

Studies of mercury pollution in a lake due to a thermometer factory situated in a tourist resort: Kodaikkanal, India

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The lake was polluted by mercury from a thermometer factory.

Abstract

Kodaikkanal, India, suffered mercury contamination due to emissions and waste from a thermometer factory. Kodai Lake is situated to the north of the factory. The present study determined mercury in waters, sediment and fish samples and compared the values with those from two other lakes, Berijam and Kukkal. Total mercury (Hg_T) of 356–465 $ng\ l^{-1}$, and 50 $ng\ l^{-1}$ of mercury in methyl mercury form were seen in Kodai waters while Berijam and Kukkal waters showed significantly lower values. Kodai sediment showed 276–350 $mg/kg\ Hg_T$ with about 6% methyl mercury. Berijam and Kukkal sediments showed Hg_T of 189–226 mg/kg and 85–91 mg/kg and lower methylation at 3–4% and 2%, respectively. Hg_T in fish from Kodai lake ranged from 120 to 290 mg/kg . The results show that pollution of the lake has taken place due to mercury emissions by the factory.

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1. Introduction

Mercury is a non-essential element with no biochemical or nutritional function to bioorganisms and is ubiquitous in the environment in its presence. Its compounds are present as trace contaminants in various biological and environmental samples such as air, water, soil, animal tissues, plant matrices and coal fly ash as a result of both natural and anthropogenic activities (WHO, 1991). Being an element, mercury is persistent. It cannot be broken down in the environment, only its form can change. All forms of mercury are extremely toxic. Mercury in its methylated form as a trace contaminant in the environment is especially of great concern because of its toxicity and its special ability to bioaccumulate to levels as high

as 100,000 in marine organisms (USPHS, 1997). Despite a number of stringent regulations anthropological sources still emit quantities that result in dangerously high levels of this contaminant in the environment (Grandjean and White, 1999). Thus constant monitoring of the environment is essential.

In a recent case, the popular tourist hill-resort Kodaikkanal (about 2130 m above mean sea level), Tamil Nadu, India, has been the subject of mercury contamination originating from a thermometer making factory (Mody, 2001). Ambient levels of mercury in air of 1.32 $\mu g\ m^{-3}$ were found immediately outside the factory premises (Balarama Krishna et al., 2003), while lichen (*Parmelia sulcata*) and moss (*Funaria hygrometrica*) samples collected from different places over the hill station showed significantly higher concentrations of mercury than another similarly situated hill station in the same state of Tamil Nadu. Outside the premises of the factory, the mercury levels in the lichen and moss were 7.9 $\mu g/kg$ and 8.3 $\mu g/kg$,

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respectively. Lichen and moss samples collected from trees surrounding Berijam Lake, situated as far as 20 km from the factory in a pristine forest area showed $0.2 \mu\text{g kg}^{-1}$ (Balarama Krishna et al., 2003).

Kodai Lake which forms the heart of the resort is a star shaped lake, covering an area of about 24 hectares and is situated to the north of the thermometer factory. Mercury vapour, once released into the air is known to travel long distances leading to widespread contamination. Similarly, the factory site is at a higher elevation and drainage is also likely to have carried the mercury deposited extensively in the soil of the factory premises (URS Dames and Moore, 2002) to the lake.

The present study aims to determine the current levels of mercury in the waters, sediments and marine organisms of Kodai Lake and compare these with the values of two other lakes, Berijam and Kukkai. The contaminating mercury at the Kodaikanal site is in metallic form, however biogeochemical processes transform it into other species such as methyl mercury (Baeyens, 1992). These species differ greatly in their bio-physico-chemical properties such as toxicity, solubility and rate of bioaccumulation by organisms, etc. (USPHS, 1997; WHO, 1990). Hence, the analyses of samples only for total mercury do not provide adequate information about the impact on human health and the environment.

Methyl mercury (CH_3Hg^+) is the major organomercury species generally found in various biological samples (Ruhling and Tyler, 2004). As methyl mercury is also the most toxic as well, the values of these two species, inorganic and methyl mercury have been determined and reported in the present studies.

2. Materials and methods

2.1. Reagents

Sub-boiled hydrochloric acid and nitric acid were prepared using quartz sub-boiling distillation stills located in a class 10 laminar flow workbench. Ultra-pure water of ca. $18.2 \text{ M}\Omega\text{-cm}$ resistivity was used throughout. Stannous chloride (10%) in 10% HCl (w/v) (E Merck, Mumbai, India) and 0.5% sodium borohydride (Lancaster, England) in 1% NaOH (w/v) were used as reducing agents for reducing species of mercury to elemental form. The reducing agents were prepared fresh daily.

Glassware was pre-cleaned with 2% KMnO_4 aqueous solution followed by thorough washing and rinsing with tap water and Milli-Q water. All glassware was cleaned just before use. All Teflon ware was soaked in 20% HNO_3 , rinsed with Milli-Q water and soaked in 10% HCl followed by thorough washing and rinsing with Milli-Q water.

2.2. Instrumentation

2.2.1. CV-AAS

Various methods have been reported for the determination of inorganic mercury and methyl mercury (Puk and Weber, 1994). However, CVAAS has always been preferred because of its simplicity, relative freedom from interferences and high sensitivity. Mercury was analysed by cold vapour atomic absorption spectrometry (CV-AAS) using a mercury analyser (Model MA 5840E, Electronics Corporation of India Ltd., Hyderabad, India).

For enhanced sensitivity a gold trap was constructed by using a 0.2 g of 0.3 mm diameter gold wire, which was wound on a platinum mesh. This was inserted into a quartz tube (4 mm i.d.). Ar gas, freed from any traces of mercury impurity by passing through a second gold trap, was used to flush mercury vapours from the reaction cell onto the gold trap. Then the concentrator trap was

removed and connected to the mercury analyser. The trap was inserted into a 22-gauge nichrome wire coil which was rapidly heated to 750°C by passing high current to release the mercury for analysis by CV-AAS. A detection limit, based on three times the standard deviation of the blank, of 0.01 ng l^{-1} was obtained when a 500 ml sample was used. The system was calibrated using aqueous Hg^{2+} standards. Repeatability of the measurement was 5–8%.

2.2.2. High intensity probe sonicator

A 130 W power and 20 kHz frequency (Cole Parmer Instruments, Illinois, USA, Model: CP 130PB-1) high intensity probe sonicator equipped with a Ti probe was used for ultrasound-assisted extraction. The amplitude control of the ultrasonic processor allowed the ultrasonic vibrations at the probe to be set at the desired level in the 10–100% range of the nominal power. Polypropylene centrifuge tubes of 50 ml capacity were used for sonication experiments. After sonication, all the extracts were centrifuged at 2000 rpm for about 5 min for the rapid separation of the solid–liquid mixture.

2.2.3. Collection and processing of samples

All the samples were collected in April 2004. As shown in Fig. 1, Kodai Lake is at the heart of the Kodaikkanal township, to the north of the factory site. Sampling points are shown in Fig. 1. The sampling points were chosen almost equidistant and to cover the whole circumference of the lake. From each sampling location, one water sample and one sediment sample were collected.

Water samples were collected about 2 m from the shore to avoid various suspended objects and debris. These were collected from a row-boat using a home-made bailer that could collect waters from the desired depth. Water samples were collected from 30 cm below the surface to avoid dead leaves and other floating debris. Sediment samples were collected from the same locations as the water samples.

Water and sediment samples were also collected from two other lakes: Berijam, situated about 20 km from Kodai Lake in a protected forest, and Kukkai, another lake in pristine surroundings, with no known industrial activity, situated 40 km from Kodaikkanal, for comparison purposes. After collection the samples were stored in ice and transported to the laboratory and then stored at -20°C until further processing. Sediment samples were separated from gravel ($>2 \text{ mm}$) and were air dried at ambient temperature in a contamination-free clean room of class 100. After drying to a constant weight, the samples were ground in an agate planetary ball mill (Fritsch, Germany), sieved and particles better than 100 mesh were collected and stored in polythene bottles.

Fresh fish were harvested from the lake, dissected and the edible muscle portions were homogenized in a mixer grinder. This mass was frozen and lyophilised subsequently and used for analysis.

3. Experimental

3.1. Determination of Hg_T in water samples

The frozen water samples were thawed, acidified to 2% with sub-boiled HCl and exposed to ultraviolet radiation by passing through a 5 m long PTFE tubing (0.8 mm id.) wound over a UV lamp (8 W) at a flow rate of 1 ml/min. The sample was transferred to the reaction vessel, the reducing agent NaBH_4 added and the mercury swept onto a gold trap for collection and subsequent analysis by CV-AAS.

3.2. Determination of methyl mercury in water samples

The method used by Logar et al. (2001) was used for the extraction of the organomercurial species. Subsequent determination of mercury was carried out using gold trap CV-AAS. A 250 ml water sample was taken in a polypropylene bottle and 17.5 ml of conc. HCl and 30 ml of CH_2Cl_2 were

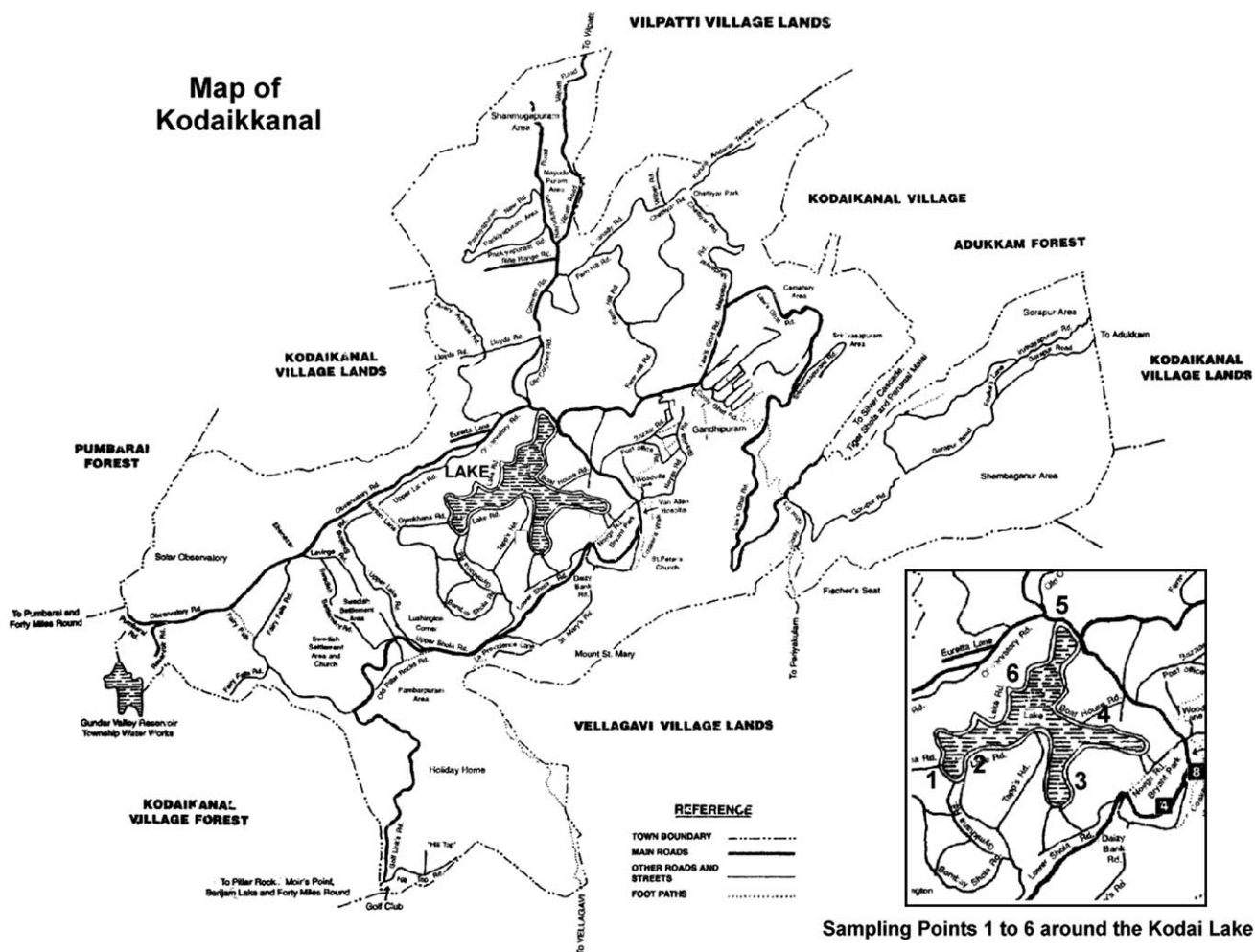


Fig. 1. Sampling points around Kodai Lake in Kodaikkanal, India.

added. The bottle was shaken on a mechanical shaker for 12 h. The aqueous layer was discarded. Fifty millilitres of Millipore water was added to the organic layer and the CH_2Cl_2 was evaporated over a water bath. Then the solution was made up to 100 ml with Milli-Q water. An aliquot of 25 ml was transferred to the reaction cell and 5 ml of 10% SnCl_2 reagent was added, mercury vapours were purged onto the gold trap and any extracted inorganic mercury was determined by CV-AAS. Another aliquot was acidified to 2% HCl, passed through the UV irradiation system to convert the methyl mercury to inorganic form, transferred to the reaction cell and the Hg_T was determined by the gold trap CV-AAS. The difference was taken as the methyl mercury.

The method was validated using spiked samples for recovery studies. The recoveries for this procedure were quantitative and therefore no correction factors were needed.

3.3. Open digestion/leaching of sediment samples for determination of Hg_T

For leaching /digestion of the sediment samples the procedure described by Akagi et al. (1995) was used. Accurately

weighed aliquots of sediment (500–1000 mg) were transferred to a 50 ml glass conical flask, 1.5 ml water, 3 ml of 1:1 HNO_3 to HClO_4 and 7.5 ml of H_2SO_4 were added and heated for 30 min at 200 °C. This was cooled and made up to the desired volume. An aliquot was analysed for total mercury by gold trap CV-AAS. Appropriate blanks were also prepared similarly and analysed.

3.4. Determination of methyl mercury in sediments

For the determination of methyl mercury in sediments, the alkali leaching–solvent extraction method developed by Liang et al. (1996) was used followed by a variation. About 0.5 g of wet sediment was taken in a 30 ml Teflon[®] bottle, 2 ml of 25% KOH–methanol were added, closed and heated to 75 °C for 3 h. Cooled, 6 ml of CH_2Cl_2 and 1.5 ml HCl were added and shaken for 10 min. The lower layer was separated and transferred to a 125 ml bottle. Another 6 ml CH_2Cl_2 portion was added to the sediment, and on separation was combined with the previous portion. Approximately 80 ml of ultra pure water was added to the bottle and heated over a water bath to remove the organic solvent. Any residual organic solvent

was removed by purging the solution with Ar gas. Now the solution was made up to 100 ml with Milli-Q water. An aliquot was transferred to the reaction cell and 10% SnCl₂ reagent was added, mercury vapours were purged onto the gold trap and any extracted inorganic mercury was determined by CV-AAS. Another aliquot was acidified to 2% HCl, passed through the UV irradiation system to convert the methyl mercury to inorganic form, transferred to the reaction cell and the Hg_T was determined by the gold trap CV-AAS. The difference was taken as the methyl mercury.

3.5. Determination of Hg_T and methyl mercury in fish

An ultrasound extraction procedure previously developed by us (Balarama Krishna et al., 2005) to rapidly extract the mercury species followed by conversion of the methyl mercury to inorganic mercury by irradiating with UV radiation was used. An accurately weighed amount (0.1–0.2 g) of sample was placed into a polypropylene centrifuge tube (50 ml volume) and 5 ml of extractant (HNO₃–thiourea) solution was added. Then the sample–extractant mixture was sonicated at room temperature. After sonication, the supernatant was separated from the solid phase by centrifugation for about 5 min at 4000 rpm. A known volume of the supernatant was then transferred to another pre-cleaned centrifuge tube. One part of the split samples was analysed for Hg²⁺ by CVAAS using SnCl₂ as reducing agent. Other part of the split sample was used for UV irradiation treatment to convert CH₃Hg⁺ to Hg²⁺, and Hg_T was analysed by CVAAS using SnCl₂/or NaBH₄ as reducing agents. Corresponding process blanks were also prepared in the same way without any sample material. Three aliquots of each sample were used for extraction procedures. With each series of extractions, a blank was also measured in parallel. This method was applied to two reference materials, NRCC-DOLT1 (dogfish liver) and IAEA 350 (fish homogenate) and their inorganic and methyl mercury contents were determined for validation.

4. Results and discussion

The present mercury concentrations of the waters, sediment and marine organisms of Kodai Lake have been determined and compared with the results of two other lakes which are situated about 20 and 40 km from Kodai Lake to obtain a snap-shot picture of the extent of prevailing contamination due to the discharge of mercury to the environment by the thermometer factory. Even though Kodaikkanal has been subject to severe mercury pollution (Mody, 2001), prior to the present investigations no detailed studies of the extent of contamination of these lakes have been reported.

The total mercury and methyl mercury values have been determined in the lake waters, sediment and bio-organisms and given in Tables 1, 2 and 3, respectively. As no data prior to the establishment of the factory in 1983, or until the detection of high levels of mercury in the environment of the township have been reported, no base line values are available. Hence the values obtained in Kodai Lake were compared against these

Table 1
Values of Hg_T, MeHg and Hg²⁺ in water samples (results are in ng Hg l⁻¹)

Sample no.	Hg _T	MeHg	Hg ²⁺ ^a
Kukkal 1	48.43 ± 7.12	ND	48.43
Kukkal 2	36.32 ± 5.23	ND	36.3
Berijam 1	110.34 ± 8.57	14.22 ± 3.52	96
Berijam 2	127.18 ± 7.55	13.16 ± 3.33	114.02
Kodai 1	374.26 ± 10.45	34.24 ± 5.81	340
Kodai 2	465.54 ± 9.65	57.36 ± 6.90	408
Kodai 3	393.42 ± 8.18	48.32 ± 5.21	345
Kodai 4	356.36 ± 8.36	45.14 ± 4.22	311
Kodai 5	402.28 ± 8.29	55.22 ± 4.45	357

^a By difference.

two other lakes, Berijam and Kukkal, to arrive at a conclusion regarding the enhancement of the mercury levels due to industrial discharge of mercury into the environment.

Once metallic mercury is released into the air, it has a very long residence time of between 0.4 and 3 years (WHO, 1991) and can travel great distances. This can account for the high mercury base line value obtained in lichen samples in the vicinity of Berijam Lake (Balarama Krishna et al., 2003). Samples collected during the current study were collected from lakes situated at progressively increasing distances from the factory site, Kodai, Berijam and Kukkal Lakes, and, as would be expected, showed decreasing concentrations of mercury. These data, however, show the distance over which the impact may be observed.

As may be seen from Table 1, Kodai Lake showed a Hg_T concentration of 356–465 ng l⁻¹ while the Berijam Lake waters showed 110–127 ng l⁻¹ and Kukkal Lake 36–48 ng l⁻¹. The value for Hg_T reported earlier for the Kodai Lake waters was <300 ng l⁻¹ (URS Dames and Moore, 2002) with no detectable methylated form of mercury.

A value of about 50 ng l⁻¹ of mercury in the form of methyl mercury is seen in Kodai Lake waters as against 14 ng l⁻¹ in Berijam and below detection limit values in Kukkal Lake. From Table 2 it may be seen that Kodai Lake sediment showed Hg_T of 276–350 µg/kg in the sediment with about 6% of methyl mercury. In comparison Berijam and Kukkal Lakes showed Hg_T levels of 189–226 µg/kg and 85–91 µg/kg, respectively. The methylation of the mercury is also lower at levels of 3–4% and about 2%, respectively.

Table 3 shows the levels of mercury determined in fish from Kodai Lake alone. Due to practical difficulties no fish from the

Table 2
Mercury in the lake sediment samples (all the values are in µg Hg kg⁻¹, dry weight)

Sample	Hg _T	MeHg
Kukkal 1	85.23 ± 5.78	1.68 ± 0.07
Kukkal 2	91.45 ± 5.96	1.27 ± 0.05
Berijam 1	226.42 ± 10.80	7.38 ± 0.17
Berijam 2	189.68 ± 8.06	8.27 ± 0.25
Kodai 1	276.44 ± 9.95	17.65 ± 1.12
Kodai 2	310.36 ± 11.43	15.58 ± 0.97
Kodai 3	302.42 ± 13.55	24.63 ± 1.34
Kodai 4	350.26 ± 12.22	21.62 ± 0.99
Kodai 5	316.38 ± 11.95	20.66 ± 0.87

Table 3

Results obtained for reference materials and fish tissue samples with the applied ultra-sound extraction followed by UV irradiation (methyl mercury)-CV-AAS methods ($n = 5$)

Sample	Certified values ($\mu\text{g g}^{-1}$)		Obtained ($\mu\text{g g}^{-1}$ dry weight)		
	Hg _T	MeHg	Hg _T	MeHg ^a	Hg ²⁺
IAEA-350	4.68 ± 0.28	3.65 ± 0.35	4.72 ± 0.42	3.59	1.13 ± 0.11
DOLT1	0.225 ± 0.037	0.080 ± 0.011	0.236 ± 0.02	0.078	0.158 ± 0.021
Carp	—	—	0.294 ± 0.028	0.154	0.140 ± 0.019
Cat fish	—	—	0.186 ± 0.018	0.108	0.078 ± 0.010
Shrimp	—	—	0.123 ± 0.011	0.062	0.061 ± 0.008

^a Values calculated as the difference between total mercury and inorganic mercury.

other two lakes could be obtained for analysis. The Hg_T in the fish samples collected ranged from 120 to 290 $\mu\text{g/kg}$. About 50–58% of the Hg_T was in methylated form. These total mercury concentrations obtained are well below the maximum limit of 0.5 ppm recommended for consumption.

The results obtained for the samples show elevated concentrations of mercury in Kodai Lake in comparison to the farther placed lakes, Berijam and Kukkal. Kodai Lake also showed higher levels of methyl mercury, 50 ng l^{-1} in waters, and 20 $\mu\text{g kg}^{-1}$ in sediment.

Biomethylation is the important mechanism by which methyl mercury is formed in sediments in fresh water bodies and oceans and also in columns of fresh and sea waters (Lindberg et al., 1987). Fish intestinal contents (Rudd et al., 1980) and the outer slime of fish have also been found to methylate inorganic mercury (McKone et al., 1971; Jernelov, 1972; Rudd et al., 1980).

Various factors such as organic acids, geochemical factors and bacteria influence the transformation between metallic mercury and different organic forms. Considerably more eutrophication is observed in Kodai Lake than in Berijam and Kukkal Lakes as this lake suffers much inflow of domestic sewage and also bears the brunt of tourists unlike the other two lakes. About one-fourth of the surface of Kodai Lake, towards the south and south-east is covered by water hyacinth. As more quantity of organic material is available in the waters, this serves as a source of food for microorganisms. This resulting increase in microbial activity leads to an increase in the production of methyl mercury from inorganic mercury present in the sediment (Furutani and Rudd, 1980; Ramlal et al., 1986), which is consistent with the results obtained. The presence of natural organic acids in the waters can also result in reaction with mercury residing in the sediment or emission sources to form soluble Hg-organo-complexes. The formation of these complexes is believed to greatly enhance mercury transport and be an important step in the formation of Me-Hg.

A close look at Tables 1 and 2 reveals an interesting observation. The sediment of Kukkal Lake shows methyl mercury of about 1.4 $\mu\text{g/kg}$ while in the waters, it is below the detection limit. The other two lakes show methyl mercury in both water and sediment.

Kodai Lake and Berijam Lake are situated in thickly wooded areas with the later being in a protected forest. Kukkal Lake has no forest tree cover nearby and is surrounded by hillocks. Among the water chemistry factors that are responsible for the lake responses towards mercury speciation, interactions

between mercury forms and dissolved organic matter are most important. Methyl mercury has been reported to undergo photolysis with a characteristic time of the order of a few days when exposed to ambient light conditions in surface water (Bloom et al., 2001). Photodecomposition of methyl mercury in lake water has also been reported by Sellers et al. (1996). Photoreduction of methyl mercury is very important because this is a potentially important loss process for the most toxic species of Hg in aquatic systems. Diurnal patterns of dissolved gaseous mercury in surface waters, with maximum concentrations in daylight hours, has been seen to provide evidence for the photochemical origin for this species. Because dissolved organic matter in the waters absorbs light in the photochemically important UV and low visible part of the spectrum, it may be an important agent in these photochemical reactions. It has been reported by Bloom et al. (2001) that in the photodecomposition of methyl mercury, hydroxyl radicals produced by photolysis of aqueous humus play an important part. Thus the light that reaches the lake surface has a strong bearing on the extent of methyl mercury decomposition. As all three lakes that have been studied in the present investigation are at an altitude of above 2000 m the UV flux will also be considerably higher than at sea level. However, Kukkal Lake, because of no forest cover, receives more sunlight resulting in increased decomposition of the oxidised mercury species to elemental mercury.

The spread of mercury contamination of Kodai Lake appears to have occurred through two routes:

- (i) Due to dispersion of elemental mercury in air, from the improperly stored and disposed mercury bearing waste and leakages from the factory processes. Once in air, mercury can be widely dispersed and transported for many kilometres from emission sources. The residence time of mercury in the atmosphere may be of the order of a year, allowing its distribution over long distances before being deposited to the earth. This is consistent with our previous observations (Balarama Krishna et al., 2003) that elevated mercury levels were found in lichen and moss samples on trees adjacent to Berijam Lake, with levels of 0.2 $\mu\text{g kg}^{-1}$ found more than 20 km away from the factory site. In this work it is significant to note that the Berijam Lake waters and sediments showed higher levels of mercury than the Kukkal Lake waters, which is situated farther away, about 40 km from the source.

(ii) The other way contamination may have occurred is due to surface water run off. The primary direction for surface water run off from the factory site is to the south into the Pambar Shola leading to the Kumbhakarai falls. Earlier reports have found mercury levels as high as 330 mg/kg in the sediment in a small depression from where the factory run off merged with the Pambarai stream and dropped into the Kumbhakarai falls (URS Dames and Moore, 2002). Though no direct watershed leads the surface run off from the factory site to Kodai Lake, the lake is at a lower elevation and acts as a catchment of surface run off. It has previously been suggested by an expert group that atmospheric mercury vapour could be taken up directly by plant foliage and that this might be an important pathway to watersheds in highly forested areas (Lindberg et al., 1987).

According to many studies, fresh water without an obvious source of anthropogenic nature mercury is estimated to contain 5 ng/l of mercury (ATSDR, 1997). Generally, drinking water is assumed to contain less than 0.025 µg/l (ATSDR, 1997). Thus the results obtained indicate that the factory emissions have resulted in elevated mercury levels.

However, the values of the mercury concentrations found in the samples from the lake are surprisingly low in view of the high concentrations of mercury that have been reported in lichen and moss samples (Balarama Krishna et al., 2003) and in soil (URS Dames and Moore, 2002). It is possible that the elemental Hg emitted only had limited residence time in the vicinity of the sources, with limited deposition and re-emission to the atmosphere from the water bodies. However, the contaminated soils and the sediment are continuously eroded and serve as a continuous source of mercury discharge to the lake waters which is confirmed by the fact that even 4 years after the closure of the thermometer factory elevated levels of mercury are found.

5. Conclusion

Analysis of water, sediment and fish samples from Kodai Lake from the tourist resort of Kodaikkanal, India, showed elevated levels of mercury even 4 years after the stoppage of mercury emissions from the thermometer factory that operated for 18 years and was closed down subsequently in 2001. Only remediation of the factory site could prevent any further mercury inputs to the lake through vapour transport and drainage.

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