



Chemical characterization of PM10 in two small towns located in South Poland

Anna Turek-Fijak ,
Joanna Brania,
Katarzyna Styszko ,
Damian Zięba,
Zdzisław Stęgowski ,
Lucyna Samek

Abstract. The purpose of this study is to analyse the elements and PM10 concentrations in air samples gathered in the winter of 2017/2018 in two small towns, namely Skala and Wadowice. The chemical elements were identified for each sample using the energy dispersive X-ray method. The spectrometer was equipped, among others, with an Mo-X-ray tube which was the source of the photons and the Si(Li) detector. The following chemical elements: Cl, K, Fe, Ca, Zn, Pb, Br, Ti, Cu, Mn, V, Co, Rb, Ni, Sr, and Cr were identified in the samples. In addition, As and Se were identified in Wadowice. First, the results were compared with each other and then with the results for the nearest city. It was observed that the PM10 concentrations were significantly higher than the UE limit value for PM10, which equals $50 \mu\text{g}\cdot\text{m}^{-3}$ per 24 h. Moreover, the high concentrations of, among others, K, Pb, Cl or Zn, are likely to be linked with fossil fuels combustion and biomass burning. The levels of element concentrations in Wadowice and Skala resemble the levels observed several years earlier in Krakow.

Keywords: Air pollution • EDXFR fluorescence • PM10

Introduction

Air pollution is one of the scourges of contemporary cities, and numerous researches have demonstrated that poor air quality has a significant impact on human health. The World Health Organization (WHO) has estimated that the rates of human mortality and morbidity caused by exposure to air pollution are on the rise [1, 2]. This danger is linked, among other reasons, to the presence of airborne particulate matter (APM), which can be defined as a mixture of liquids and solid particulates. APM has both natural and anthropogenic sources. In this article, PM10 will be considered mainly to be the pollution caused by human activity like transport, households, and industry [3, 4].

In Poland, the problem of air pollution is severe. Numerous studies carried out in urban areas indicate that the air quality in Poland is one of the worst in the European Union (EU) [5]. The EU limit value for PM10 is $50 \text{mg}\cdot\text{m}^{-3}$ per 24 h [6], but this limit is often exceeded, primarily during the cold season. This is mainly caused by nonindustrial combustion processes like the burning of fossil fuels and biomass, especially during the winter [7, 8]. Moreover, adverse and windless weather has an additional impact on poor air quality. The problem of air pollution is also present in small towns, but unfortunately, the exact data concerning the air situation there is limited. However, research on air pollution

A. Turek-Fijak, J. Brania, D. Zięba, Z. Stęgowski,
L. Samek

Faculty of Physics and Applied Computer Science
AGH University of Science and Technology
Mickiewicza 30 Ave., 30-059 Krakow, Poland
E-mail: lucyna.samek@fis.agh.edu.pl

K. Styszko
Faculty of Energy and Fuels
AGH University of Science and Technology
Mickiewicza 30 Ave., 30-059 Krakow, Poland

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in small towns has been conducted and results have proven that the problem of air quality is present [9]. Further, air pollution in Polish cities in the south of the country, regardless of their size, is a serious issue [9]. More research on air pollution in small towns is needed, especially, not only on the concentration of PM, but also on its composition. PM consists mainly of carbon, elements, cations, anions, and organic compounds. The understanding of the chemical content of PM provides us with information about its health implications and sources [1].

This study aims to investigate and compare the elements and PM₁₀ concentrations for two small cities in winter. Due to the analysis of PM content and the particles' size, we can identify sources of pollution and estimate the risk to people and the environment. The following element concentrations: Cl, K, Fe, Ca, Zn, Pb, Br, Ti, Cu, Mn, V, Co, Rb, Ni, As, Se, Sr, and Cr were determined for each sample using the X-ray fluorescence method. This paper presents, in the tables and graphs, low limit detection, and elemental and PM₁₀ concentrations, including uncertainties. Element concentrations in two small towns were associated with potential sources of pollution and compared with the results relating to element concentration in the nearest large city – Krakow.

Experimental

Sampling areas

The sampling campaigns were conducted in two towns: Skala and Wadowice.

Skala is a small town located 50°25'N and 19°85'E, between the Ojców National Park in the southwest and the Dłubnia Landscape Park in the southeast. It covers an area of 2.97 km² and has a population of 3801 people. Skala is located on the slightly undulating Jurassic plateau. In its upper layer, Jurassic limestone and chalk marl covered with clay can be found. Moreover, the city is directly adjacent to the Ojców National Park, where chalk deposits are located. Skala is a densely built-up commercial and residential area, located at a junction of major roads. Heating is provided mainly by domestic furnaces, tiled stoves, and electrical devices.

Wadowice is a town situated in the valley between the hills of the Pogorze and the Beskid Mały ranges. It covers an area of 10.6 km² with a population of over 18 600 (49°53'N and 19°30'E). The area is diverse in terms of altitude, because the difference between the highest and the lowest point is about 50 m. Wadowice is a strategic spot in the region, due to the presence of local initiatives: administration, culture, and education centers. The town is also located at a junction of several major roads. Heating is provided by furnaces and power plants.

In accordance with the information presented above, the potential sources of air pollution can be as follows:

- burning of fossil fuels by transport and households (low-emission);
- industrial plants and factories;

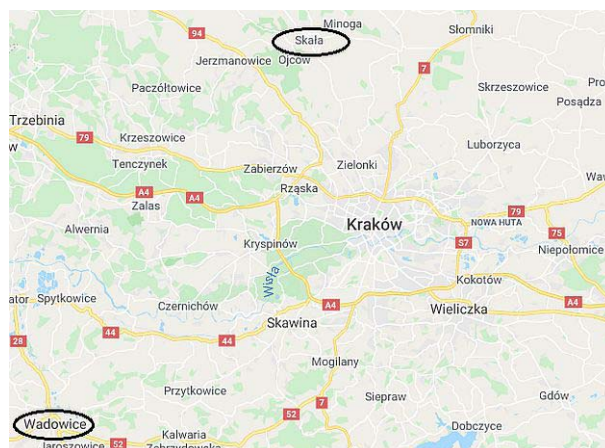


Fig. 1. Map with sampling sites.

- the re-suspension of road dust;
- neighbouring towns or villages;
- other sources.

Sampling site

The samples were collected between 22 December 2016 and 23 January 2017 in Skala and between 24 January and 20 February 2017 in Wadowice. All samples were collected in the town centers. The sites and the location of the study areas are shown in Fig. 1.

In order to collect the PM₁₀ samples, quartz fiber filters (Whatman, 47 mm) were used. The samples were taken using a low-volume sampler, PNS-15. The sampling time was 24 h for all samples. Before and after the sampling, the filters were conditioned, weighed (microbalance, A&D HM-202-EC), and stored in specific circumstances (preserving the right temperature and humidity conditions respectively at $20 \pm 1^\circ\text{C}$ and $50 \pm 5\%$). The mass was used to calculate the concentration of PM₁₀, pre and postsampling. Filters were stored in a freezer at -20°C until analysis.

Elemental analysis

The samples were analysed using the energy dispersive X-ray fluorescence (EDXRF) spectrometer available in the laboratory at the Faculty of Physics and Applied Computer Science at AGH University of Science and Technology in Krakow. EDXRF is a nondestructive analytical technique used for measuring element concentrations in the samples.

A multifunctional EDXRF spectrometer was used in this study. This instrument consisted of an Mo-X-ray tube which was the source of photons: the Si(Li) detector with a 170 eV resolution at 5.9 keV, a specimen chamber, and the necessary electronics with a PC. Due to the fact that the X-ray tube had a molybdenum anode, a Mo secondary target was used to gain monochromatic X-ray beams. The excitation energy of Mo is 17.4 keV, which means that the elements must have lower excitation energy. The specifications of the tube were as follows: voltage of 55 kV and current of 30 mA. The measurement time for one sample was 10000 s.

The qualitative and quantitative analysis was conducted using AXIL software. This software was used to analyse the X-ray spectrum and gain necessary information to calculate the element concentrations, uncertainty, and the limit of detection.

The calibration of the spectrometer was performed prior to the time of taking the measurements of the PM10 samples. Calibration was conducted using Micromatter USA thin-film standards. By using AXIL software, the spectra were fitted to a Gaussian curve to obtain the area under peak. Subsequently, sensitivity was established. On the basis of the area under peak and sensitivity, the concentrations were determined.

The results of calibration were satisfactory; the results were verified by analysis of the National Institute of Standards and Technology (NIST) Standard Reference Material (SRM). The measured results of the SRM 2783 were compared with the certified values (Table 1), which were equal to the certified values within the measured uncertainties. Only the measured value for Zn was equal to the certified values within extended uncertainties.

Spectra for samples were fitted to a Gaussian curve to obtain the area under peak. The quantitative analysis was conducted using parameters such as area under peak and sensitivity. Figures 2 and 3 indicate the EDXRF spectra of PM10 samples collected in Skala and Wadowice, respectively.

Results and discussion

The results of the element concentrations are shown in Table 2. Figures 4 and 5 show the values of PM concentrations and the red line shows the limit value for PM10. Uncertainty for PM10 concentrations is approximately 10%.

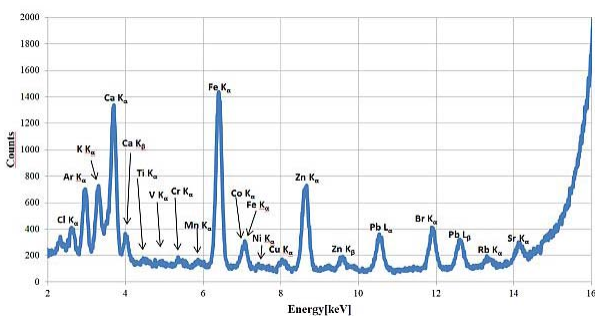


Fig. 2. EDXRF spectra of sample collected in Skala.

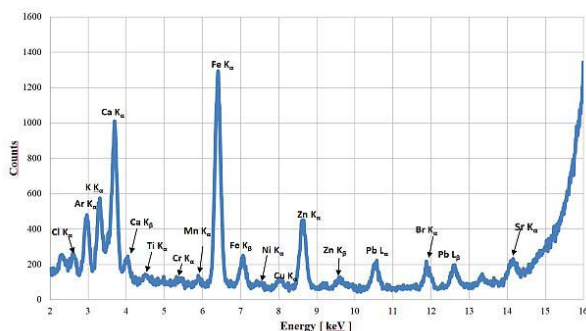


Fig. 3. EDXRF spectra of sample collected in Wadowice.

Table 1. The measured and certified values – SRM 2783

Element	Measured values (ng)	Certified values (ng)
K	6 120 ± 4 550	5 280 ± 250
Ca	11 240 ± 3 540	13 200 ± 1 700
Ti	1 400 ± 170	1 490 ± 240
Cr	150 ± 85	135 ± 25
Mn	284 ± 50	320 ± 12
Fe	24 800 ± 1 910	26 500 ± 1 600
Cu	480 ± 70	404 ± 42
Zn	2 350 ± 400	1 790 ± 150
Pb	310 ± 64	317 ± 54

According to laws enacted by the WHO and the EU, the limit value for PM10 is 50 µg·m⁻³ per 24 h. In Skala, the EU limit was exceeded for 18 days out of 30 during which the samples were collected, whereas in Wadowice the EU limit was exceeded on all the days with the exception of one day. Further, the highest concentration of PM10 was 406 µg·m⁻³ for Wadowice and 301 µg·m⁻³ for Skala. The concentration of PM10 of more than 150 µg·m⁻³ (three times above the limit) occurred 15 times in Wadowice and five times in Skala. The concentration which did not exceed the limit occurred only once in Wadowice and 11 times in Skala.

The mean values of element concentration for Cl, Ca, K, Ti, Mn, Fe, Co, Cu, Zn, Br, Rb in Wadowice is higher than the values in Skala; some of the elements such as Sr and Cr have higher mean values in Skala, but Ni, V, and Pb have a similar concentration in both of these towns.

High concentrations of chemical elements such as Cl, K, Pb, Ca, Ni, V, and As were observed; these elements are connected with fossil fuels combustion [10], whereas K is typical for wood burning or biomass burning [11].

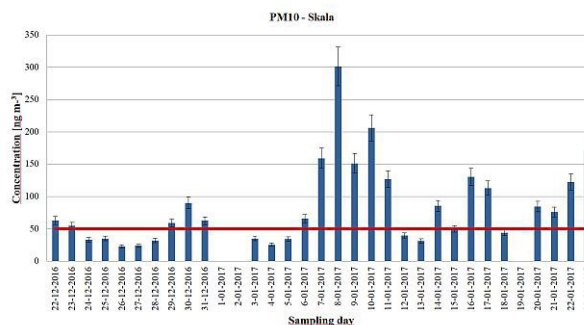


Fig. 4. PM10 concentration for Skala.

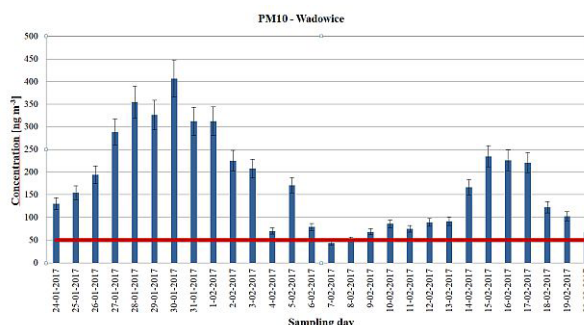


Fig. 5. PM10 concentration for Wadowice.

Table 2. Average PM₁₀ ($\mu\text{g}\cdot\text{m}^{-3}$) and elemental concentrations in Wadowice and Skala ($\text{ng}\cdot\text{m}^{-3}$) with low limit of detection ($\text{ng}\cdot\text{m}^{-3}$)

PM ₁₀	Wadowice			Skala			LLD
	Min	Max	Mean	Min	Max	Mean	
	43	407	174	23	301	84	
Element	Min	Max	Mean \pm SD	Min	Max	Mean \pm SD	
Cl	407	23 849	9 228 \pm 6 200	362	17 733	4 268 \pm 2 740	144
K	467	2 544	1 400 \pm 540	223	1 584	718 \pm 270	33
Ca	190	2 697	590 \pm 290	191	754	296 \pm 83	23
Ti	25	139	56 \pm 18	17.26	64.39	29 \pm 7.1	13
V	10	20	15 \pm 2.5	11.43	44.86	17.5 \pm 3.7	9.6
Cr	1.02	10	4 \pm 1.6	4.06	25.32	9.4 \pm 3.1	1.4
Mn	8.62	81	27 \pm 14	6.09	34.26	12.3 \pm 4.1	5.8
Fe	157	2 098	760 \pm 400	67	1 004	308 \pm 150	3.5
Co	5.84	14	8.6 \pm 1.7	4.06	7.11	5.46 \pm 0.71	3.9
Ni	2.29	11	4.2 \pm 1.8	1.52	5.08	3.09 \pm 0.79	2.0
Cu	5.08	62	27 \pm 16	2.29	17.75	8.3 \pm 4	2.1
Zn	93	749	360 \pm 180	57	723	207 \pm 110	3.3
As	2.54	20	11 \pm 4.7	–	–	–	–
Se	2.54	6	4.4 \pm 1.1	–	–	–	–
Br	10	246	95 \pm 63	12.2	193.5	53 \pm 30	2.1
Rb	2.79	14	7 \pm 2.3	1.52	6.59	3.3 \pm 1.3	1.7
Sr	0.51	11	4.2 \pm 2	4.57	30.39	8.3 \pm 2.9	0.4
Pb	28	286	120 \pm 64	34.3	357.4	98 \pm 50	1.9

SD: standard deviation – the variability of concentrations during the measuring period.

Zn and Pb can be associated with the presence of metal works, factories, or smelters located in Wadowice and in the neighbouring towns (e.g., Andrychow) [10]. This also confirms the presence of Mn, As, Se, Cr, Ni, and Co [3, 12]. Further, industrial factors significantly affect air quality, particularly in Wadowice, which is a larger industrial area as compared to Skala. On the other hand, in Skala, the main source of pollution is the combustion of

fossil fuels and the transport of aerosols from other towns [13]. The levels of element concentration are higher in Wadowice than in Skala, since the former is a larger town with more sources of pollution.

Furthermore, both in Wadowice and Skala, there is significant car traffic which explains the presence of elements originating from traffic emission: Fe, Cu, and Rb from brake and engine wear, and Zn from tires [14, 15]. Exhaust emissions from motor

Table 3. Comparison of PM₁₀ ($\mu\text{g}\cdot\text{m}^{-3}$) and the chemical element concentrations for Wadowice, Skala, and big cities ($\text{ng}\cdot\text{m}^{-3}$)

Element	Wadowice	Skala	Krakow 2010 [9]	Krakow 2011 [16]	Krakow 2012 [16]	Krakow 2010 [17]	Krakow 2010 [17]	London 2012 [18]
PM ₁₀	174	84	–	93	40	124	92	–
Cl	9 228	4 268	–	–	1 040	–	–	790
K	1 400	718	1 042	386	193	737	339	36.2
Ca	590	296	1 216	452	918	858	342	202
Ti	56	29	52	66	17	66	63	7.5
V	15	17.5	–	–	–	–	–	2.2
Cr	4	9.4	66	–	–	47	17	6.3
Mn	27	12.3	164	52	16	118	39	9.4
Fe	760	308	4 350	1 300	507	2 960	743	693
Co	8.6	5.46	–	–	–	–	–	–
Ni	4.2	3.09	–	<LLD	<LLD	–	–	2.1
Cu	27	8.3	24.6	23	8.7	31	23	26
Zn	360	207	442	176	141	422	223	10.9
As	11	–	21	8	<LLD	–	–	–
Se	4.4	–	–	–	–	–	–	–
Br	95	53	61	33	49	51	37	2.3
Rb	7	3.3	–	<LLD	<LLD	–	–	–
Sr	4.2	8.3	–	4.5	43.3	–	–	–
Pb	120	98	121	52	41	108	68	1.6

<LLD: the value is below the detection limit.

vehicles are connected with the occurrence of, in particular, Br, V, Zn, and Cu [15]. In addition, Ti and Sr may come from re-suspended road dust [3].

The reason for such high concentrations of PM10 and chemical elements, both in Wadowice and Skala, can be the unfavourable landform and windless weather, but also anthropogenic sources (e.g., combustion of fossil fuels in households, transport and industrial emission).

The values of element concentration in Wadowice and Skala are as follows:

- Wadowice: Cl>K>Fe>Ca>Zn>Pb>Br>Ti>Cu>Mn>V>As>Co>Rb>Se>Ni>Sr>Cr;
- Skala: K>Fe>Ca>Zn>Pb>Br>Ti>V>Mn>Cr>Cu>Sr>Co>Rb>Ni.

Comparison of the results of chemical element concentration study in Wadowice and Skala with the results obtained in Krakow in 2010 and 2011 leads to the following conclusions: the concentration for K is the highest in Wadowice, but it is similar in Krakow (2010) [9] (see Table 3). Further, the value of K is similar in Skala and in Krakow (in the Nowa Huta borough) [16]; Ca, Cr, and Mn levels were lower in Wadowice and in Skala than in Krakow in 2010 [16]; Ti levels were similar in Wadowice and in Krakow in 2010; Fe was similar in Wadowice and Krowdrza (Krakow) [16]; Cu level is similar in Wadowice and in Krakow (Nowa Huta borough) and in Krakow in 2011 [9, 12]; Zn and As were similar in Wadowice and in Krakow in 2010 [9]; Br concentration was similar in Skala and in Krakow in 2012, and in Nowa Huta and Krakow in 2010 [9, 16, 17]; Sr concentration was similar in Wadowice and in Krakow in 2011 [17]; Pb concentration was similar in Wadowice and in Skala and they were both similar to Krakow (Nowa Huta) and to Krakow in 2010 [9, 16].

For illustrative purposes, the results for cities larger than Wadowice and Skala are presented in Table 3. The values for most concentrations are different for Wadowice, Skala, and roads in London. The differences for Cl, K, Br, Zn, and Pb are particularly interesting between Wadowice, Skala, and London. However, it can be also observed that there is a similarity for Cr, Fe, and Ni.

Conclusions

The aim of this study was to assess and compare the air quality in two small towns in the region of Lesser Poland (Wadowice and Skala).





The EU limit levels of PM10 concentrations were exceeded on most of the sampling days. The main result of this study was based on the concentration of chemical elements. The mean values of chemical concentrations, for example, Cl, K, Ca, Fe, and Br were higher for Wadowice than Skala. On the other hand, Cr and Sr were higher in Skala and a few elements whose concentrations were similar. The combustion of fossil fuels and biomass can be the source of element concentration of Cl, K, Pb, Ca, and V. It can be observed that the presence of metal works and factories could have a significant impact

on poor air condition because of the toxic metals that were released into the environment. Moreover, the pollution caused by road dust and neighbouring towns or villages may have contributed to the deterioration in air quality.

The element concentrations for Wadowice and Skala were compared with the results obtained in Krakow in 2010 and 2011. This analysis has proved that many similarities exist in the results of element concentrations. This similarity in the composition of air pollutants in towns of different sizes is encountered throughout Europe [7]. The results clearly demonstrate that the PM10 concentration is high and, perhaps more surprisingly, that it is not altogether different from the level of pollution which occurred in large cities a few years ago.

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ORCID

- L. Samek  <http://orcid.org/0000-0001-8636-2771>
 Z. Stegowski  <http://orcid.org/0000-0001-7480-5811>
 K. Styszko  <http://orcid.org/0000-0003-0092-3772>
 A. Turek-Fijak  <http://orcid.org/0000-0003-1025-6141>

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