

Approbation of express-method for benzo[a]pyrene extraction from soils in the technogenic emission zone territories

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Abstract

The benzo[a]pyrene accumulation and migration regularities in chernozemic soils under the aerotechnogenic emission zone were revealed on the basis of long-term monitoring researches. A new method of subcritical water extraction has been developed for determination of benzo[a]pyrene from soils of the emissions zone of Novocherkassk Power Station one of the largest thermal power enterprises in Russian Federation. It is shown that the soils adjacent to a source of emission are polluted by benzo[a]pyrene at distance to 5 km. Trends in the accumulation of benzo[a]pyrene in soil zones of the thermal power plant influence have been researched over a 5-year period of monitoring observations. The assessment of soils pollution extent by benzo[a]pyrene is given.

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Introduction

Polycyclic aromatic hydrocarbons (PAHs) are a typical class of persistent organic compounds derived from natural sources such as forest fires or/and anthropogenic processes such as urban and industrial activities, and are prevalent in the environment. Sixteen PAHs compounds have been recommended as priority pollutants by the United States Environmental Protection Agency (USEPA) because of their carcinogenicity, mutagenicity, and toxicity (Anonymous, 2002; GOST, 2004; Jian, 2004; Wenzl et al., 2006). These compounds are likely to accumulate in soils for many years because of their persistence and hydrophobicity. As a result, soils may be an important reservoir of PAHs (Khoei et al., 2013; Tumin et al., 2013; Tuan Habibet al., 2009). The assessment of soils contamination is one of the major indicators in the environmental monitoring system. The main marker of soils pollution by PAHs is benzo[a]pyrene (BaP) (Gennadiev et al., 2007). Knowledge of soil contamination with BaP is needed to minimize the risk of human exposure and of environmental contamination (Oshunsanya, 2010; El-Batrawy, 2013).

Enterprises of power energy are the active sources of polycyclic aromatic hydrocarbons (PAHs) environmental pollution. The most powerful enterprise of power complex in Rostov region is coal-fired Novocherkassk's power station (NPS). The total amount of emissions takes more than 90000 tons per year,

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about 10 percent from them are PAHs. Researches shows us that individual heating systems represent more powerful source of PAHs environmental emissions than transport (Maliszewska-Kordybach et al., 2009; Khan et al., 2008).

The research object was the soil in the emissions zone of NPS. Researches of the environment of NPS emissions zone from 2001 to 2012 showed that this enterprise, one of the largest thermal power plants of Russia working since 1965 - 1971, is the main pollutant contribution in Rostov region (Antizar-Ladislao et al., 2006; Gorobtsova et al., 2005; Anonymous, 2012). An extremely negative influence on the environment in the city and adjacent territories also amplifies the fuel used at the enterprise, which don't conforms to quality requirements (Bogush et al., 1998). The station annually consumes over 4,5-4,7 million tons of coal, 0,7 million tons of fuel oil and 380 million m³ of gas. Coal arrives at the station without processing at concentrating factories. Actually NPS uses coal which has an ash-content and the sulfur content considerably exceeding projected. Coal is enriched with a wide range of heavy metals and toxic elements (Cu, Zn, Hg, Pb, Mn, As, etc.), and also radionuclides (Th, Sr, etc.) (Bogush et al., 1998; Minkina et al., 2014).

The main components of NPS's emissions are ashes, sulphurous anhydride, nitrogen oxides, soot (over 30 tons/year), vanadium pentoxide (about 8 tons/year), iron oxide (over 5 tons/year), chromic anhydride (about 0,1 tons/year), fluoric hydrogen (7 kg/year), etc. Up to 85% of chemical elements containing in initial coal remains in ashes.

The purpose of research is the assessment of BaP content in soils of emission zone of the power complex enterprises in soils with use of ecologically clean and effective extraction method.

Material and Methods

Studies were conducted on the soils of monitoring plots subjected to NPS emissions. In 2000, monitoring plots were established at different distances from the NPS (1.0–20.0 km). They coincided with the air sampling sites for the ecological certificate of the plant (plots 1, 2, 3, 5, 6, 7) (Figure 1). The most attention was paid to the main wind direction from the contamination source to the northwest through the residential areas of Novocherkassk (zones 4, 8, 9, 10). The monitoring plots were located on virgin lands or fallow areas. Soil samples for the determination of soil properties and the contents of BaP were taken from a depth of 0–20 cm. The soil cover in the region under study consisted of ordinary chernozems, meadow-chernozemic soils, and alluvial meadow soils (Table 1).

Table 1. Properties of NPS emissions zone soils (an average for 2008-2011gg.)

| Number of monitoring plot | Soil | Physical clay, % | Clay, % | C _{org} , % | pH | CaCO ₃ , % | CEC, mmol(+) 100g ⁻¹ |
|---------------------------|---|------------------|---------|----------------------|-----|-----------------------|------------------------------------|
| 1 | Low-humus medium-thick calcareous clay loamy ordinary chernozem on loess-like loams | 52 | 27 | 4,3 | 7,6 | 0,5 | 35,0 |
| 2 | Low-humus calcareous sandy alluvial meadow soil on alluvial deposits | 7 | 3 | 3,1 | 7,5 | 0,4 | 10,9 |
| 3 | Low-humus silty clayey flood-plain meadowchernozemic soil on alluvial deposits | 67 | 37 | 4,6 | 7,3 | 0,2 | 44,8 |
| 4 | Low-humus medium-thick calcareous clay loamy ordinary chernozem on loess-like loams | 55 | 29 | 4,6 | 7,5 | 0,7 | 31,2 |
| 5 | Low-humus medium-thick calcareous clay loamy ordinary chernozem on loess-like loams | 53 | 27 | 4,3 | 7,5 | 1,0 | 35,7 |
| 6 | Low-humus medium-thick clay loamy meadowchernozemic soil on loess-like loams | 55 | 30 | 4,1 | 7,7 | 0,8 | 32,4 |
| 7 | Low-humus medium-thick calcareous clay loamy ordinary chernozem on loess-like loams | 51 | 27 | 4,1 | 7,6 | 0,7 | 31,3 |
| 8 | Low-humus medium-thick clay loamy meadowchernozemic soil on loess-like loams | 60 | 32 | 5,0 | 7,4 | 0,4 | 47,6 |
| 9 | Low-humus medium-thick calcareous clay loamy ordinary chernozem on loess-like loams | 52 | 30 | 4,2 | 7,6 | 0,6 | 31,4 |
| 10 | Low-humus medium-thick calcareous clay loamy ordinary chernozem on loess-like loams | 53 | 28 | 4,6 | 7,6 | 0,5 | 36,0 |

The majority of the soils of monitoring plots were ordinary chernozems; the soils of plots 9 and 10 were used as control soils. The low-humus calcareous sandy alluvial meadow soil (plot 2), which had a light texture and a low cation exchange capacity (CEC), and the low-humus silty-clayey meadow-chernozemic floodplain soil (plot 3) with a high CEC that differed from the control soils. These differences are considered in the discussion of the results.



Figure 1. Schematic map of monitoring plots in the zone affected by the Novocherkassk power station

| Plot No. | The direction and distance from NPS | Plot No. | The direction and distance from NPS |
|----------|-------------------------------------|----------|-------------------------------------|
| 1 | 1 km on the northeast | 6 | 2,0 km on the northwest |
| 2 | 3 km on the southwest | 7 | 1,5 km to the north |
| 3 | 2,7 km on the southwest | 8 | 5 km on the northwest |
| 4 | 1,6 km on the northwest | 9 | 15 km on the northwest |
| 5 | 1,2 km on the northwest | 10 | 20 km on the northwest |

According to the prevailing direction on the district of a wind rose the main direction of distribution of NPS atmospheric emissions was defined. It is a zone located on a straight line from a source of pollution through inhabited territories of Novocherkassk and Krivyanskiy village. Samples were collected in soils of monitoring platforms No. 4, No. 8, No. 9, No. 10, in the area of the prevailing direction of a wind rose. The requirement for the location of the monitoring platforms is the existence of a virgin soil cover or deposits (not subjected to technical processing).

The samples were collected layer-by-layer for the BaP content definition in soils. Soil samples were selected and prepared for the chemical analysis according to GOST 17.4.4.02-84 (GOST, 2004) requirements. The soils cover of the monitoring territory is presented by ordinary carbonate heavy loamy chernozem, grassland-chernozems heavy and medium loamy, alluvial types of a flood plain of river Tuzlov (Table 1).

The most part of the territory is occupied by ordinary chernozems revealed the following physical and chemical properties: a clay content of 286 g kg⁻¹ and a physical clay content of 471 g kg⁻¹, pH of 7.3, organic C content of 23 g kg⁻¹, CaCO₃ content of 1 g kg⁻¹, CEC of 37.1 mM kg⁻¹ and exchangeable Ca, Mg and Na contents of 29.5, 5.5 and 0.1 mM kg⁻¹, respectively.

Solvents and reagents were HPLC grade and included ethanol (96%, analytical grade), *n*-hexane (99%, analytical grade), potassium hydrate (98%, analytical grade), acetonitrile (99.9%, analytical grade), NaOH (97%, analytical grade), and anhydrous Na₂SO₄ (purchased from Aquatest, Rostov on Don, Russia). A BaP

standard solution in acetonitrile (Sigma-Aldrich, Germany) was used to prepare standards for HPLC analyses.

BaP extraction from soils was carried out by a subcritical water extraction method (Sushkova et al., 2013; Sushkova et al., 2014). The sequence of operations when carrying out the analysis is schematically shown in Figure 2. Subcritical water extraction of BaP from soil samples was conducted in a specially developed extraction cartridge made of stainless steel and equipped with screw-on caps at both ends. It was also equipped with a manometer that included a valve for pressure release to maintain an internal pressure of 100 atm. The extraction cartridge containing a sample and water was placed into an oven connected to a temperature regulator.

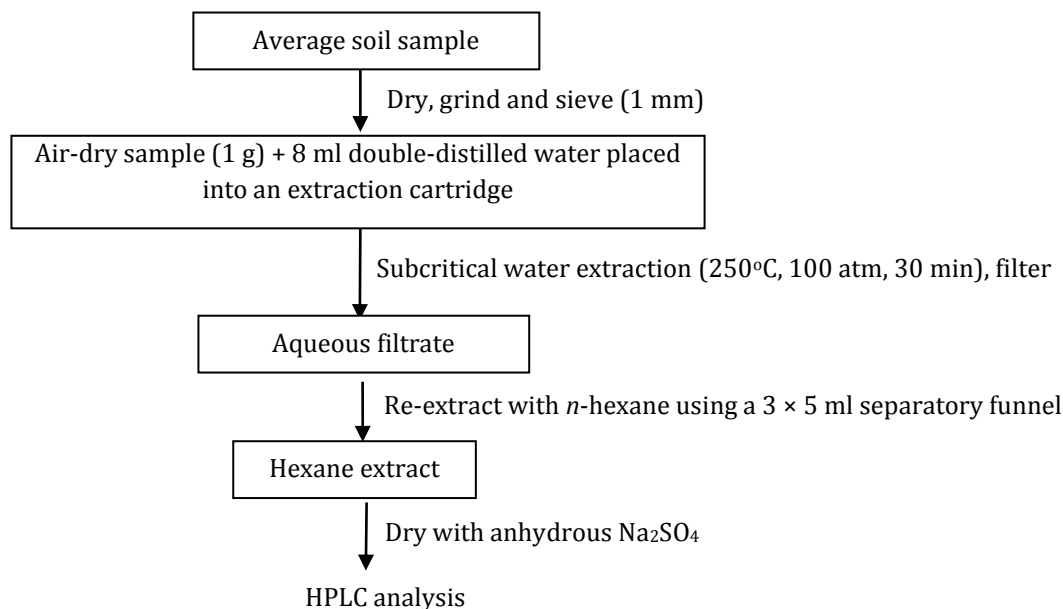


Figure 2. Scheme of BaP analysis in soil material using subcritical water extraction and HPLC analyses of the extract

The process of BaP analyses in soils based on subcritical water extraction consisted of the following step-by-step operations. An air-dried sample of the soil was ground in a porcelain mortar and passed through a 1 mm sieve. One gram of sample was placed into the extraction cartridge and 8 ml of double-distilled water was added. The extraction cartridge was sealed from both sides with the screw caps. The cartridge was placed into an oven held at 230, 240, 250, 260 or 270°C for 20, 30 or 40 min. Subsequent extractions were conducted under optimum conditions (30 min at 250°C and 100 atm). After cooling, the content of the cartridge was filtered (Whatman no. 1) into a conical glass flask and washed with 2 ml of double-distilled water. This operation was repeated two or three times, until the filtrate was clear. The aqueous extract was re-extracted three times with 5 ml of *n*-hexane by shaking for 15 min in a separatory funnel. The hexane extracts were combined and filtered through anhydrous Na₂SO₄ and evaporated to dryness in a pear-shaped flask on a vacuum evaporator in a 40°C water bath. The residue was dissolved in 1 ml of acetonitrile by shaking for 30 min. The BaP concentration in the acetonitrile extract was determined by HPLC.

The efficiency of BaP extraction from soil was determined using a matrix spike. The air-dried vegetation sample (1 g) was placed into a round-bottom flask and BaP standard solution in acetonitrile was added to give BaP concentrations of 2, 4, 6, 8, 16 or 32 ng/g. After evaporating the solvent for 30 min under a hood under ambient conditions, the BaP-spiked soil samples were incubated for 24 h at 7°C. The samples were then analyzed by the subcritical extraction method described above.

BaP in the extracts was quantified by HPLC (Model 2000, Thermo Separation Products, Waltham, MA, USA) with simultaneous ultraviolet (UV-1000) and fluorescence (FL-3000) detection following ISO 13877 requirements (ISO 13877-2005, 2005). The BaP peak on chromatograms of soil sample extracts was identified by comparing retention time to that of the analytical standard sample using the two detectors. The limit of BaP detection and quantification were determined using standard solutions and calibration curves. A calibration standard was inserted after every six samples to correct for drift in retention time within a run.

BaP concentrations in soil samples (A, ng/g) were calculated as follows:

$$A = k SI \times Cst \times V / (Sst \times m) \quad (1)$$

where Sst and SI = respective areas of BaP peaks in chromatograms of standard and sample solutions; Cst = BaP concentration in standard solution (ng/ml); k = coefficient of BaP recovery from a sample; V = volume of acetonitrile extract used for HPLC (ml); m = mass of the sample (g). Data handling and statistical analyses were conducted using Microsoft EXCEL.

Soil properties were analyzed using Russian standard methods (Sokolov, 1975) (Table 1). Soil organic carbon was measured using 0.4 N potassium bichromate (the Tyurin method modified by Simakov). Soil particle size distribution (silt and clay content) was determined by the pipette method after the pyrophosphate treatment. Cation exchange capacity (CEC) of the soil was determined using 1M ammonium chloride (the Bobko–Askinazi method). The exchangeable potassium was determined by the Machigin (molybdenum blue) method. Adsorbed Na was analyzed by flame atomic adsorption spectrophotometry. Soil pH was measured with a pH electrode using a 1:5 suspension of a soil to water ratio. Exchangeable calcium and magnesium were measured by the titration at pH 12.5-13 and 10 respectively. Carbonates were measured by the Kudrin method using 0.005 NH₂SO₄ and then an excess of the acid was titrated with alkali.

Results and Discussion

An early researches of the ecological condition of the NSP zone showed us that the most dangerous pollutants are BaP and heavy metals (Pinsky and Minkina, 2013; Minkina et al., 2014). The content of BaP in all objects of the ecosystem must be under obligatory control. However monitoring researches of BaP content are conducted by Committee of environment and natural resources only within the settlement of Novochoerkassk. It doesn't demonstrate an overall pollution extent of all NPS emissions area (Anonymous, 2012).

Monitoring researches of BaP content in soils from 2008 to 2011 allow to identify the main trends in pollution by NPS emissions in such important components as the impact zone of the soil (Table 2). Data analysis within 4 years established that the number of defined compound in soils vary widely (Table 2). The main reason for the variation of the absolute values of heavy metal contents in soils adjacent to NPS can serve as a different load on the power plant energy units, which worked at various power in different years of observations. Every year emissions to the atmosphere changed. Two from nine NPS power units were transferred to natural gas from 2007 to 2008. It reduced emissions of BaP in the atmosphere practically twice. Since 2010 the enterprise has been actively working on installing purification filters for catching emissions, also the amount of anthracite coal which used as fuel at the power plant is reduced, transition to natural gas is occurring (Anonymous, 2012).

The accumulation of BaP in the investigated steppe ecosystem occurred as a result of precipitation of solid emissions of NPS in the neighborhood and was dependent on the prevailing wind direction and consumption of vegetation. Should be noted the similar regularities by years in BaP accumulation in the test soils which were taken from the zone of greatest technogenic NPS emissions (Table 2).

The main accumulation of pollutant in 20 cm layer of soils is noted directly in affected zone. These are soils from the territory where plots No. 4, 5, 8, 9, and 10 (Table 2) are located. At the same time, some increasing of the BaP content is observed in soils at distance of 20 km from state district power station that is connected with proximity of a site to the highway.

Thus within the observation period in the area there is an active reduction of BaP content in the surface layer of the soil, especially in nearby soils to the enterprise. It can be caused by decrease of emissions of polluting components from enterprise.

The maximum quantity of a pollutant was founded in the soil of the plot No. 4 located mostly close to a source of pollution in the direction of prevailing winds. Value from 2008 to 2009 reached to 275,6 ng/g in 5 cm soil layer and 117,8 ng/g in 5-20 cm soil layer that exceeded the BaP maximum concentration limit level in the soil up to 14 times (Table 2). The BaP level in a 5 cm soil layer gradually decreased Since 2010 the quantity of a pollutant in the soil of the most polluted platform decreased 84,2 ng/g and in 2011 it consist 62,4 ng/g in a 5-cm layer.

Table 2. The BaP content in soils of monitoring plots for 2008 – 2011 years

| Number of monitoring plots, direction and distance from NPS | Selection depth, cm | BaP content, ng/g | | | |
|---|---------------------|-------------------|-----------|----------|----------|
| | | 2008 | 2009 | 2010 | 2011 |
| 1. 1,0 Northeast | 0-5 | 62,7±3,4 | 72,3±1,7 | 54,9±1,4 | 24,0±0,9 |
| | 5-20 | 24,8±1,1 | 23,9±1,7 | 26,6±0,9 | 17,1±2,1 |
| 2. 3,0 Southwest | 0-5 | 28,0±1,3 | 18,2±1,4 | 18,4±1,6 | 22,7±0,1 |
| | 5-20 | 27,3±2,1 | 26,3 ±2,1 | 24,7±2,4 | 19,4±1,8 |
| 3. 2,7 Southwest | 0-5 | 69,5±3,3 | 123,9±6,7 | 57,3±4,7 | 24,5±2,7 |
| | 5-20 | 18,4±0,6 | 62,8±5,4 | 19,8±1,5 | 8,6±1,2 |
| 4. 1,6 Northwest | 0-5 | 182,5±3,5 | 275,6±9,2 | 84,2±6,8 | 62,4±5,8 |
| | 5-20 | 113,9±16,4 | 117,8±7,6 | 47,8±3,8 | 37,1±2,6 |
| 5. 1,2 Northwest | 0-5 | 118,5±4,0 | 138,1±8,6 | 65,4±3,4 | 45,4±4,3 |
| | 5-20 | 72,8±8,2 | 92,1±3,7 | 45,3±2,0 | 35,9±2,8 |
| 6. 2,0 Northwest | 0-5 | 46,6±2,8 | 67,4±2,8 | 32,4±1,4 | 34,3±2,6 |
| | 5-20 | 25,7±1,5 | 36,7±2,2 | 16,2±1,5 | 8,1±0,5 |
| 7. 1,5 North | 0-5 | 38,5±2,1 | 87,6±3,7 | 39,5±2,4 | 36,4±2,3 |
| | 5-20 | 17,3±1,7 | 35,9±2,1 | 14,4±1,5 | 16,3±0,4 |
| 8. 5,0 Northwest | 0-5 | 60,3±3,4 | 82,4±3,6 | 47,8±3,0 | 35,9±1,6 |
| | 5-20 | 41,7±1,7 | 28,4±1,7 | 28,6±2,7 | 24,6±2,1 |
| 9. 15,0 Northwest | 0-5 | 27,4±0,9 | 25,6±2,6 | 18,4±1,2 | 13,0±0,7 |
| | 5-20 | 11,8±0,3 | 18,4±0,7 | 11,9±0,7 | 11,9±0,4 |
| 10. 20,0 Northwest | 0-5 | 26,9±1,5 | 48,0±4,2 | 28,6±2,2 | 12,1±0,8 |
| | 5-20 | 17,4±0,7 | 32,5±3,2 | 14,1±0,7 | 7,5±0,6 |

Similar regularities are observed in changes of pollutant concentrations that proceeded almost synchronously in the soil in 0-5 and 5-20 cm layers (Table 2). However in the majority of soils their concentration in an underlying layer was lower. Decreasing in BaP content in 5-20 cm layer of soil was recorded in soils of all monitoring plots for the entire period of research. Superficial accumulation of BaP prevails in soils of researched territories. Decrease in BaP concentration in a layer of 5-20 cm on the average was caused by low solubility in water of studied carcinogen and its weak mobility (Khan et al., 2008; Gennadiev et al., 2007). The exception is made by the sandy alluvial soil of plot No. 2 that points to the increased migratory ability of BaP to underlying layers of this plot soil.

Conclusion

Thus, a method of BaP determination in soils has been approved as based upon the subcritical water extraction under the optimum conditions. The efficiency of the given method involves the use of subcritical water as an environmentally friendly solvent, a shorter time for analysis. BaP distribution and accumulation tendencies were investigated during the 4 years of monitoring researches in studied soils. The main factor of technogenic influence on the soil the investigated area are toxic emissions of the enterprise of a power complex - NPS; transport exhausts can be sources of BaP additional issue. Gradual decrease in the pollutant content in soils of the studied territory was established for 4 years period of supervision. It was explained by considerable reduction of volumes of polluting substances emission by the enterprise. Despite the environmental activities of in the enterprise, the impact of emissions on the environment location nearby today is still primary one.

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