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PRELIMINARY STUDY OF THE TOXICITY OF RAINWATER RUNOFF FROM THE ROOFS WITH VARYING COVERAGE

WSTĘPNE BADANIE TOKSYCZNOŚCI SPŁYWÓW DESZCZOWYCH Z DACHÓW O RÓŻNYM POKRYCIU

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Streszczenie. Deszczowe ścieki miejskie uważane są obecnie za jedno z głównych źródeł zanieczyszczenia wód. Zasadniczy ładunek tych zanieczyszczeń pochodzi z infrastruktury transportowej, a część z nich wykazuje silne oddziaływanie toksyczne na organizmy żywe. Dodatkowym źródłem substancji toksycznych w ściekach deszczowych mogą być spływy z dachów. Różnorodność pokryć dachowych stosowanych w budownictwie powoduje, że do wód opadowych spływających po powierzchni dachu uwalniana jest ogromna ilość substancji chemicznych mogących powodować zatrucie i degradację środowiska. Badano toksyczność spływów deszczowych z czterech rodzajów dachów: o pokryciu miedzianym, z blachy cynkowej, z papy oraz z płyt cementowo-azbestowych (eternitu) wobec skorupiaka słodkowodnego *Daphnia magna* Straus. Pobrano próbki z czterech niezależnych opadów deszczu na przełomie marca i kwietnia 2015 roku. Do badań wykorzystano ścieki deszczowe pierwszego spływu, o największej koncentracji zanieczyszczeń. Toksyczność ścieków deszczowych określono poprzez wyznaczenie parametrów 24hEC50 i 48hEC50 metodą probitową oraz metodą graficznej interpolacji. W przypadku małej toksyczności próbki, która uniemożliwiała wyznaczenie parametru EC50, obliczano parametr EC10. Toksyczność spływów deszczowych z dachów porównano z toksycznością opadu zebranego bezpośrednio, bez spłukiwania jakiejkolwiek powierzchni. Stwierdzono, że największą toksyczność wobec rozwielitek wykazywały spływy z dachu pokrytego blachą miedzianą (średnia wartość 24hEC50 wynosiła 0,17%). Spływy z dachu cynkowego wykazywały mniejszą toksyczność (24hEC50 ok. 25,6%), lecz ze względu na częstość występowania pokryć z blachy cynkowej i ocynkowanej znaczenie tego metalu w toksyczności miejskich ścieków deszczowych może być znacznie większe niż miedzi. Najmniejszą toksyczność wobec skorupiaków *Daphnia magna* stwierdzono w przypadku pokryć azbestowych. Wody opadowe zbierane bezpośrednio nie wykazały działania toksycznego wobec organizmów testowych.

Key words: rainwater runoff, roof covering, toxicity, *Daphnia magna.*
Słowa kluczowe: spływy deszczowe, pokrycia dachowe, toksyczność, *Daphnia magna.*

INTRODUCTION

The urbanized areas in the world are constantly growing, and the urban management has become one of the greatest challenge of the twenty-first century. According to the report of the Department of Economic and Social Affairs of the United Nations (DESA), in 1950 30% of the

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world's population inhabited in the cities, today it is about 54%, and according to forecasts, in 2050 two thirds of the human population will be living in cities.

The huge quantities of contaminants are produced in urban areas: municipal wastewater, solid waste, pollution from industry, energy production and transport. As a result of the expansion of sewage systems and legal regulations for the disposal of liquid and solid waste, domestic and industrial sewage are becoming less of a threat to the environment. Currently, the urban runoff and the combined sewage overflows events occurring during wet weather are considered as the major sources of water pollution (Hatt 2006; Zgheib 2011).

Along with urbanization impervious surface areas increase, which causes a number of problems associated with flooding, runoff pollution and effective management of rain wastewater. Volatile compounds and dust generated in urban areas settle on a variety of surfaces, and they are transferred to the sewer system or directly into the environment with rainwater or meltwater. In the storm wastewater of various origin more than 30 different substances that can adversely affect human health (by direct consumption or during recreation) and aquatic life have been identified (Makepeace et al. 1995). Among the compounds of greatest importance, both for water reservoirs and for water streams, there are nutrients, substances that reduce the concentration of dissolved oxygen, hydrocarbons, metals and pesticides. Large concentrations of these substances often cause very high toxicity of the urban stormwater (Tang et al. 2013).

Among the rain wastewater from different origins, runoff from the multi-line highways is considered as the major source of toxic substances. The main identified toxicants are copper, zinc, cadmium, lead, platinum, some PAHs (e.g. pyrene, fluorantan, phenanthrene), and chlorides which in addition to a direct toxicity may also increase the mobility of heavy metals (Wei and Morrison 1994; Boxall and Maltby 1997; Marsalek et al. 1997).

Roof is one of the form of impervious surface found in urban catchments. Roof runoff is an important transport pathway for pollutants originated from the atmosphere (deposited on the roof surface or washed out by rainwater) and then discharged to the sewage system or directly to the receiving waters. It is known, however, that chemical pollutant levels are generally higher in roof runoff samples than those found in the rainwater itself (Melidis 2007).

Contaminants in roof runoff may come from:

1) roofing material – compounds contained in roof covering, flashings and gutter may be leached into runoff;
2) dry deposition on the roof surface – airborn pollutants (dust and dirt), insects, leaves, bird's wastes;
3) atmospheric contaminants washed out by rainwater.

The studies confirm that the runoff from roofs may be responsible for more than 80% of the load of some heavy metals in the combined sewage system during wet weather (Simmons et al. 2001) and it is the most important source of heavy metals in aquatic ecosystems (Good 1993). The most critical effect of heavy metals (especially copper) is their high toxicity to aquatic life (Förster 1996). Copper concentrations are highest in the runoff from full copper roofs, although Cu flashings and gutters can also elevate Cu level in runoff (Good 1993; Förster 1999).

Volatile compounds have also the great impact on the quality of rainwater. In rural areas the main source of this type of contaminants is agricultural use of chemicals, in cities – highly industrialized areas and heavily used highways. Rooftop runoff from buildings located along main transportation routes show a high concentration of contaminants exhibiting toxicity (Polkowska et al. 2002).

Runoff from the roofs shows the highest concentration of pollutants at the beginning of the rainfall (Förster 1996,1998,1999; Zobrist 2000). This phenomenon is described as "the first flush effect". Some studies indicate, however, that some pollution concentrations and toxicity may remain high even three hours after the beginning of the storm event (Good 1993).

Contamination level in the roof runoff depends also on other parameters such as climate, season, antecedent dry periods, type, age, inclination, chemical characteristic, size and surface exposure of the roof (Förster 1996; Chang et al. 2004; Clark et al. 2008; Wicke et al. 2014) and on characteristic of rainfall itself: intensity and duration, temperature, pH, chemical substances washed from atmosphere (pesticides, hydrocarbons, organhalogens).

One of the factors strongly influenced the quality of roof runoff is the type of roofing material (Chang et al. 2004). The results of the laboratory “leaching” studies indicates that most of the common construction materials (see Table 1) may release pollutants into water flowing down the roof (Chang et al. 2004; Clark et al. 2008). Surprisingly even vegetated roofs can be a source of runoff pollution (Berndtsson et al. 2006).

Table 1. Typical components used for roof construction
Tabela 1. Najczęściej stosowane materiały dachowe

Roofing materials – Materiały dachowe	Roof element – Element dachu
Concrete and clay – Beton i ceramika	tiles – dachówki
Tar paper – Papa smołowana	flat roofs – dachy płaskie
Asphalt/asbestos – Asfalt/azbest	shingles – gonty
Wood – Drewno	shingles and shakes – gonty
Zinc, copper, aluminium, galvanized metal Cynk, miedź, aluminium, metale ocynkowane	flashings, roofing panels, roofing nails, gutter and drain obróbki blacharskie, blachy dachowe, gwoździe, rynny
Plastic/rubber – Plastik/guma	membrane roofing – membrany dachowe
PVC plastic – Tworzywo PCV	gutter and drain material – rynny i drenaż
Plastic glue/mastic – Klej/kit	patching compound – materiały naprawcze

Source – Źródło: Pit and Lalor (2001), modified – zmienione.

Some contaminants may originate from compounds added to the main roofing material to improve its performance or durability. Painting of the metal constructing material, for example, act as the environmental barrier but paints can contain metals (usually lead) (Davies and Burns 1999). Impregnation of wood shingles with copper-containing compounds may cause the release of large amounts of copper (Pit and Lalor 2001).

The chemical composition is the basic criterion for determining the quality of water. Chemical analysis can, however, be insufficient to estimate the potential impact of wastewater on the environment since some harmful factors are not covered by standard physico-chemical analysis. Moreover, chemical compounds contained in wastewater may exhibit a synergistic effect and their impact on living organisms may be greater than would appear from routine quantitative research. Therefore, it is recommended to perform bioassays to determine the overall impact of wastewater on the environment.

While the level of pollutants in storm water and roofs runoff is quite well studied (Chang et al. 2004; Cheah et al. 2007; Lye 2009), there is a shortage of information on the toxicity of this type of urban wastewater and its potential impact on receivers and environment. Toxicity of most substances may be contained in the roof runoff is known but in the case of the environmental studies total chemical composition of the sample is difficult to determine. Moreover the toxic effect may be the result of synergistic action of various compounds present in the sample. In water and wastewater samples toxicity cannot be determined as the concentration or dose of a substance inducing a specific effect. In these studies, the value of toxic levels is defined as a dilution of the original sample and classification of the toxicity also refers to dilution (Table 2).

Table 2. Toxicity assessment of wastewater, where the exact composition of the sample is not known
Tabela 2. Szacowanie toksyczności ścieków przy nieznanym składzie próbki

Effect level Poziom efektu	Toxicity point value Wartość toksyczności	Immobilisation of *D. magna* after 48 h Unieruchomienie *D. magna* po 48 h
No toxicity Nietoksyczny	0	EC_{10} at 100% dilution EC_{10} w rozcieńczeniu 100%
Potential toxicity Potencjalnie toksyczny	1	EC_{20} – EC_{40} at 100% dilution EC_{20} – EC_{40} w rozcieńczeniu 100%
Confirmed toxicity Toksyczny	2	EC_{50} at 100% dilution EC_{50} w rozcieńczeniu 100%
Severe toxicity Silnie toksyczny	3	EC_{50} at dilution ≤ 75% EC_{50} w rozcieńczeniu ≤ 75%

EC – effective concentration required to affect some percentage of the organisms tested – stężenie wywołujące efekt toksyczny w określonym procencie organizmów testowych.
Source – Źródło: Dutka, after Marsalek et al. (1999).

This study investigated the toxicity of the runoff from the four most common types of roofing construction materials to the freshwater crustacean *Daphnia magna* Straus. The aim of the work was the preliminary assessment of the potential effects of roofs runoff of the initial phase of rainfall, with the highest concentration of pollutants, on aquatic invertebrates without specifying the type of harmful substances in the studied wastewater.

MATERIAL AND METHODS

Sample collection

Rainwater runoff samples were collected from the roofs with four various coverage: zinc sheet, copper sheet, tar paper and cement-asbestos boards, in centrum of Lodz, during four rainfall events from March 22 to April 4, 2015. All types of roofs were located on fairly low buildings (up to three stories). Samples always came from the same roofs and were collected simultaneously from the parts of roofs of the western or north-western exposure (prevailing wind direction). The sampling used specially constructed 1L vessels for collecting only first flush of runoff, with the largest content of contaminants. The vessel automatically closed after filling. Then runoff samples were poured into 0.5L plastic bottles and stored at 6°C in a refrigerator and were applied for acute toxic tests within two days after sampling. When the time gap between sample collection and toxicity test were longer than two days, samples were frozen and used within one month after sampling.

Test organisms

The *Daphnia magna* used in this study was cultured at 22 ± 2°C with a natural light-dark cycle. Cultured organisms came from ephippia delivered by Microbiotest Inc. The daphnids were fed daily with green algae *Scenedesmus subspicatus* cultured in laboratory. The natural culture water with parameters as follows: pH 7.4–7.6 Ca^{2+} concentration of 42 $mg \cdot L^{-1}$ (aerated 30 min. before use) was exchanged twice a week.

Bioassay method

Acute toxic tests with *D. magna* were performed according to the PN-ISO 6341 method on up to 24-h old newborns of *Daphnia magna* St. A day before test all pregnant females were transferred from culture to individual vessel with dilution water (composition in accordance with PN-ISO 6341). After 24 h, the surviving newborns were transferred into a fresh dilution water and fed with algae two hours before experiment.

Test solutions of rooftop runoff were prepared in 100 mL glass volumetric flasks in the geometric series where the next sample was a double dilution of the previous one. For runoffs from roofing tar, asbestos and zinc roofs the dilution series were 100, 50, 25, 12.5 and 6.25% of the runoff. In the preliminary test for copper roof runoff such dilutions cause 100% toxic effect to all organisms used in the test. The final series of dilutions in these tests were 0.5, 0.25, 0.125, 0.063 and 0.031%. Test organisms were placed in multicell test plate (5 neonates in each of 4 cell for every dilution and control) and after 24 and 48 h the percentage of surviving (active) newborns was counted. During the tests, test plates with the newborns were kept in darkness at 20 ± 2°C. Each test was performed twice.

Calculation method

Toxicity of the rooftop runoff was determined by the designation of parameters 24hEC50 and 48hEC50 by Probit and the Graphical Interpolation Methods. EC50 means the concentration (or dilution) with 50% test effect (immobilization of 50% organisms used in the test). The low toxicity of same samples did not allow to determine the EC50 parameter. In this cases the parameter EC10 was calculated. The mean and standard deviation values for four rainfall events were determined.

Toxicity of runoff from rooftops was compared with the toxicity of rainfall collected directly, without rinsing any surface.

RESULTS AND DISCUSSION

Most animals were alive in the control solution after 24 and 48 h of experiment: mortalities were less than 10% in control at the end of the test (guideline value of this test).

Samples of rain collected directly, without rinsing any surface, turned out to be nontoxic to neonates of Daphnia magna (data not shown). Toxic effect of 100% dilution was less than 10%. Toxicity of rainwater mainly results from presence of pesticides (Sakai 2006). In the sampling period (late March and early April) the level of use of pesticides is still very low, hence the low toxicity of rainwater.

The results of toxicity tests for rainwater runoff from the four studied types of roofing materials are shown in Fig. 1. The graphs show the average values of toxic effect for four independent rainfall events. Due to the different scale of dilutions of the samples from the copper roof covering, Fig. 2A shows the graph of the toxicity of the runoff from this type of roofing material.

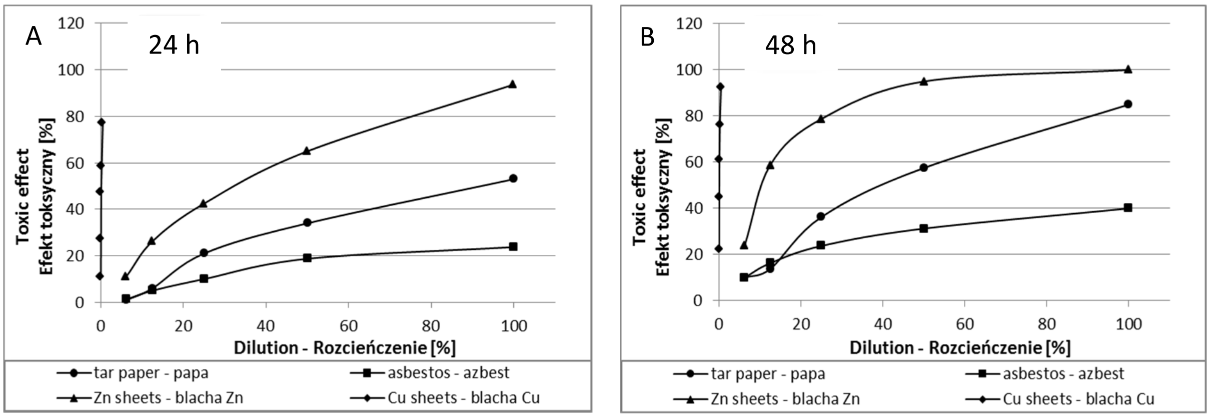

Fig. 1. Graphs of rainwater runoff toxicity from the roofs of varying coverage for 24 (A) and 48 (B) hours of exposure of the test organisms on the rooftop wastewater
Ryc. 1. Wykresy toksyczności spływów deszczowych z dachów o różnym pokryciu po 24 (A) i 48 (B) godzinach ekspozycji organizmów testowych na próbki ścieków

LC50 or EC50 parameters are the measure of the toxicity of chemicals. They indicate concentration of a substance causing death or toxic effect in 50% of the population used in the assay during 24 or 48 hours exposition to the toxic compound. In the case of the environmental studies chemical composition of the sample is difficult to determine. Moreover the toxic effect may be the result of synergistic action of various compounds present in the sample. In these studies the parameter EC50 is determined by the value of dilution of water or wastewater samples.

Table 3 shows toxicity tests results presented as 24hEC50 and 48hEC50 parameters calculated by Probit Method (PM) and designed in Graphical Extrapolation Method (GIM). Due to the low toxicity of asbestos roof runoff, the EC10 for 24 and 48h parameters were determined.

Table 3. Toxicity effect of roof runoff on *Daphnia magna* neonates
Tabela 3. Toksyczność spływów deszczowych z dachów wobec neonatów *Daphnia magna*

Roof coverage Rodzaj pokrycia	EC50-24 h		EC50-48 h	
	PM	GIM	PM	GIM
Tar paper Papa smołowana	80.70	92.10	35.40	41.20
Asbestos Azbest	29.60 (EC10)	25.00 (EC10)	12.80 (EC10)	6.30 (EC10)
Zn sheets Blacha cynkowa	25.60	33.30	11.50	6.90
Cu sheets Blacha miedziana	0.19	0.17	0.09	0.08

Calculated by probit method (PM) and designated by Graphical Interpolation Method (GIM) values of EC50 or EC10 (in % of dilution of sample) for 24 and 48 h exposure are shown – Obliczone metodą probitową (PM) i wyznaczone metodą graficznej interpolacji (GIM) wartości EC50 lub EC10 (w % rozcieńczenia próbki) po 24 i 48 h ekspozycji.

The toxicity of individual runoff from the roof of the same coverage varied considerably between rainfall events. Figure 2B and Table 4 show the obtained toxicity curves and calculated EC50 parameters for runoff from the copper roofing for each rainfall events. Since the samples were collected every time from the same roof, differences in runoff toxicity could be due to the impact of variable parameters of rainwater itself and weather conditions on the day of precipitation. It is known that acidic nature of the rainwater may react with compounds in the roofing materials and cause many substances to leach out (King and Bedient 1982). Additionally higher roof temperatures (in warmer or more sunny day) may accelerate chemical reactions and decomposition of the roofing constructing materials and compounds deposited on roof surface (Chang and Crowley 1993). Thomas and Greene (1993) found differences in metal levels associated with variations in atmospheric deposition and related to antecedent dry periods. All these factors make the runoff can differ significantly from one another.

Table 4. Calculated EC50-24 h and EC50-48 h values for full copper roof for four individual rainfall events
Tabela 4. Obliczone wartości EC50-24 h i EC50-48 h dla próbek z dachu miedzianego w przypadku czterech opadów deszczu

Date of rainfall event Data opadu	EC50 [% of dilution – rozcieńczenia]			
	24 h	$\bar{x}$ ± SD	48 h	$\bar{x}$ ± SD
22.03.2015	0.117	0.19 ± 0.09	0.070	0.09 ± 0.03
28.03.2015	0.330		0.134	
31.03.2015	0.085		0.049	
04.04.2015	0.228		0.100	

The high level of toxicity of wastewater from the roof made of copper (sample dilution giving 50% effect after 48 h of exposure were more than a thousandfold) is undoubtedly the result of high concentrations of this element in runoff. Full copper roofs may generate runoff with concentrations of up to 11.1 mg · L^{-1} Cu, predominantly in dissolved, bioavailable Cu^{2+} form due to the low pH of the rainwater (Clark et al. 2008; Pennington and Webster-Brown 2008).

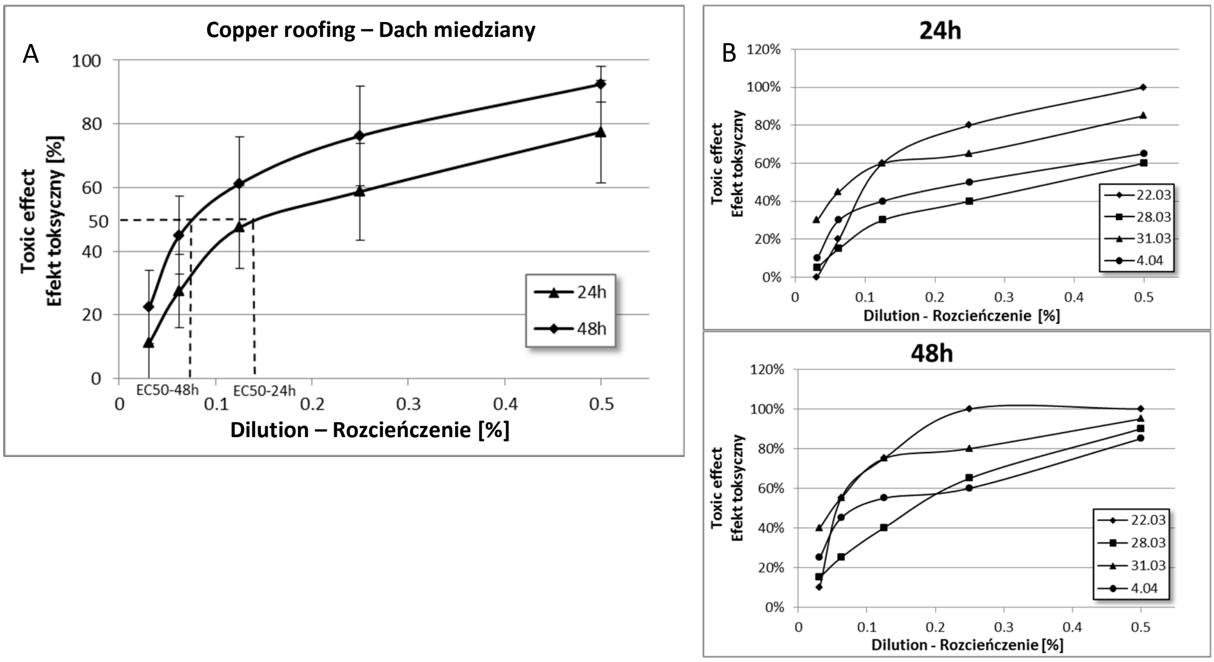

Fig. 2. Toxicity of runoff from copper roof coverage. A – means for four rain events, B – toxic effect plots for each rain event for 24 (up) and 48 (down) hours of exposure of the test organisms on the rooftop wastewater

Ryc. 2. Toksyczność spływów z dachu miedzianego. A – średnia dla czterech opadów, B – wykresy toksyczności dla poszczególnych opadów po 24 (wykres górny) i 48 (wykres dolny) godzinach ekspozycji organizmów testowych na ścieki deszczowe

Previous studies of heavy metals content in the runoff from the same copper roof as in this research showed concentrations of copper up to 7 mg · L^{-1} (Sakson and Olejnik 2013). Copper toxicity studies for small aquatic invertebrates such as *Ceriodaphnia dubia*, *Daphnia carinata* and *Daphnia magna* showed values of 48hEC50 in the range of 6.5 µm · L^{-1} for *D.magna* to 37.3 µm · L^{-1} for *D.carinata* (Dave 1984; Cooper 2009). These data are in good correlation with the results obtained in this work.

Toxicity of runoff from the roof of zinc was significantly lower than of copper roof. Values of the 24hEC50 and 48hEC50 parameters (with respect to dilution of the sample) were 25.6% and 11.5% respectively. However, due to the widespread use of zinc sheet and galvanized steel in the construction industry, its share in the toxicity of urban runoff can be much greater than copper. Although in typical residential areas roof runoff may only contribute about 10 to 20 percent of all stormwater runoff, almost all of the zinc has been found to originate from this source due to the common use of galvanized metal for roof covering and drainage system like gutters and downspouts. Furthermore the zinc concentration in the runoff from roofs made of this metal may reach 17 mg · L^{-1} (Zawilski et al. 2014). It has been found by Bailey et al. (1999) that much of the toxicity of runoff may have been a result of divalent cations, in particular, zinc from galvanized roofs.

Samples from roof covered with tar paper also proved to be toxic. Dilution causing toxic effect in 50% of test subjects after 48 h of exposure was approximately 40% of the initial sample. It is known that tar paper can be a source of petroleum derivatives (Brandt et al. 1985).

During the runoff of rainwater the polycyclic aromatic hydrocarbons (PAH), tarry substances, paper tar particles and surface active compounds may enter the environment, which may contribute to the contamination of the ecosystem.

Runoff from the roof covered with asbestos proved to be the least toxic to daphnia. As it is known, this material is highly carcinogenic but only by absorption through the respiratory system. Presumably toxicity runoff from these roofs can arise only from the content of the substance applied by air (dry deposition).

CONCLUSIONS

Although rainwater is widely regarded as a clean source of water, runoff from the roofs are characterized by significant concentrations of varying pollutants including metals, nutrients, polycyclic aromatic hydrocarbons (PAHs), pesticides and others. Particularly metals such as Zn and Cu are present in relatively high concentrations. These metals can be released from weathered roofing material or derived from the particles deposited on the surface of the roof.

Toxicity studies of rainwater runoff from the roofs of four different types of coverage have shown that each type of material can be a source of substances harmful to living organisms. The high toxicity of samples from roofs made of copper and zinc was undoubtedly the result of high concentrations of these metals in runoff. In the toxicity of runoff from the roofs covered with tar paper and asbestos the presence of substances deposited on the roof during the dry weather periods may have a large share.

According to the classification of the toxicity of environmental samples (Table 2), roof runoff from zinc, copper and tar paper roofing may be considered to be severe toxic and wastewater from asbestos roof – potentially toxic. Only rainwater samples not flushing any surface showed no toxic levels of contaminants.

Due to the limited availability, increasing consumption and costs of clean fresh water, rainwater and runoff are increasingly being considered as the alternative to tap water sources of water for irrigation, economic purposes or for consumption (Hatt 2006; Fletcher et al. 2008). This research shows, however, that runoff from roofs (especially with copper and zinc coating), is highly toxic and should be cleaned before using (even for economic purposes) and before discharging it into environment.

Significant differences in the quality of rainwater from various sources indicates the need for detailed research for runoff from specific areas or even from specific places. To determine the exact effect of the type of roofing material on the toxicity of runoff comparative studies on model roofs (the same area, inclination and orientation relative to directions of the world) are required. This will be the subject of our further research.

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Abstract. At present the urban rainwater runoff is considered as one of the main sources of water pollution. The basic load of the contaminants comes from transport infrastructure and some of them have a strong toxic effect on living organisms. An additional source of toxic substances in the urban runoff is rooftop wastewater. Because of variety of roof coverings, a large amount of different chemicals is released into the wastewater that can cause environmental poisoning and degradation. This study investigated the toxicity of rainwater runoff from the four types of roofs: coated with copper, zinc sheet, with tar paper and cement-asbestos sheets to the freshwater crustacean *Daphnia magna* Straus. Samples were taken during four independent rainfalls in March and April 2015. The study used the first part of runoff with the greatest concentrations of contaminants. Toxicity was determined by the designation of parameters 24hEC50 and 48hEC50 by Probit and the Graphical Interpolation methods. If the low toxicity of the sample did not allow to determine the EC50 parameter, the parameter EC10 was calculated. Toxicity of runoff from rooftops was compared with the toxicity of rainfall collected directly, without rinsing any surface. It was found that runoff from the roof covered with copper sheets shows the greatest toxicity to *Daphnia magna* (average 24hEC50 was approximately 0.17%). Runoff from the roof of zinc showed less toxicity (24hEC50 approx. 25.6%), but because of the prevalence of zinc and the galvanized sheet covering, the importance of this metal in toxicity of urban rainwater can be much greater than copper. The smallest toxicity to crustacean has been found for asbestos covering. Rainwater collected directly showed no toxicity to the test organisms.