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Phthalates in the Lithuanian Environment and the Need for Human Biomonitoring

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Control of chemical substances and correct evaluation of danger and risk caused by them based on scientific research are necessary for keeping the environment and public health safe. A regulation signed by the European Parliament and the Council to control registration, evaluation, authorisation and limitation of chemical substances (REACH) is the main juridical act of a new EU policy regarding regulations of chemical substances. The objective of this research was to collect and analyse the data concerning phthalates used in Lithuanian industry, to present data on their environmental levels in surface water, sediments, sewage treatment plant effluents, and to discuss the need for the human biomonitoring in Lithuania. The amount of diethylhexyl phthalate (DEHP) yielded to Lithuanian industry and market increased 65 times from 3.7 t in 2003 to 240.81 t in 2007. DEHP concentrations in Lithuanian wastewater exceeded the regulated annual average maximum allowable concentrations up to 27 times and the maximum allowable concentrations up to 13 times. High environmental concentrations of phthalates pointed out to the need for establishing permanent human biomonitoring in Lithuania.

Key words: phthalates, REACH registration, environmental levels, human biomonitoring.

1. Introduction

The European Environment and Health Strategy adopted by the European Commission in 2003 presented a new vision on how to address the environment and health in an integrated way and to place the health in the centre of environmental policy. Based upon the Strategy the Commission adopted in 2004 the European Environment and Health Action Plan 2004 – 2010. Under Action 3 in the Action Plan the European Commission announces the development of a coherent approach to Human Biomonitoring (HBM) in Europe in close cooperation with the Member States.

In December 2009 the COPHES consortium began working towards a EU HBM framework. To perform Human Biomonitoring on the European Scale (COPHES), this consortium declares a pan-European (all member states of the EU participating) approach to Human Biomonitoring (www.eu-hbm.info). The aim of COPHES is to work out a coherent and harmonized approach on Human Biomonitoring throughout Europe by means of commonly developed protocols including analytical methods for monitoring both toxic chemical substances registered in the REACH system and quality assurance to obtain reliable and comparable analytical results in all countries.

Biomonitoring connects environmental monitoring to human health. It integrates the contributions of all exposure media from all sources, every contact pathway and route of entry, all locations, activities and consumer products.

In Europe monitoring of phthalates is not linked with human exposure yet. Dimethyl phthalate (DMP), diethyl phthalate (DEP), di-n-butyl phthalate (DBP), benzyl-butyl phthalate (BBP), di-2-ethyl hexyl phthalate (DEHP), di-octyl phthalate (DOP) are main phthalates of interest found in surface water (Dudutyte et al. 2007, Marti et al. 2011, Dargnat et al. 2009b, Pejnenburg et al. 2006, Fromme et al. 2002) and waste water (Dudutyte et al. 2007, Sanchez et al. 2009, Dargnat et al. 2009a, Clara et al. 2010, Fauser at al. 2003).

Only one assessment of phthalate levels was done in Lithuania until today. It was carried out by the

Lithuanian Environmental Protection Agency (EPA), the Finnish Environment Institute (SYKE), the Baltic Environmental Forum, Lithuania, and the Centre for Environmental Policy, Lithuania. This assessment was part of the project "Screening of dangerous substances in the aquatic environment of Lithuania" funded by the Finnish Ministry of Environment in 2006.

Phthalates are among 74 substances from the national list of pollutants included in the Monitoring programme which are not analysed in the Environmental Research Department of Lithuanian EPA.

The aim of our study was to collect and analyse data on phthalates and chemical substances containing phthalates found in Lithuanian industry during the 2002-2009 year, to present data on their environmental levels in surface water, sediments, sewage treatment plant effluents, and to discuss the need for the establishment of human biomonitoring in Lithuania.

2. Methodology

We studied literature on an adverse effect of phthalates on human, especially children, health. We collected and analysed data on the quantities of phthalates or the chemical substances containing phthalates imported, used in Lithuania and the amounts sold in the EU market. We used Lithuanian Environmental Protection Agency data from the REACH data base to calculate the amounts of phthalates registered in Lithuania. To present the environmental levels of phthalates we used the data of an international project "Screening of dangerous substances in the aquatic environment of Lithuania" funded by the Finnish Ministry. In this study every phthalate is identified by its unique CAS (Chemical Abstracts Service) number. Sampling concentrated on discharges from the largest urban wastewater treatment plants and selected sampling points on trans-boundary rivers covered 40 sites during 2005-2006.

Methodology of determining phthalates levels in water, wastewater, sediments and sludge differs by additionally including internal standards, liquid-liquid extraction using dichloromethane or ethyl acetate, column clean up, concentration of extract and gas chromatography (Dudutyte et al. 2007).

3. Results and discussion

3.1. Phthalates effect on human health.

Phthalates are mainly added to hard plastics to make them soft. These plastics are used to make many products such as carpets, paints, glue, insect repellents, hair spray, nail polish (Agency.., 2001). One of the most popular and probably also the most studied phthalate is DEHP (Frederiksen et al. 2007). As phthalates are not chemically bound to the polymeric matrix in soft PVC, they can enter the environment either by losses during manufacturing processes or by leaching and evaporating from final products. Once released to the environment they tend to adsorb on various particles (Clara et al. 2010). More than 15 metabolites of DEHP, many of them identical with metabolites previously identified in rodents, have been identified in human urine (Koch et al. 2006; Silva et al. 2006). Exposure to phthalates may occur through inhalation of air, ingestion of drinking water, incidental ingestion of soil, ingestion of dust and ingestion of food, and for infants – through infant formula and breast milk. Ingestion of dust and inhalation of indoor air represent the most important on-food sources of exposure to phthalates (Clark et al. 2003). Phthalates can be incorporated into breast milk and transferred to the nursing child (Calafat et al. 2004).

Metabolism of phthalates begins hydrolysing diester phthalate into the primary metabolite monoester phthalate (Calafat et al. 2006) and further metabolizing to several oxidative metabolites which are the major metabolites of these phthalates which are subject of chemical analysis (Hines et al. 2009). Normally, hydrolysis would be a detoxification, but in vitro and in vivo studies have shown that diester phthalates become more bioactive when they are hydrolysed to monoester phthalates (Rusyn et al. 2006). Short-branched phthalates are mainly excreted in urine as monoester phthalates, while the more long-branched phthalates undergo several biotransformations, including further hydroxylation and oxidation before they are excreted in urine and faeces (Koch et al. 2005). DEHP excretion half-time is suggested to be 44 h (Koch et al. 2006). Secondary metabolites such as mono (2-ethyl-5-hydroxyhexyl) phthalate and mono (2-ethyl-5-oxohexyl) phthalate along with the primary metabolite mono (2-ethylhexyl) phthalate have been used to predict exposure to DEHP (Barr et al. 2003; Kato et al. 2004).

Animal studies present an established di (2-ethylhexyl) phthalate's (DEHP) negative impact on the development of the hippocampus in male rats during early postnatal days (Smith et al. 2011). In utero the exposure to di-butyl-n-phthalate was associated with significantly reduced weights of testes and accessory sex organs in F1 Sprague-Dawley male rats. This might be due to disruption of the stage-specific expression of genes related to androgen dependent organs development (Kim et al. 2010).

Prenatal exposure to mono (2-ethyl-5-hydroxyhexyl) phthalate, mono (2-ethyl-5-oxohexyl) phthalate, and mono-n-butyl phthalate may be inversely associated with the mental and psychomotor of infants, particularly males, at six months (Kim et al. 2011). It is suggested that DEHP exposure begins *in utero* (Latini et al. 2003) and may be associated with shorter gestation (Whyatt et al. 2009). Results of the nested case control study in China revealed negative association of prenatal di-n-butyl phthalate exposure with low birth weight, and DEHP negative association with birth length (Yolton et al. 2011).

Higher prenatal exposures to 2,5-dichlorophenol (up to 13.3 μg/L of phthalate detected in urine sample) predicted lower birth weight in boys (–210 g average birth weight difference between the third tertile and first tertile of 2,5-dichlorophenol; 95% CI, 71–348 g). Higher maternal benzophenone-3 concentrations (up to 92.7 μg/L of phthalate detected in urine sample) were associated with a similar decrease in birth weight among girls but with greater birth weight in boys (Wolff et al. 2008).

Table 1. Officially declared amounts of phthalates in Lithuania

	2002	2003	2004	2005	2006	2007	2008	**Total**
Diethylhexyl phthalate (CAS 117-81-7)	-	3.70 t	5.11 t	5.30 t	7.30 t	240.81 t	47.00 t	**309.22 t**
Di-n-octyl phthalate (CAS 117-84-0)	-	32.21 t	-	214.18 t	-	-	6.70 t	**253.09 t**
Polyethylene terephthalate (CAS 25038-59-9)	-	-	-	-	-	-	576.00 t	**576.00 t**
Dibutylphthalate (CAS 84-74-2)	12.00 t	12.32 t	1.60 t	0.81 t	0.70 t	92.13 t	-	**119.56 t**
Total	**12.00 t**	**48.23 t**	**6.71 t**	**220.29 t**	**8.00 t**	**332.94 t**	**629.70 t**	

Table 2. Officially declared amounts of chemical substances containing phthalates

	2003	2004	2005	2006	2007	2008	2009	2010	**Total**
Varnish “Merit Sanding” (CAS 84-74-2 up to 5%)	-	-	-	1.540 t	-	-	-	-	**1.540 t**
Varnish "MERIT SEALER" (CAS 84-74-2 up to 5%)	-	-	-	3.132 t	1.751 t	1.368 t	-	-	**6.251 t**
Varnish "Danspeed 80" (CAS 84-74-2 up to 1.5%)	-	-	-	4.880 t	0.220 t	-	0.117 t	-	**5.217 t**
Varnish "NC SEALER" (CAS 84-74-2 up to 1%)	-	-	-	4.060 t	0.520 t	-	-	-	**4.580 t**
Paint "NC" (CAS 84-74-2 up to 1.3%)	-	-	-	1.400 t	0.300 t	-	-	-	**1.700 t**
Varnish “NC” (CAS 84-74-2 up to 1.3%)	-	-	-	2.600 t	-	-	-	-	**2.600 t**
Varnish “NC Sealer” (CAS 84-74-2 up to 1%)	-	-	-	-	-	1.020 t	0.536 t		**1.556 t**
polyvinyl acetate emulsion (CAS 84-74-2) % **unknown**)	13.210 t	-	-	-	-	-	-	-	**13.210 t**
Varnish "MERIT SEALER" (CAS 117-81-7 up to 5%)	-	-	-	3.132 t	1.751 t	1.368 t	-	-	**6.251 t**
Varnish Capon Extra (CAS 117-81-7 up to 0.5%)	-	-	-	-	0.330 t	-	-	-	**0.330 t**
Paint BX-GR-DECKE (CAS 117-81-7 up to 2.5%)	-	6.000 t	2.000 t	2.000 t	-	2.100 t	2.100 t	-	**14.200 t**
Paint BX-PVC-ACRYL-DS-EG-DECKE (CAS 117-81-7 up to 2.5%)	-	10.000t	7.000 t	7.000 t	-	7.000 t	7.000 t	-	**38.000 t**
Dispersive paint (CAS 117-81-7 % **unknown**)	-	-	-	1,120.000 t	-	-	-	-	**1,120.0 t**
Glue PVC 5119/3 (CAS 85-68-7 % **unknown**)	-	-	-	-	-	-	2.178 t	2.130 t	**4.308 t**
Total	**13.210 t**	**16.000 t**	**9.000 t**	**1,149.744 t**	**4.872 t**	**12.856 t**	**11.931 t**	**2.130 t**	

3.2. Amount of phthalates and substances containing phthalates found in Lithuanian market.

Lithuanian Environmental Agency provided data on declared amounts of phthalates (Table 1) and declared the amounts of substances containing phthalates (Table 2), covering the year period from 2002 to 2010. During the period of 2003-2008, usage of diethylhexyl phthalate increased up to 65 times from 3.7 t (2003) to 240.81 t (2007). It was declared that usage of polyethylene terephthalate increased more than 6 times from 32.21 t (2003) to 214.18 t in 2005, but decreased in 2008 to 6.7 t. Polyethylene terephthalate is used in various types of plastics to increase durability and flexibility. Usage of dibutylphthalate has increased more than 7 times, namely, from 12 t to 92.13 t. This plasticizer is considered to have an adverse reproduction effect, it is classified toxic to human health and harmful to the environment. During the year of 2008 the amount of 576 t of polyethylene terephthalate was used. Di-(2-ethylhexyl) phthalate is a dominant plasticizer used in PVC due to its low cost.

Based on the information provided by Lithuanian Environmental Agency (Table 2). we

calculated the amounts of phthalates in chemical substances. In 2006 more than half of a ton of dibutylphthalate was used in production of varnish and paint. Additionally to the amount declared in the previous Table in 2007 approximately 0.4 t of dibutylphthalate was used additionally to that mentioned in the Table above.

We identified a lack of information on phthalate proportions in chemical substances. It is impossible to calculate the content of dibutylphthalate in polyvinyl acetate emulsion, diethylhexyl phthalate in dispersive paint without it. According to the law producers of chemical substances must report the quantities of chemicals used during the production process of final products. There is a record in 2006 on 1.120 t of dispersive paint produced but no information on phthalates.

3.3. Phthalates in the environment.

DEHP, BBP, DBP are the substances that meet the criteria for classification as toxic for reproduction (Category 1B) and are included in Annex XIV of Regulation (EC) No 1907/2006. From 21 January 2015 their use and placing on the market are prohibited (Commision... 2011).

According to the studies performed in different European countries the biggest concern over phthalate in waste water is DEHP (Table 3). DEHP concentrations in Lithuanian wastewater ranged from 0.42 µg/L to 53.2 µg/L in some samples exceeding annual average maximum allowable concentrations (AA-MAC) (2 µg/L) and maximum allowable concentrations (MAC) (4 µg/L) (Dudutyte et al. 2007). Mean concentration of DEHP in Spain was 47.90 ± 25.00 (Sanchez et al. 2009), in Denmark - 35.40 ± 10.60 (Fauser et al. 2003).

Table 3. Phthalate concentrations (µg/L) in waste water in various countries

	DMP	DEP	DBP	BBP	DEHP	DOP
Lithuania (Dudutyte et al. 2007)	<0.05 – 0.32	<0.05 – 1.92	<0.05 – 1.08	<0.05 – 1.31	<0.05 – 53.20	<0.05 – 0.12
Spain (Sanchez et al. 2009)	0.60 ± 0.50	50.70 ± 19.00	46.80 ± 15.00	0.67 ± 0.80	47.90 ± 25.00	-
France (Dargnat et al. 2009a)	0.82 ± 1.13	7.71 ± 5.21	1.10 ± 0.37	1.12 ± 0.54	22.46 ± 13.22	0.10 ± 0.16
Austria (Clara et al. 2010)	0.95	4.10	2.20	0.95	18.00	0.49
Denmark (Fauser et al. 2003)	-	-	1.03	0.39 ± 0.30	35.40 ± 10.60	0.57 ± 0.19

Table 4. Phthalate concentrations (µg/L) in surface water in various countries

	DMP	DEP	DBP	BBP	DEHP	DOP
Lithuania (Dudutyte et al. 2007)	<0.05	<0.05 – 0.07	<0.05 – 1.25	<0.05 – 1.55	<0.05 – 3.85	<0.05
Spain (Marti et al. 2011)	<0.20	<0.20 – 20.00	<0.10 – 0.30	-	<0.25 – 15.00	-
France (Dargnat et al. 2009b)	-	0.07 – 0.18	0.07 – 0.32	0.01 – 0.02	0.16 – 0.31	0.01
Denmark (Pejnenburg et al. 2006)	-	-	0.04 – 1.32	-	0.05 – 4.96	-
Germany (Fromme et al. 2002)	-	-	0.12 - 8.80	-	0.33 – 97.80	-

Mean concentration of DEHP in Spain was 47.90 ± 25.00 (Sanchez et al. 2009), in Denmark - 35.40 ± 10.60 (Fauser et al. 2003).

The highest DEHP concentrations in surface water (Table 4) were determined in Germany, up to 97.80µg/L (Fromme et al. 2002).

The data (Table 1) show that the amount of diethylhexyl phthalate yielded to Lithuanian industry and market, increased 65 times from 3.7 t in 2003 to 240.81 t in 2007. DEHP concentrations in wastewater exceeded the regulated annual average maximum allowable concentrations up to 27 times and the maximum allowable concentrations up to 13 times. Since human exposure to phthalates is associated with different routes, the human biological monitoring might best present the total internal pollutant amounts in the body.

The majority of EU countries are mandated by their Ministries of Health and/or Environment for the implementation of HBM. Till now Lithuania and other Baltic States do not perform HBM at the national level.

HBM is an effective tool to assess human exposure to environmental pollutants and potential health effects of such pollutants. To ensure environment protection and human health recent activities have shown that in the EU significant efforts are taken through HBM network to standardise the methods and to collect biomarkers data uniformly. Improved comparability of the European HBM data will allow cross boundary evaluation of human exposure and support the elaboration of background levels and guidance values. This will facilitate the identification of potential high exposure subpopulation groups in Lithuania and to apply preventive measures for decreasing health risk.

4. Conclusions

Lithuania is among a few EU countries that do not perform HBM. Based on the findings in industry wastewater emissions some phthalates exceed the allowed limits. Therefore industrial enterprises using phthalates should establish monitoring of pollutants in their wastewater. It is recommended to include phthalates, their metabolites as priority substances in the National Human Biomonitoring program to assess their exposure to population

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Ftalatai Lietuvos aplinkoje ir biomonitoringo poreikis

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Aplinkos kokybei ir visuomenės sveikatos saugai užtikrinti, remiantis moksliniais tyrimais, būtina kenksmingas medžiagas tinkamai įtraukti į apskaitą ir jas tvarkyti, bendradarbiauti tarptautiniu mastu, taip pat įvertinti cheminių medžiagų pavojingumą. Europos Parlamento ir Tarybos reglamentas dėl cheminių medžiagų registracijos, įvertinimo, autorizacijos ir apribojimų (REACH) yra pagrindinis Europos Sąjungos naujosios cheminių medžiagų tvarkymo politikos teisės aktas. Šio tyrimo tikslas – surinkti ir išanalizuoti duomenis apie Lietuvos pramonėje naudojamas toksiškas medžiagas ftalatus, jų koncentraciją paviršiniuose vandenyse, dugno nuosėdose, nuotekų dumble ir nustatyti žmogaus biomonitoringo poreikį Lietuvoje. 2003–2007 m. Lietuvos pramonėje sunaudotas di(2-etilheksil) ftalato kiekis padidėjo apie 65 kartus – nuo 3,7 t iki 240,81 t. DEHP koncentracijos kai kuriuose nutekamuosiuose vandenyse viršijo didžiausią leistiną vidutinę metinę koncentraciją iki 27 kartų, didžiausią leistiną koncentraciją iki 13 kartų. Didelės ftalatų koncentracijos paviršiniuose vandenyse rodo, kad būtina sukurti nuolatinę žmogaus biomonitoringo sistemą Lietuvoje, siekiant apsaugoti gyventojų sveikatą.