, the contaminants are found to be highly diverse and their patterns keep changing with climate change and fate of these pollutants. For decreasing the risk of pollutants in polar regions, more effective monitoring system is required, which can control the emission of these contaminants.

Research and tourism increase the anthropogenic activities in Antarctica, which affects its pristine ecosystem. If consider the emission sources in Antarctica, fossil fuel used by helicopter, generators, ships, flights and other transport resources is also the insignificant contributor of contaminants. The usage of plastic, personal care products, etc. if they are not monitored properly will leave the chemical contaminations in the future. Foods such as vegetables/fruits for the Antarctic researchers are also very much prone to carry the contaminant in a trace amount and might be one of the contributors of these pollutants in the Antarctic environment. Monitoring the emission of pollutants at the global level is the key to control its continental migration and to protect the pristine Antarctic ecosystem as well as to reduce the global pollutants contamination.

ACKNOWLEDGEMENTS

The authors are thankful to Director, National Centre for Polar and Ocean Research (NCPOR) & Ministry of Earth Sciences

for our participation in 34th Indian Scientific Expedition to Antarctica.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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