

A comparative study of portland cements CEM I used for water pipe renovation in terms of pollutants leaching from cement coatings and their impact on water quality

Anna Młyńska and Michał Zielina

ABSTRACT

Obtained EDS and ICP elemental analyses results showed some small differences between the compositions of two of the most common Portland cements, CEM I types, used for water pipe renovation. An evident link between the cements' composition and total amounts of leached elements from tested cement coatings was observed. The performed experiments confirmed that the greatest risk of water contamination soon after pipe renovation by cement mortar lining is related to aluminium leaching. As it turns out, the degree of aluminium leaching from cements produced by two different manufacturers is indicated to be very similar, in comparison to the leaching of calcium compounds and some trace elements. In both cases, the pollutants leaching from the cement coatings into water is most intensive in the initial period of contact and decreases over time. Despite some differences between the calculated concentrations of chemical elements in water, especially lead and chromium, in most cases, their estimated concentrations at the outflow from freshly renovated pipelines cemented with different Portland cements CEM I types and for different pipe diameters, lengths and water flow velocities were much lower than the maximum permissible levels in drinking water.

Key words | CEM I, leaching, pollutants, Portland cement, renovation, water quality

Anna Młyńska (corresponding author)

Michał Zielina

Faculty of Environmental Engineering, Department of Water Supply, Sewerage and Environmental Monitoring,

Cracow University of Technology, Warszawska 24 St., 31-155 Cracow, Poland

E-mail: a.mlynska13@gmail.com

INTRODUCTION

The European standard (EN 197-1:2011), which is used worldwide, defines five classes of common cement that comprise Portland clinker as a main constituent (CEM I–CEM V). It is noted that for the water pipe renovation process using the cement mortar lining method, Portland cement CEM I is the most commonly used type of cement. Each type of cement consists of four groups of components: the main group of components (inorganic materials), secondary group of components (inorganic materials, but in smaller quantities), a binding time regulator component (calcium sulphate) and additive components to improve the cement quality (EN 197-1:2011). In the case of Portland cement CEM I, its main component is Portland clinker, constituting 95–100% of the total mass of cement. Portland clinker

consists of many different chemical elements, which are classified into three groups, taking into consideration their amount (Gruener 1983; Bye 1999; EN 197-1:2011). The main group comprises calcium, silicon, aluminium and iron oxides (96–97% of total Portland clinker mass), the second group comprises minor chemical elements, as magnesium, sulphur, sodium and potassium oxides (3–4% of total Portland clinker mass) (Gruener 1983; Bye 1999) and the third group contains trace elements, among which can be found heavy metals, such as lead, cadmium, chromium, arsenic, nickel, arsenic, barium or vanadium (in total, less than 0.1% of total Portland clinker mass). Heavy metals may be introduced into the cement from raw materials or during its production process, mainly during clinker burning

(Gineys *et al.* 2010; Vollpracht & Brameshuber 2016). The source of different trace metals in cements are waste materials from industrial or agricultural applications, that are commonly used in cement kilns as a fuel supplement (Horsley *et al.* 2016). Although the composition of cements is regulated by current standards in engineering practice, the percentage share of particular cement components, especially trace elements, even in the case of the same cement type, can be slightly different, which is related to the individual cement production process in different cement factories.

Hardened cement mortar is the most exposed for leaching elements in contact with low-content ions soft waters (Bonds 2005). In the case of the initial period of contact with cement coatings made of Portland cement, the greatest risk of water contamination is related to calcium leaching (Douglas & Merrill 1991; Ong & Wong 2003; Clark 2009; Deb *et al.* 2010) and aluminium leaching (Clark 2009; Deb *et al.* 2010; Zielina *et al.* 2014, 2015; Młyńska & Zielina 2016, 2017). As it turns out, some heavy metals leaching is also particularly dangerous for human health, thus many studies on this aspect (Rankers & Hohberg 1991; Germaneau *et al.* 1993; Kanare & West 1993; Guo 1997; Guo *et al.* 1998; Hillier *et al.* 1999; Kowalska *et al.* 2011) have also been performed. As was observed, hardened cement mortar in contact with water may release different kinds of toxic metals and its leaching process may vary to some degree. Thus, a different impact of cement coatings made of the same type of cement is expected soon after water pipes' renovation. For this purpose, the influence of the two commonly used for water pipe renovation, Portland cements CEM I, produced by two different manufacturers, on water quality was analysed. The study performed includes an analysis and comparison of the pollutants leaching from cement coatings made of different Portland cements CEM I during water pipe renovation and their expected impact on drinking water quality parameters. Relations between the amount of pollutants leaching from mortar lining and the composition of cements were tested. Two major factors that affect the leaching of hardened cement mortar components are the volume of water per internal cement coating surface area and the length of contact time of water with cement lining (Neville 2001). Thus, the

calculations performed include different cemented water pipe diameters, lengths and water flow velocities.

MATERIALS AND METHODS

Characteristics of the cements

Two samples of the Portland cement CEM I produced by different manufacturers: Cemex (No. 1) and Dyckerhoff (No. 2) were tested. They are the two most commonly commercially available cements and they are the most common Portland cement CEM I used for water pipe renovation in Poland. What is more, both of them were used for the renovation of the water pipeline section in Cracow.

Elemental composition of both the tested Portland cements – CEM I (No. 1) and CEM I (No. 2) – including calcium (Ca) and aluminium (Al) contents, was analysed using electron dispersive spectroscopy (EDS) technique in a scanning electron microscope (SEM) HITACHI S-3400N equipped with EDS spectrometer manufactured by Thermo Scientific. The EDS quality analysis of investigated cement samples without light elements was determined with a vacuum of 25 Pa and electron energy accelerating voltage of 15 kV, using standardless analytical algorithms, where the detected element concentrations are normalized to 100%. If the solid sample contains several percent of the chemical element, its detectability, with a large number of counts, high voltage and vacuum, is about 0.1%.

In turn, trace element contents such as chromium (Cr) and lead (Pb) were measured according to the ISO 11885:2007 standard using inductively coupled plasma optical emission spectrometry (ICP-OES) technique in an ICP emission spectrometer PLASMA 400, manufactured by Perkin Elmer (quantification limit QL = 2 ppm, detection limit DL = 0.2 ppm). Three samples were tested for each of two types of cements, CEM I (No. 1 and No. 2). Cement samples for chromium and lead detection were prepared by grinding to less than 90 µm size fraction, drying at a temperature of 105 °C and homogenized. Samples were prepared by microwave assisted digestion with a mixture of HCl + HNO₃ acids and filtered. Each of the prepared samples was measured 10 times.

Test stands

The experiments were carried out on two pieces of steel water pipes with internal cement coatings under stagnant water conditions. At the beginning, for the needs of the conducted research, both of the water pipes were cemented manually in the laboratory with Portland cement CEM I (Figure 1). However, the first piece of pipe ($L = 13.7$ cm, DN = 110) was cemented with Portland cement CEM I originating from one of the cement manufacturers (No. 1), while the second one ($L = 25.0$ cm, DN = 82) was cemented with Portland cement CEM I produced by the other manufacturer (No. 2).

Besides cement, in the composition of both of the prepared cement coatings was quartz sand and water in proper amounts (cement to sand to water ratio was 1:1:0.35). The final thickness of the CEM I (No. 1) coating was 8.5 mm and the CEM I (No. 2) coating was 6.5 mm. All of the laboratory cementation process conditions and parameters were similar to those that are maintained during renovations in real conditions on a building site. Both pieces of water pipe were installed on the test stands one month after cementation.

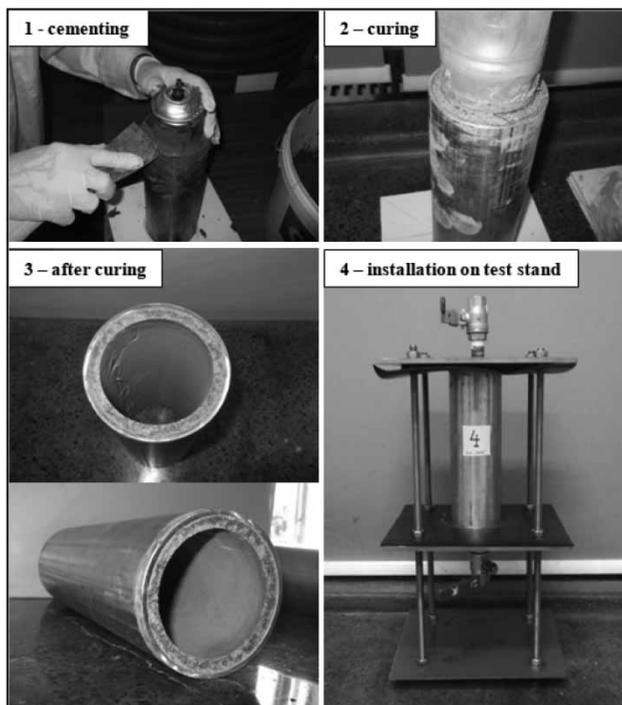


Figure 1 | Laboratory water pipe cementing process for test stand preparation.

Characteristics of water used in experiments

Drinking water used in experiments was collected at the out-flow from the Raba Water Treatment Plant (WTP) located on Dobczyce Reservoir (one of the water treatment plants for Cracow). Water produced by the Raba WTP was characterized by the following approximate values of parameters: pH: 8.18, alkalinity: 2.96 mmol/dm^3 , hardness: $130.0 \text{ mg CaCO}_3/\text{dm}^3$ (soft water) and aluminium concentration: 0.030 mg/dm^3 , calcium concentration: 36.0 mg/dm^3 , chromium concentration: 0.0 mg/dm^3 and lead concentration: 0.0 mg/dm^3 .

Laboratory experiments

Experimental research was performed under laboratory conditions, based on the cement components leaching tests methodology presented in EN 14944-3:2007 and EA NEN 7375:2004 standards. The two pieces of cemented water pipe were filled with the same type of water, which was in contact with cement coatings for the same period of time. One square decimetre of cement coatings surface area covers 0.2 dm^3 of water volume. A water sampling schedule was elaborated with a partial compliance with two standards: EN 14944-3:2007 and EA NEN 7375:2004. The whole amount of water from both water pipes' interiors was simultaneously collected and replaced by new fresh water after about 1, 2, 4, 7, 9, 12, 16, 19, 36 and 64 days. After each sampling and replacement, water was tested in the laboratory for the concentration of certain chemical elements and some physical indices. Water temperature during experiments was kept at about 23°C .

Elaboration method

Elemental analyses of the collected water samples after water contact with the two types of cement coatings included those of calcium, aluminium, chromium, lead and cadmium concentration. Water pH values and alkalinity also were measured. Based on the measured concentration of chemical elements, the cumulative leaching amount of each of them and the leaching elements' intensity over time was calculated.

The obtained results allowed us to perform calculations simulating the expected water quality parameters, including calcium, aluminium, chromium and lead concentration directly after renovation, at the outflow from the freshly cemented water pipelines using different Portland cements CEM I: (No. 1) and (No. 2). An estimation of the water quality parameters delivered to consumers was elaborated only based on the contact time of water with the cement coating, not taking into account hydraulic conditions during water flow in the pipeline. Calculations were performed for different water pipe diameters (DN) (200 mm, 500 mm, 1,000 mm) and different lengths (L) (500 m, 1,000 m, 2,000 m, 5,000 m, 10,000 m). Cement coating thickness was assumed based on the DVGW standards that describe cementing process (DVGW W 343) and was equal to 4 mm (for DN = 200 mm), 6 mm (for DN = 500 mm) and 10 mm (for DN = 1,000 mm). In addition, calculations were performed for two different water flow velocities in distribution systems: $v = 0.5$ m/s and $v = 1.5$ m/s. Flushing time period before returning the pipeline to use after renovation was assumed to be equal to 6 hours (similar as in real conditions on a building site). Chemical elements' concentrations in drinking water inflowing to the cemented pipelines were the same as in drinking water used in experiments (calcium: 36.0 mg/dm³, aluminium: 0.030 mg/dm³, chromium and lead: 0.0 mg/dm³).

RESEARCH RESULTS

Elemental composition of tested Portland cements

CEM I

Results of EDS elemental analyses showed that the chemical composition both of the tested cements relating to the calcium and aluminium content is typical for the Portland cement CEM I (Figure 2). Energy dispersive spectra presented in Figure 2 indicated very small differences between the aluminium weight content in CEM I (No. 1) and in CEM I (No. 2). It was observed that CEM I (No. 1) contains a little more calcium than CEM I (No. 2).

Conducted ICP analysis of trace elements showed quite a low content of chromium in both analysed cements and even lower lead content. Nevertheless, tested Portland

cement CEM I (No. 1) contained a slightly larger amount of chromium than Portland cement CEM I (No. 2) and even more than twice the amount of lead compounds (Figure 3). Standard deviation (SD) for CEM I (No. 1) was equal to: 0.67 ppm (Cr) and 0.31 ppm (Pb) and for CEM I (No. 2) was equal to: 0.78 ppm (Cr) and 0.56 ppm (Pb). Measurement uncertainty (U) for CEM I (No. 1) and for CEM I (No. 2) was equal to: 2.0 ppm (Cr) and 3.0 ppm (Pb).

Leaching pollutants from cement coatings

Total amounts of leached elements from the two tested cement coatings (Figure 4) were strongly related to the typical chemical element composition of Portland cement CEM I (Figures 2 and 3).

The results presented in Figure 4 indicated that leaching of calcium from cement coating made of Portland cement CEM I was more intensive than other chemical elements. During the experiments, leaching of aluminium was also observed. However, aluminium was leached in lower amounts than calcium oxides, but in higher amounts than the two analysed trace elements. The total amount of leached aluminium from the cement coating made of CEM I (No. 1), over the 64-day investigation period, was more than 170 times greater than the total amount of leached chromium. In the case of Portland cement CEM I (No. 2), this amount was nearly 300 times higher. It was observed that the amounts of leached lead were even smaller than the total amounts of leached chromium. Research results presented by Deb *et al.* (2010) indicated similar dependences. After the first day of water contact with fresh cement mortar, calcium concentration was about 275.0 mg/dm³, the aluminium concentration was about 0.20 mg/dm³ and chromium – 0.075 mg/dm³. These metals were basically the only ones detected in the water samples (Deb *et al.* 2010).

Over two and a half times greater amounts of calcium were leached from cement coating made of CEM I (No. 1) than from the second cement coating (No. 2) (Figure 4). In the case of both analysed cement coatings, the total amounts of leached aluminium were very similar, while the amounts of leached chromium and lead over the 64-day experiment were more varied. Larger amounts of trace elements were leaching from the cement coating made of CEM I (No. 1)

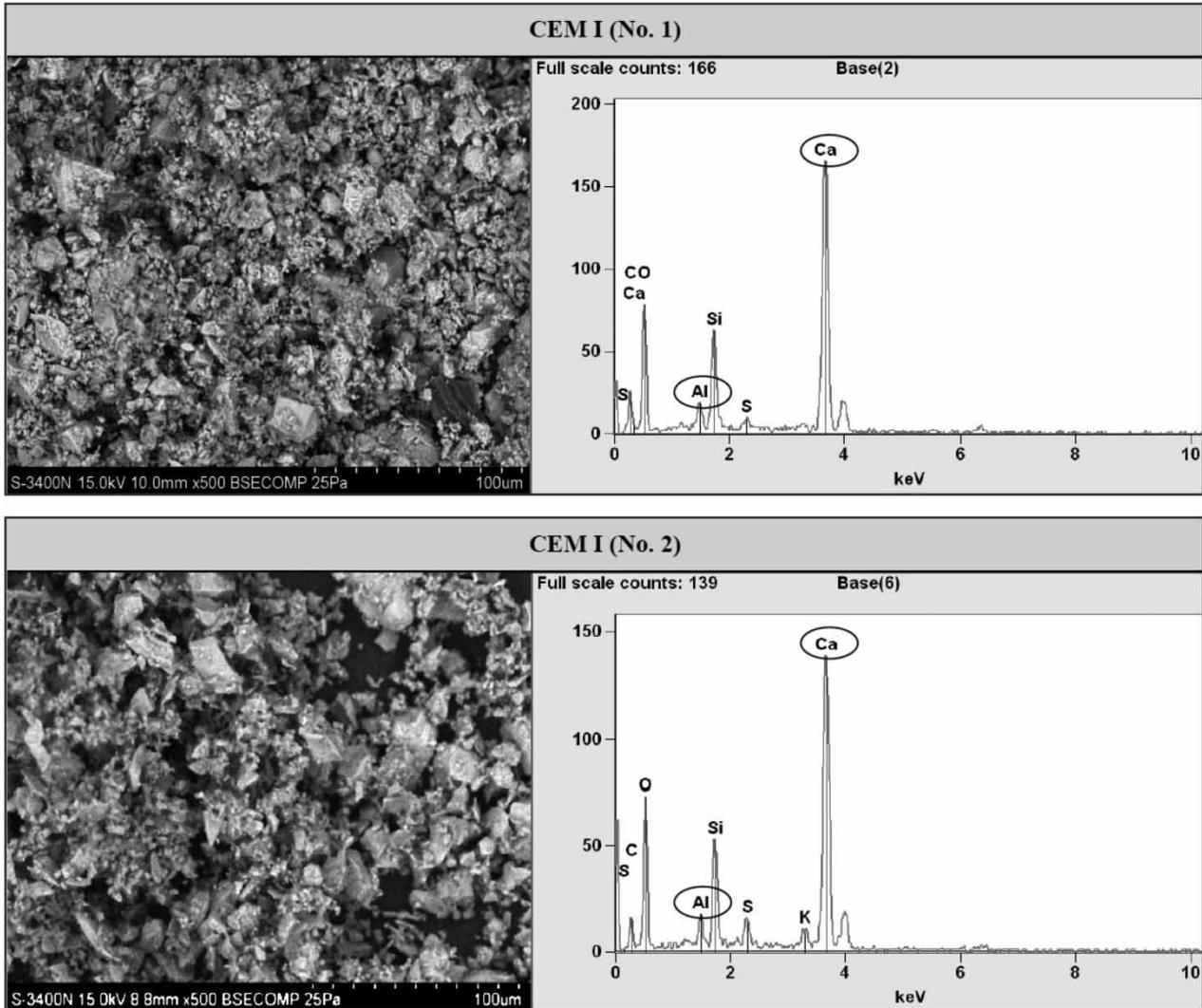


Figure 2 | The chemical composition and microstructure of the tested Portland cements: CEM I (No. 1) and CEM I (No. 2).

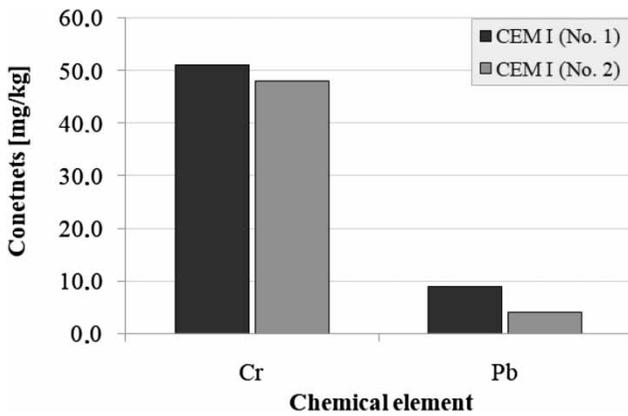


Figure 3 | Chromium and lead contents in tested Portland cements: CEM I (No. 1) and CEM I (No. 2).

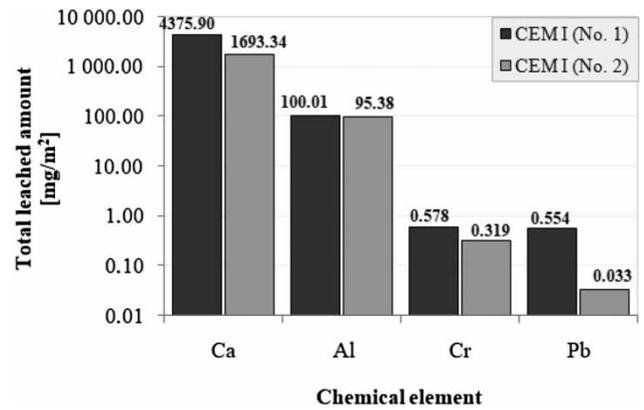


Figure 4 | Total amounts of leached chemical elements from the two tested cement coatings over the 64-day investigation period.

than from the second one – CEM I (No. 2) (Figure 4). This may indicate that the individual cement production process may have an impact on the content of some chemical elements in the cement. This is mainly related to the content of trace elements.

During the experiments, cadmium concentration in water after contact with the two cement coatings also was controlled. Nevertheless, noted cadmium concentration in all of the collected water samples was under instrument detection limit (0.005 mg/dm^3), regulated by the current standards (Council Directive 1998). Therefore, further calculations for cadmium were not performed.

The performed experiments have shown that the greatest intensity of pollutants leaching from cement coating is expected in the initial period of contact time of water with fresh cement mortar. It was observed that leaching process intensity decreased over time (Figure 5).

During the conducted experiments, calcium compounds were leached from both the analysed Portland cements only for the first 4 days of the experiment. Nevertheless, in the first 24 hours of the study, leaching calcium intensity from the cement coating made of CEM I (No. 1) was over five times greater than from the second coating (No. 2) and about two times greater within the next days (Figure 5(a)).

Although in the case of aluminium, the leaching process was most intensive in the first few days of the experiment, the leaching process was further observed, even until the end of the investigation period, especially in the case of cement coating made of CEM I (No. 2). The intensity of aluminium leaching from both analysed coatings was very similar only over the first 4 days. Over the following days, the leaching intensity was more varied (Figure 5(b)).

It was noted that chromium was leaching from the cement coating made of Portland cement CEM I (No. 1)

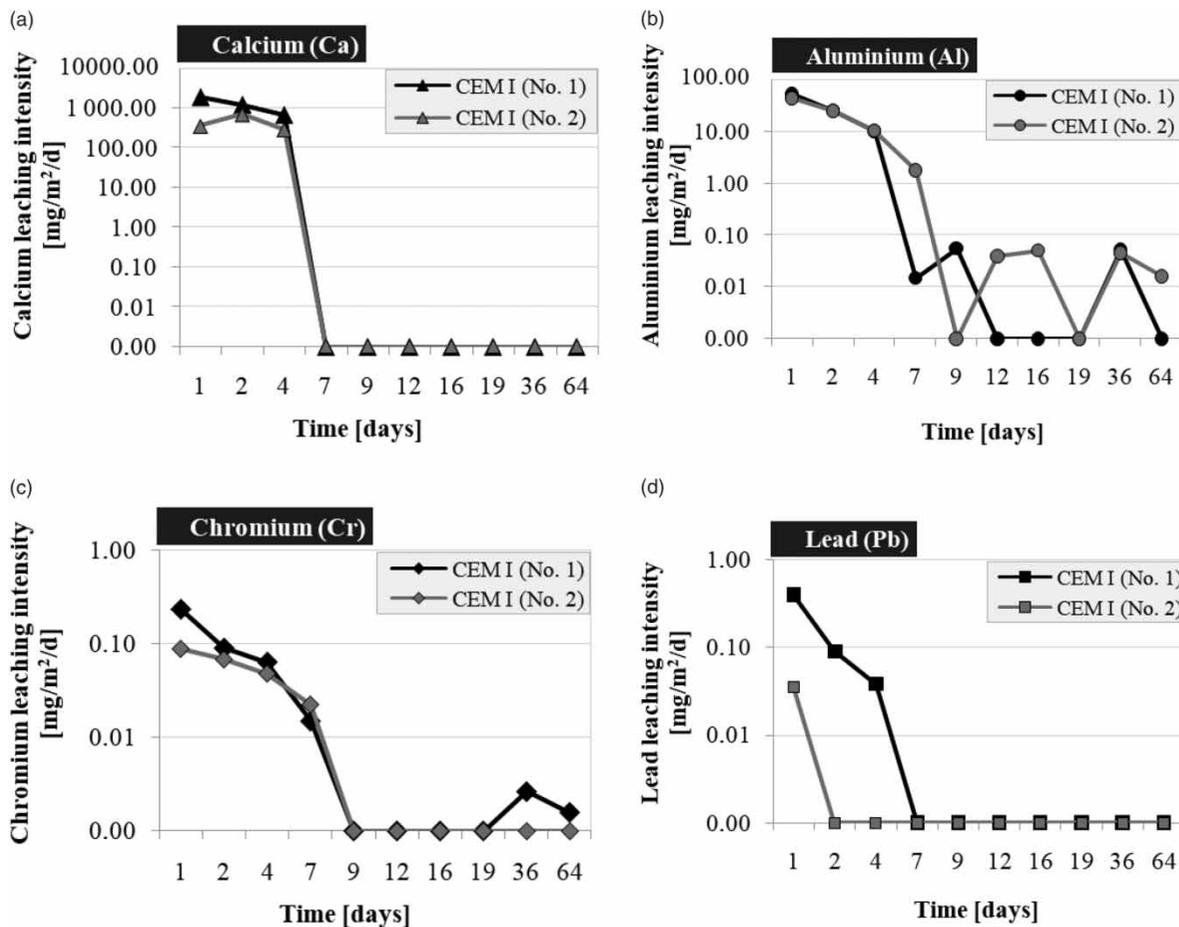


Figure 5 | Leaching intensity of chemical elements from the two tested cement coatings over the 64-day investigation period.

and CEM I (No. 2) in the first week of the study. On the first day, the intensity of the chromium leaching process was greater in the case of CEM I (No. 1) than in the case of the second coating. Nevertheless, the intensity of chromium leaching was more similar over time. At the end of the investigation period, water caused chromium leaching again only from the CEM I (No. 1), although in trace amounts (Figure 5(c)).

Differences between the amounts of leached lead from the two analysed cement coatings and the intensity of this process were even more evident than in the case of chromium. From the cement coating made of CEM I (No. 2), lead was leached in trace amounts only during the first 24 hours of water contact with the cement coating. In contrast, the lead leaching process from the cement coating made of CEM I (No. 1) was observed for 4 days of the experiment and was much more intensive (Figure 5(d)).

Experimental results of some authors (shown in Figure 6) confirm the above and show that the greatest growth of chemical elements' concentration in water is

expected in the initial period of contact of fresh hardened cement mortar with water. Then, in a short period of cement with water contact, the most rapid growth of elements' concentration in water is observed. After the next water replacements, the leaching process is weaker.

During the experiments, very rapid growth of the water pH values was observed both in the case of cement coating made of CEM I (No. 1) and in the case of the second coating made of CEM I (No. 2) (Figure 7(a)). During the first day of the experiments, water pH significantly exceeded 12. In the next days, growth of water pH after contact with cement coatings decreased gradually. Nevertheless, for most of the investigation period, pH values were recorded as close to 12. In general, the changes of water pH values were similar in contact with both cement coatings. Similarly to pH, rapid growth of alkalinity was observed in the first 24 hours of contact water with tested cement coatings (Figure 7(b)). At the time, the differences between the water alkalinity after contact with both cement coatings were visibly significant. In the next days, the growth of water alkalinity gradually

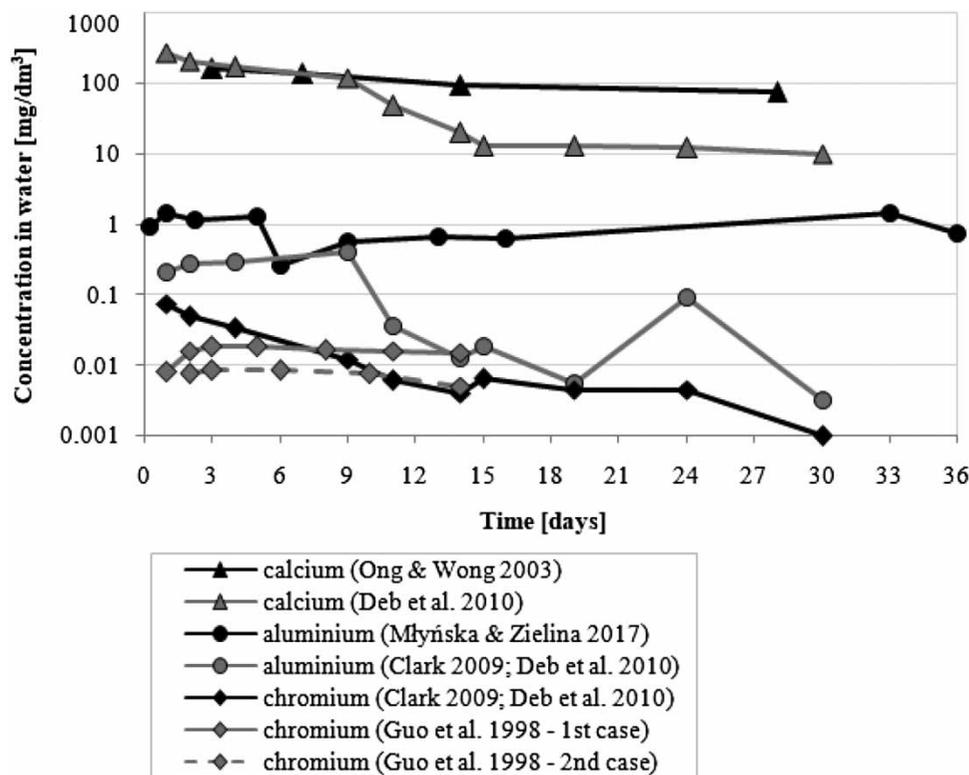


Figure 6 | The changes of chemical elements' concentration in water after contact with hardened cement mortar presented by authors in different studies.

decreased and increased again after a long time, especially in the case of the cement coating made of CEM I (No. 2) (Figure 7(b)).

Expected water quality parameters after renovation of cement mortar pipe lining (based on calculations)

As is shown in the graphs (Figures 8–11), chemical elements' concentration in the flowing water increases proportionally to the cemented pipe length and reversely proportional to pipe diameter. It was also observed that the smaller the water flow velocity, the greater growth of elements' concentration in water as a result of longer contact time with water (Figures 8–11).

Despite the fact that using Portland cement CEM I in water pipe renovation with the cement mortar lining technique is related to the leaching of cement compounds, it turns out that the risk of water contamination caused by the leaching of pollutants is expected to be at a very low level. In the most cases of aluminium, chromium and lead, the calculated concentration of each of them in water at the outflow from the freshly cemented pipelines was below the maximum permissible level in drinking water, as regulated by current standards (Council Directive 1998) (Figures 9–11).

Calcium concentration in drinking water is not limited. More intensive calcium leaching from the CEM I (No. 1) than from the CEM I (No. 2) initially (Figure 5(a)), may

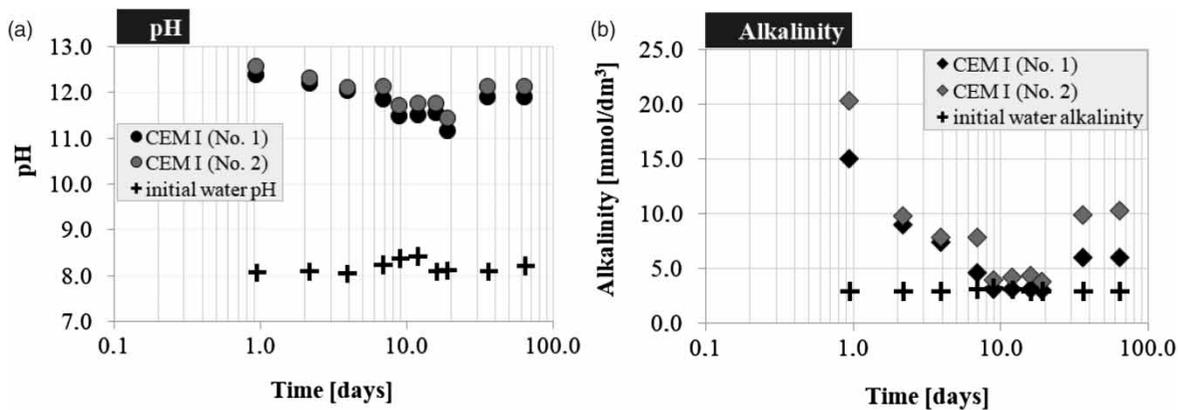


Figure 7 | The changes of water pH values and alkalinity after contact with the two tested cement coatings over the 64-day investigation period.

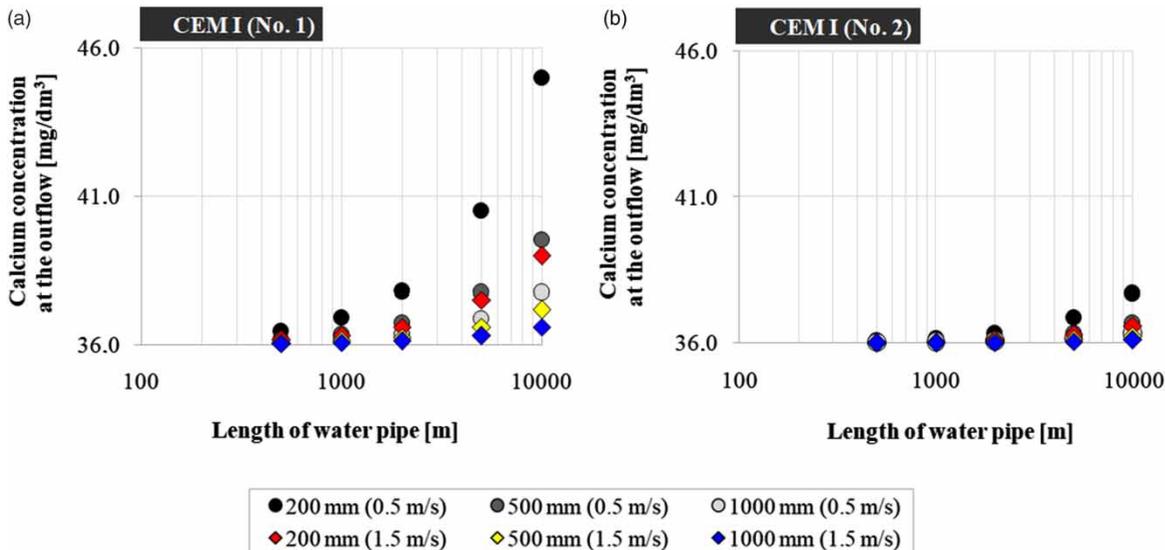


Figure 8 | Calculated calcium concentration in water at the outflow from freshly cemented pipelines with different diameters and lengths and for two different water flow velocities.

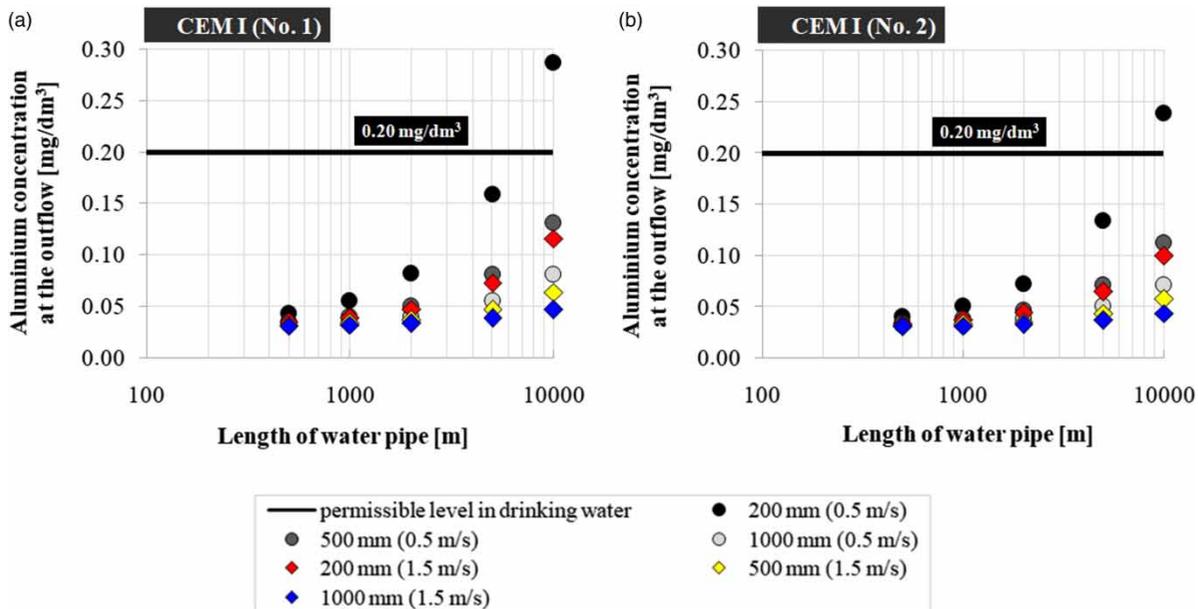


Figure 9 | Calculated aluminium concentration in water at the outflow from freshly cemented pipelines with different diameters and lengths and for two different water flow velocities.

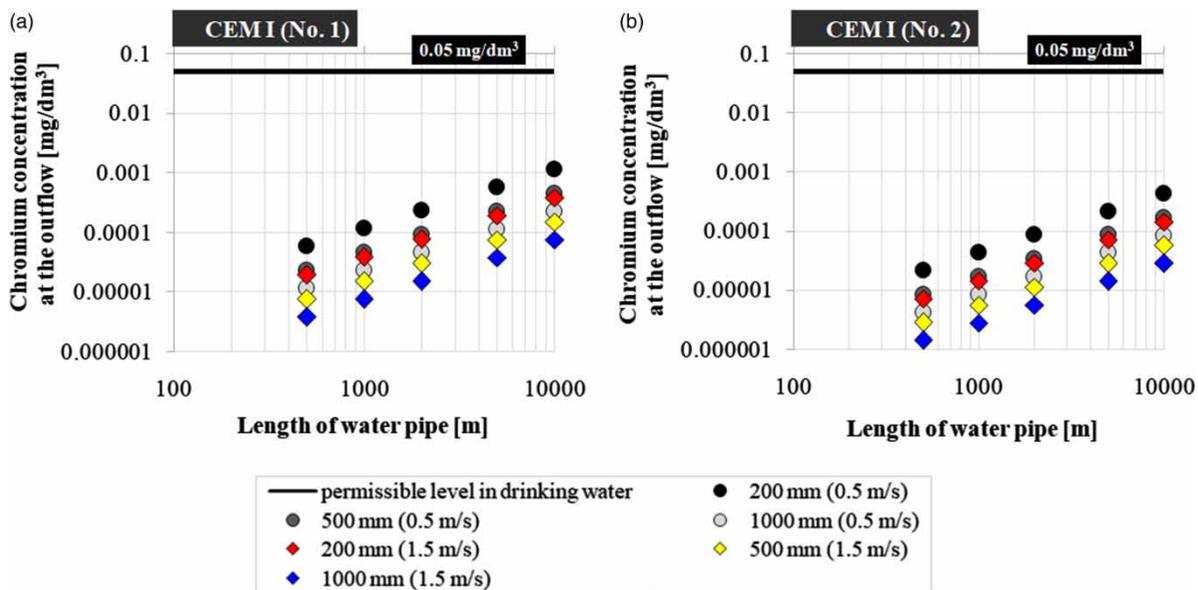


Figure 10 | Calculated chromium concentration in water at the outflow from freshly cemented pipelines with different diameters and lengths and for two different water flow velocities.

contribute to the greater growth of calcium concentration in the short term after renovation (Figure 8). Nevertheless, in both cases, in comparison to the typical calcium concentration in soft water produced by WTP (in performed calculations 36.0 mg/dm^3), the growth of calcium concentration due to contact of water with fresh cement mortar is very small.

Based on the calculated aluminium concentration at the end of the freshly renovated pipelines, the greater risk of water contamination is related to cement CEM I (No. 1) than cement CEM I (No. 2), but the impact of both cements on aluminium content is not much different. In both cases, exceeding the maximum value of aluminium concentration in drinking water (0.20 mg/dm^3 according to the Council

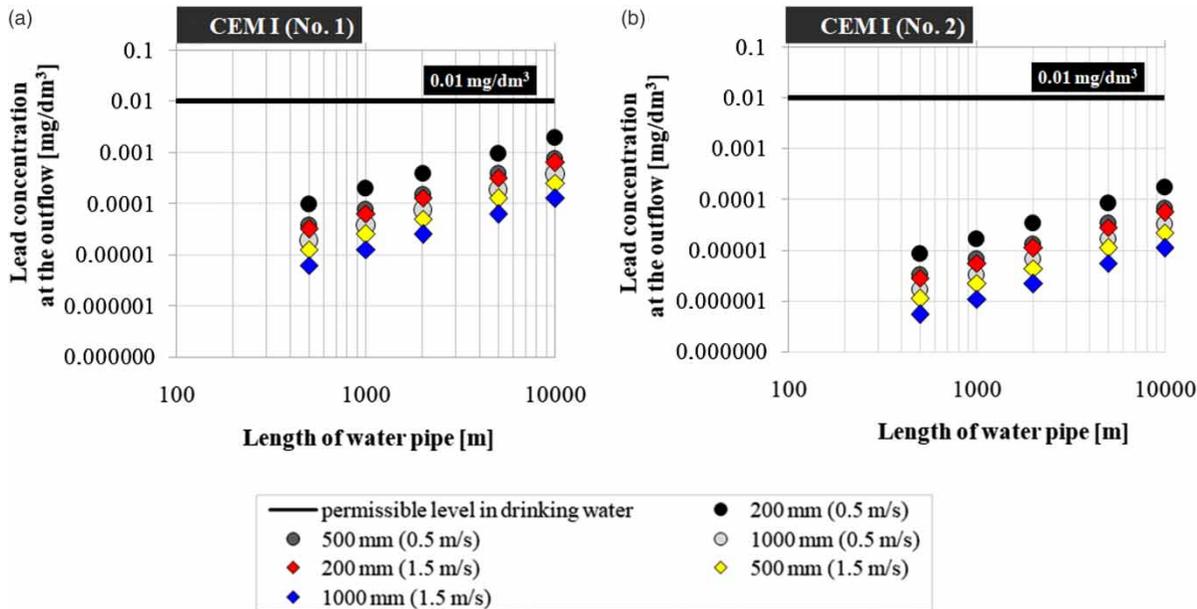


Figure 11 | Calculated lead concentration in water at the outflow from freshly cemented pipelines with different diameters and lengths and for two different water flow velocities.

Directive 1998) is expected only for the very long once-cemented pipe section (10,000 m) with relatively small diameter (200 mm) and for relatively low water flow velocity (0.5 m/s) (Figure 9). Nevertheless, usually such long pipelines in water distribution systems are characterized by much greater diameters than 200 mm. What is more, while calculations were performed, it was noticed that the increase of aluminium concentration in water as a result of its contact with cement coating is quite small. Thus, the water quality delivered to consumers in terms of aluminium concentration is mainly dependent on the quality of water inflowing to the cemented pipe section, expressed by the concentration of aluminium in water produced by the WTP.

It was estimated that after water pipe renovation with cement coatings made of Portland cement CEM I, the chromium concentration in water even at the end of the extremely long cemented water pipe section (10,000 m) with extremely small diameter (200 mm) and for water flow velocity of 0.5 m/s is about 44 times smaller (No. 1) or 117 times smaller (No. 2) than its permissible level in drinking water (0.05 mg/dm^3) as Council Directive (1998) determines (Figure 10). The differences between these two cements were also noticed in the case of lead analysis (Figure 11). Independently from water flow velocity, the lead concentration at the end of the pipelines cemented

with CEM I (No. 1) was over 10 times greater than in the case of the pipeline with an internal coating made of CEM I (No. 2). In the most unfavourable case ($L = 10,000 \text{ m}$, $DN = 200 \text{ mm}$, $v = 0.5 \text{ m/s}$), lead concentration is expected to be over 5 times smaller or about 60 times smaller than its limitation value in drinking water (0.01 mg/dm^3) for CEM I (No. 1) and CEM I (No. 2) coating, respectively (Figure 11). Thus, it was proven that the composition of Portland cement CEM I produced by different manufacturers may have a different degree of impact on water quality, especially in terms of trace element leaching.

CONCLUSIONS

Portland cement CEM I, as well as other cement types, contains many chemical elements. Taking into consideration the fact that this cement is the most commonly used for water pipe renovation with the cement coating method, the control of drinking water quality soon after renovation, in terms of chemical element concentration, seems to be very important. Although the composition of cements is regulated by current standards, it is supposed that the percentage share of some chemical elements, especially trace elements, in the same type of cement but produced by different manufacturers,

may vary to some degree. Thus, their impact on water quality may have a greater or lesser significance.

The conducted elemental analyses showed some small differences between the compositions of the two commonly used for water pipe renovation, Portland cements CEM I produced by two different manufacturers. Both cements, defined in the paper as CEM I (No. 1) and CEM I (No. 2), consist of very similar amounts of aluminium. However, CEM I (No. 1) contains a little more calcium and chromium than CEM I (No. 2) and more than twice the amount of lead. A clear dependence was observed during the experiments between the chemical composition of the cements and the leached elements to the water from the coatings. However, although the observed dependence was not linear, it is clearly seen that much more intensive leaching of lead was noticed from CEM I (No. 1) than CEM I (No. 2), which strongly correlates with the chemical compositions of both cements.

The performed analysis showed and confirmed that the most significant effect on water quality having direct contact with fresh cement coating is related to aluminium leaching. In the case of both analysed cement coatings, the amounts of leached aluminium similarly decreased within the time, but this was still observed even over the 64-day investigation period. Calcium was leached only up to 4 days, and after this time the leaching disappeared. Nevertheless, it was observed that the final concentration of calcium compounds in the water delivered to the consumers is mainly dependent on the quality of the water produced by the WTP. Leaching of chromium was relatively similar for both samples and disappeared after 7 days, but some secondary leaching was observed in the case of CEM I (No. 1) after a long period of time. In the case of lead leaching, the differences between the two cement types were more evident. Soon after the first day, the lead leaching from the coating made of cement CEM I (No. 2) disappeared, whereas the lead leaching from the coating made of CEM I (No. 1) was still continuing up to the fourth day.

The risk of hazardous water contamination caused by pollutants leaching from a fresh cement coating soon after renovation is very small. It was observed that the smaller the diameter and the longer once-cemented water pipe section, as well as the lower water flow velocity, the greater growth of chemical elements concentration in flowing

water. In the case of the water pipeline cemented with CEM I (No. 1), the calcium, aluminium, chromium and lead concentrations are expected to be greater than in the case of CEM I (No. 2). In both cases, only the concentration of aluminium leached from the coatings may exceed the permissible value for very long and small water pipe cross-sections and for relatively low water flow velocities. Nevertheless, it is supposed, that even in the case of high aluminium concentration in water produced by WTP, temporary exceeding of the limitation of aluminium concentration in drinking water should not affect the human body. In turn, the highest expected chromium concentration at the end is over 40 times and about 120 times smaller than the permissible value in drinking water for CEM I (No. 1) and CEM I (No. 2), respectively, whereas the expected lead concentration is over 10 times smaller in the case of using Portland cement CEM I (No. 2) than cement (No. 1) for renovation.

Strong increases of pH and alkalinity, observed within the experiments, affect the human body mainly aesthetically, i.e., related to taste and odour. However, alkaline water can form scaly deposits and reduce efficiency of disinfectant, affecting the human body indirectly. Nevertheless, it was noted that the water quality parameters, especially in terms of trace element concentration, may depend on the cement type used.

To summarize the above, it may be stated that the individual Portland cement CEM I production process may have an impact on its composition. This is mainly related to the content of trace elements, among which are heavy metals. Thus, the water quality parameters after contact with cement coatings made of different Portland cement CEM I may differ. Therefore, the choice of some of the most common commercially available Portland cements CEM I for water pipeline renovation may be associated with a varied impact on drinking water quality.

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