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**ASSESSMENT  
OF THE QUALITY OF SURFACE WATER  
FROM SELECTED AREA OF ACTIVE OIL EXPLOITATION\*\***

**1. INTRODUCTION**

Poland has the second smallest water resources per capita in Europe and at the same time a relatively low reservoir capacity. Water for human consumption is supplying partly (*ca.* 30%, year 2013) from surface waters [1] which are poor quality on the area of almost all country. Only *ca.* 5% of Polish monitored rivers (in terms of length) meet quality standards for resources as source of drinking water [2, 3]. Thus, a potential water deficit in Poland results not from the lack of water, but from the lack of good quality water. In that context, the main challenge regarding water resources is reaching a proper level of surface water quality by controlling of discharge of pollutions originated from the industrial sewage, the municipal wastewater and the agricultural sources.

The study area of oil exploitation is located in the south of Poland, in the basin of the Vistula River. The catchment area of the Vistula River, the biggest river in the country, covers of 19 4424 km<sup>2</sup> (the small part of the basin of 25 458 km<sup>2</sup> is located on the territory of Ukraine, Belarus and Slovakia). In the catchment area of the Vistula River live *ca.* 60% of habitants of Poland [4, 5]. In the study area the surface waters are the main source of water for human consumption (*ca.* 80%) [1]. Therefore, improvement of the quality of these waters is extremely desirable. In consequence, the quality of surface waters originated from the investigated region has potential impact on quality of water in macro-region of Upper Vistula.

During the last decade, the monitoring of the quality of surface water used for drinking water recovery in Poland is become more active. The previous data demonstrate poor

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\*\* The research leading to these results has received funding from the Polish-Norwegian Research Programme operated by the National Centre for Research and Development under the Norwegian Financial Mechanism 2009–2014 in the frame of Project Contract No Pol-Nor/235294/99/2014

screening of the parameters needed for a reliable water quality assessment. This data has shown that the obligatory programs for monitoring of surface water which are a source of water for human consumption [6, 7] were not respected. Among the required parameters, some of them were not measured and many of them like arsenic, cyanide, cadmium, lead, mercury and polycyclic aromatic hydrocarbons (PAHs) were determined sporadically [8].

Nowadays, Poland as a Member State of the European Union is obligated to implement the Directive establishing a framework for community action in the field of water policy [9]. The aim of this Directive is to create a framework for the protection of inland surface waters, transitional waters, coastal waters and groundwater. Member States should adopt procedures to eliminate pollution of surface water by the priority substances and gradually to reduce pollution by other hazardous substances. In that context, the monitoring of polish surface waters is currently more effective. Despite it covers only part of the body of river waters very often allow to investigate the changes in the water environment in the selected areas [10].

It was found, that the quality of surface waters is relatively poor. In years 2004–2007 eutrophication occurs in 44,9% of investigated sampling places and the main reason of that situation was the high values of Kjeldahl nitrogen, total phosphorus, total organic carbon (TOC) and biochemical oxygen demand (BOD<sub>5</sub>) [11].

Current data (years 2010–2012) [11, 12] show that the bad chemical condition of some surface waters is caused mainly by to high concentration of two polycyclic aromatic hydrocarbons (PAH), benzo(ghi)perylene and indeno-1,2,3-cd-pyrene and two heavy metals mercury and cadmium. It was found that sediments in the rivers of south of Poland contain high level of concentration of TOC. The higher values of concentration of polycyclic aromatic hydrocarbons and polychlorinated biphenyls (PCB) were detected in some samples.

The pollution by stable organic compounds such as hydrocarbons can be dangerous not only for surface waters, but also for other types of water. The interesting example is a risk of pollution by hydrocarbons of the mineral waters from Iwonicz-Zdrój (Poland). The commune of Iwonicz-Zdrój has the status of the spa, and is located in the mining area of the mineral water, oil and gas exploitation. The investigations of soil and ground water in this area show that protection of natural environment in the place of co-occurrence of both the mineral waters and hydrocarbons sources is difficult [14, 15].

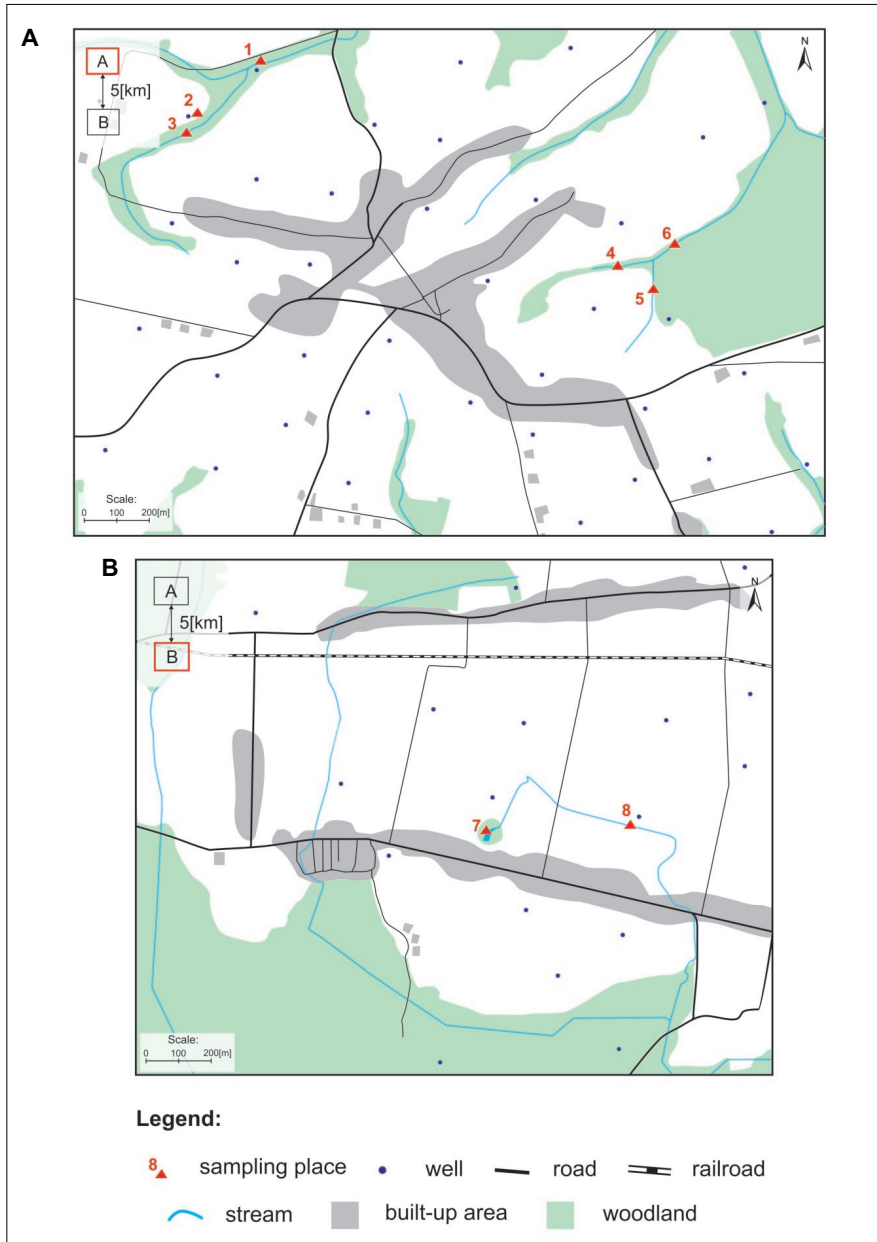
The main goal of this work is the study of surface water quality in the selected area of active oil exploitation. Among many chemical water quality parameters, the hydrocarbons concentration as an indicator of contamination of petroleum products and nitrates concentration as a marker of agriculture pollution were investigated.

## **2. METHODOLOGY**

### **2.1. Samples**

Water samples were collected in month of October 2014 and the sampling places are presented in the Figure 1. Waters were sampling from the streams (samples No. 1, 3, 4, 5, 6 and 8), pound (No. 7) or directly from surface tank situated in the vicinity of operating well (No. 2) in the present oil exploitation area. Samples were collected in the polyethylene and

glass bottles dependent on requirements for the type of examinations (organic and inorganic). The pH and electroconductivity were measured in the field with portable equipment. Next, the samples were transported to the laboratory, stored at 4°C and analyzed successively.



**Fig. 1.** Localization of sampling places

## 2.2. Measurements of selected physical and inorganic parameters of water quality

In Table 1 were gathered analytical methods applied for the detection of selected parameter of water quality of collected samples. Table 1 includes also some details which describe the method.

**Table 1**  
Analytical methods applied for detection of selected physical and inorganic parameters of water quality

Parameter	Analytical method
Turbidity	Colorimetric (apparatus: the Hach Model DR/2000)
pH	Potentiometric
Electroconductivity (EC)	Conductometric: ISO 27888:1993 Water quality – determination of electrical conductivity
Hardness ( $\text{Ca}^{2+} + \text{Mg}^{2+}$ )	Titrimetric: ISO 6059:1984 Water quality – determination of calcium and magnesium content – EDTA titration method Titration with EDTA (0.05 M) at pH 10 with buffer (ammoniac) with indicator Eriochrome Black T. When all of the magnesium and calcium has been complexed the solution terns from red wine to blue, marking the end point of titration
$\text{Ca}^{2+}$	Titrimetric: ISO 6058:1984 Water quality – determination of calcium content – EDTA titration method Titration with EDTA (0.05 M) with 16% potassium hydroxide with indicator Calcon. When all of the calcium has been complexed the solution terns from red wine to blue, marking the end point of titration
$\text{Mg}^{2+}$	Titrimetric: Hardness minus calcium
$\text{Cl}^-$	Titrimetric: ISO 9297:1994 Water quality – determination of chloride – Silver nitrate titration with chromate indicator (Mohr's method) Titration with $\text{AgNO}_3$ (0.01 M) with indicator $\text{K}_2\text{CrO}_4$ . When all of the chloride has been precipitate the solution terns from yellow to pinkish yellow, marking the end point of titration
$\text{HCO}_3^-$ , $\text{CO}_3^{2-}$	Titrimetric: ISO 9963-1:1994 Water quality – determination of alkalinity Titration with HCl (0.1 M) with indicator phenolphthalein and methyl orange
$\text{O}_2$ (dissolved)	Colorimetric: The chemical determination of oxygen concentrations is based on the Winkler method. The basis of the method is the quantitative oxidation of iodide ions to iodine by the oxygen. The amount of iodine generated in that way is determined by titration with a standard thiosulfate solution. Next, the endpoint is determined by the absorption of ultraviolet light by the tri-iodide ion
$\text{SO}_4^{2-}$	Turbidimetric: Sulfate ions are precipitated in an acetic acid medium with barium chloride. As a result barium sulfate crystals of uniform size are formed. Light absorbance of the barium sulfate suspension is measured by a photometer

**Table 1** cont.

NH <sub>4</sub> <sup>+</sup>	Colorimetric: The ammonia is determined calorimetrically by Nesslerization. Ammonium forms with Nessler reagent K <sub>2</sub> [HgI <sub>4</sub> ] a yellow-brownish substituted ammonium salt.
NO <sub>3</sub> <sup>-</sup>	Colorimetric: Cadmium metal reduces nitrate in the sample to nitrite. The nitrite ion reacts in an acidic condition with sulfanilic acid to form an intermediate diazonium salt. Next, the salt couples with gentisic acid and an amber coloured solutions formed
Fe	Colorimetric: Iron is reduced to the ferrous state by boiling with acid and hydroxylamine, and treated with 1,10-phenanthroline at pH 3.2 to 3.3. Molecules of phenanthroline chelate atoms of ferrous iron to form an orange-red complex

As is seen in Table 1 the majority of measurements of physicochemical parameters were done by using protocols of study on the basis of international standards. One of an exception is the turbidity test which measures an optical property of water sample. This optical property results from the scattering and absorbing of light by particulate matter present. The amount of turbidity registered is dependent on such variables as the size, shape and refractive properties of the particles. This procedure is calibrated using formazin turbidity standards, and the readings are in terms of FTU (Formazin Turbidity Unit). FTU is equivalent to a nephelometric turbidity unit (NTU) when readings are made on a nephelometer.

### 2.3. Measurements of selected organic parameters of water quality

#### IR spectroscopy

Analyses were performed using a spectrometer Nicolet 6700 FT IR under the following operation parameters: the cells with 3.0 mm light path length with KBr windows, resolution of 4 cm<sup>-1</sup>, number of scans of 20 and spectral range of 2400–3400 cm<sup>-1</sup>. The analysis was performed on the basis of the protocol of PN-82 / C-04565.01. Dichloromethane was used as a solvent. The calibration curves were prepared for the vibrations at 2927 cm<sup>-1</sup> (for aliphatic hydrocarbons C-H, vibration of –CH<sub>2</sub>–) and at 3072 cm<sup>-1</sup> (for aromatic bonds C-H vibration in C<sub>Ar</sub>-H).

#### Chromatographic analysis

The analysis was performed using HP 5890 gas chromatograph equipped with a flame ionization detector (FID) in a splitless mode. The measurements were done under the following conditions: temperature of inlet system of 300°C, septum purge flow rate of 3 cm<sup>3</sup>/min, temperature of FID detector of 320°C, hydrogen and air flow rate through detector of 30 cm<sup>3</sup>/min and 300 cm<sup>3</sup>/min, respectively, nitrogen (carrier gas) flow rate of 5 cm<sup>3</sup>/min, inlet liner 4 mm splitless one trap with quartz wool and chromatography column

of RTX-5 with a length of 60 m, 0.53 mm diameter and 1µm thickness of the stationary phase. Solutions of standards of individual PAHs and BTX were prepared from chemically pure substances. Dichloromethane was used as a solvent.

### 3. RESULTS AND DISCUSSION

The newest Polish regulation, Journal of Laws No 1558, item 155b, of 21 November 2013, Minister of Environment, makes obvious that fully accurate monitoring of any kind of surface and ground water requires hundreds of determinations of various elements. These numerous important elements are divided into four groups *i.e.* biological (*ca.* 30 parameters), hydromorphological, physicochemical with subgroup of specific *non*-synthetic and synthetic contaminations (*ca.* 70 parameters) and finally group of chemical indicators which characterize substances harmful for water environment (*ca.* 50 parameters).

According to the relevant Polish Regulation [16] and the WHO recommendation [18], selected parameters of the investigated in this work waters like turbidity and Fe ions concentration are exceeded in some water samples in comparison to standards for drinking water (see in Table 2). Turbidity is a one of the principal physical attribute of water and is not desirable in drinking water and in many manufacturing uses. High turbidity levels may cause health problems, because it can provide food and shelter for pathogens in the next step their spawning [19]. The values of turbidity are relatively big in almost all water samples. The study area is a hilly agricultural region. Thus, extensive turbidity is caused very probably by suspended solid originate from soil of agricultural land.

As is seen in Table 2, in all investigated water the values of nitrate concentration are quite high and are in the range of 7.0–14.7 mg/L. As a result, amount of nitrates for four samples of flowing waters (streams) exceeded the admissible limit for water of the 1<sup>st</sup> category of cleanness and met with the standard of the 2<sup>nd</sup> category of cleanness. The changes of NO<sub>3</sub><sup>-</sup> concentration in surface waters is predominantly caused by human activities [20, 21]. Nitrogen is an essential nutrient for cultivated plants and influences on crops its high concentration can be harmful to humans and nature. In the agricultural area nitrification of fertilizer and sewage are the main sources of nitrate [22]. Contamination of drinking water by nitrate is an important problem in the modern world, because high concentration of nitrate ions can lead to death of infants by methemoglobinemia or gastric carcinomas [23]. The Members State of the European Union are obligate to respect a special Nitrate Directive dedicated to protection of waters against pollution by nitrates from agricultural sources [24].

Places of oil exploitation are potential source of contamination by hydrocarbons. The pollution by hydrocarbons substances is a big threat for environment. Migration of petroleum substances to the soil and surface or groundwater can cause serious consequences due to the hazardous nature of such chemical substances [25]. The fact that crude oil and petroleum products are complex mixtures of many individual hydrocarbons is a complicating factor in determining the potential risks to water environment.

**Table 2**

The comparison of selected physical and chemical parameters of investigated water samples. All concentration in mg/L

Parameter	Number of sample								MAC for drinking water <sup>A</sup>	MAC for classification of surface water bodies <sup>B</sup>		
	1	2	3	4	5	6	7	8		I class	II class	III/IV/V class
Turbidity [FTU]	6	5	3	8	7	7	30	1	1 NTU	-	-	-
Odor	odorless	odorless	odorless	odorless	odorless	odorless	muddy	odorless	-	-	-	-
pH	7.0	7.0	7.1	7.5	7.5	7.5	7.0	7.1	6.5–9.5	6–8.5	6–9	-
EC [ $\mu$ S/cm]	824	281	850	841	1012	855	418	817	2500 $\mu$ S/cm	$\leq$ 1000	$\leq$ 1500	-
Dissolved oxygen	8.8	5.3	9.0	9.4	7.4	9.3	3.4	7.5	-	$\geq$ 7	$\geq$ 5	-
Chloride	36	12	43	53	155	* <sup>C</sup>	32	37	-	$\leq$ 200	$\leq$ 300	-
Sulfate	70	10	77	63	73	70	5	75	250	$\leq$ 150	$\leq$ 250	-
Nitrate	12.1	14.5	8.9	14.0	7.0	13.0	*	11.4	50	9.7	22.1	-
Ammonia ion	0.13	0.63	0.26	0.22	0.17	0.18	0.15	0.33	0.5	1	2	-
Iron	0.05	0.25	0.12	0.09	0.02	0.08	0.84	0.84	0.2	-	-	-
Calcium	29	38	37	29	46	40	68	132	-	$\leq$ 100	$\leq$ 200	-
Magnesium	110	11	110	111	122	112	17	34	30–12.5	$\leq$ 50	$\leq$ 100	-
Carbonate	60	-	85	-	-	*	-	90	-	-	-	-
Bicarbonate	160	11	160	534	392	*	210	650	-	-	-	-
Aliphatic hydrocarbons	5	6	<5	*	*	<5	6.5	5	-	-	-	-
Aromatic hydrocarbons	<5	<5	<5	*	*	<5	<5	<5	-	-	-	-

<sup>A</sup>MAC (maximal accessible concentration) according to Journal of Laws No. 61, item 417, of 29 March 2007 [16], <sup>B</sup>MAC (maximal accessible concentration) according to Journal of Laws No. 2014, item 1482, of 30 October 2014 [17], <sup>C</sup> some chemical analyses were missed because of insufficient quantity of investigated water samples to provide analyses in triplicates and were denoted in Table 2 as stars (\*), <sup>D</sup> no data.

For that reason, water quality standards did not refer to petroleum products in general and have been established for individual petroleum hydrocarbons. The most dangerous for water environment are the most soluble in water fractions, consisting largely of lower molecular weight aromatic hydrocarbons (e.g., benzene, ethylbenzene, toluene, xylenes, known as BTX) and individual polycyclic aromatic hydrocarbons (e.g., benzo(a)pyrene, benz(a)anthracene, chrysene, fluoranthene, 3,4-benzfluoranthene, 11,12-benzfluoranthene, 3,4-benzpyrene, 1,12-benzpyrene, indeno (1,2,3-cd)pyrene and many others, known as PAHs). These hydrocarbons with a high capacity for bioaccumulation and a low susceptibility to degradation are considered to be ‘permanent organic pollutants’. They also reveal toxic, mutagenic, and carcinogenic properties [25, 26].



**Fig. 2.** Sampling places No. 1 and 3 (phot. Bogumiła Winid)

The investigation by IR spectroscopy shows that water from the streams flowing in the short distance from operating well (sample 1 and 8) are polluted by crude oil (Tab. 2). Figure 2 illustrate sampling place No. 3 which is located above the well, and above the sampling place No. 1. In Figure 2 the plant condition on the bank of investigated stream is also shown. The hydrocarbon pollution was also found in the pond and in surface tank situated in the vicinity of operating well. For sample 7 for which hydrocarbons were detected at the highest level, chromatographic analyze was performed for the content of polycyclic aromatic hydrocarbons (PAHs) and the fraction of benzene, toluene and xylene (BTX).



The results were gathered in Table 3. For comparison, the Table 3 includes also chromatographic data of crude oil sample from the study area. The laboratory results indicate that the content of benzene, toluene and xylene is below the limits for drinking water. On the hand, the environmental quality standards were exceeded for PAHs content. The concentrations of anthracene and fluoranthene are significantly bigger than the maximal accessible content for surface water bodies. Concentration of benzo(g,h,i)perylene (2.1 µg/litre) is also high and exceed environmental quality standards (as a one of the compound of  $\Sigma_{\text{PAH}}$ ).

**Table 3**

Results of the chromatographic analysis of BTX and PAHs. All values in µg/L

Chemical Compounds		Water sample	Crude oil sample	MAC for drinking water <sup>A</sup>	Maximum average concentration per year for surface water bodies <sup>B</sup>	MAC for surface water bodies <sup>B</sup>
BTX	Benzene	0.3	3737.0	1	10	50
	Toluene	0.2	2587.2	700 <sup>C</sup>	–	–
	Xylene	1.3	1343.6	500 <sup>C</sup>	–	–
PAHs	Phenanthrene	0.2	3133.8	–	–	–
	Anthracene	0.8	2305.3	–	0.1	0.4
	Fluoranthene	1.0	2491.2	–	0.1	1.0
	Fluorene	<0.1	2548.3	–	–	–
	Pyrene	<0.1	691.1	–	–	–
	Benz(a)anthracene	<0.1	593.9	–	–	–
	Chrysene	<0.1	660.5	–	–	–
	Benzo(a)pyrene	<0.1	660.5	0.01	0.05	0.1
	Dibenz(a,h)anthracene	8.8	12611.9	–	–	–
	Benzo(g,h,i)perylene	2.1	13136.5	–	–	–
	* $\Sigma_{\text{PAHs}}$ (benzo(g,h,i)perylene, indeno-1,2,3-cd-pyrene, benzo(b)fluoranthene, benzo(k)fluoranthene)	≥2.1	≥13136.5	0.1	0.032	–

A – MAC (maximal accessible concentration) according to Journal of Laws No. 61, item 417, of 29 March 2007 [16],

B – MAC (maximal accessible concentration) according to Journal of Laws No. 2014, item 1482, of 30 October 2014 [17],

C – WHO recommendation [18], \*according [16, 17].

In Table 3 are presented data for a broad family of PAH compound. They differ in toxicity and not all of them are present in the relevant regulations. The sum of PAHs concentration is also one of the water quality parameter, but definition of that parameter is different in different countries. According to the relevant Polish Regulation [16, 17]  $\Sigma_{\text{PAH}}$  is

a sum of concentration of four compounds: benzo(g,h,i)perylene, indeno-1,2,3-cd-pyrene, benzo(b)fluoranthene, benzo(k)fluoranthene (MAC=0.1 µg/litre), on the other hand The WHO Organization mention about sum of six compounds: fluoranthene, 3,4-benzfluoranthene, 1,12-benzfluoranthene, 3,4-benzpyrene, 1,12-benzpyrene and indeno-1,2,3-cd-pyrene (should not, exceed 0.2 µg/litre). Thus, very probably with the passage of time, the regulation of water quality standards will include the larger list of PAH compounds.

#### 4. CONCLUSION

Water for human consumption in Poland is partly produced from surface waters which are strongly affected by anthropogenic pollution. As a result about 18% of the population has tap water not meeting quality standards. The study area of oil exploitation is located in the south of Poland, in the catchment area of the biggest river in the country, the Vistula River. The water for human consumption in this area is mainly produce from surface water (ca. 80%). In that context these surface waters are extremely valuable and should be effectively protected. Moreover, the quality of surface waters in this region can potentially influence on quality of water of the upper course of the Vistula River.

The preliminary investigations of surface water from the study area show the occurrence of contaminations related to the agricultural anthropopression and mining industry. The most hazardous seems to be the pollution by polycyclic aromatic hydrocarbons.

The correct assessment of risk associated with measured pollutions in the study area requires more active monitoring of surface waters during the whole annual cycle and collection of biggest amount of samples for laboratory examination. This monitoring should include not only the identification and the determination of concentration of the contaminating substances but also the mobility of the pollutions.

*The authors also thank Michał Maruta for preparation of Figure 1.*

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