

APPLICATION OF COMPOSITE CONSTRUCTION AND DEMOLITION DEBRIS IN HEAVY METALS REMOVAL FROM INDUSTRIAL WASTEWATER

by

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The utilization of construction and demolition debris in industrial wastewater treatment by sorption of Co^{2+} and Ni^{2+} ions was investigated. Selected waste composites are cost-effective and locally available, still their sorption characteristics and application are not sufficiently investigated. The samples of concrete, facade, ceramic materials, and asphalt were characterized in terms of mineral and surface composition, radioactivity, and stability at different pH values, as well as pH values of suspension and filtrate and electrical conductivity of the filtrate. The sorption capacities were determined in batch experimental conditions in one- and multi-component solutions. Characterization showed different crystal structures and mineralogical compositions of components. The results of gamma spectrometry confirmed the radiological safety of samples. Based on stability testing results, waste materials are suitable for further utilization and do not pose any risk to the environment. The overall sorption results suggested that cement-based materials, in addition to high affinity for the tested ions, represent a sorbent that binds contaminants firmly enough, reduces their mobility and bioavailability, and are suitable for removing Co^{2+} and Ni^{2+} ions from wastewaters.

Key words: heavy metals, water treatment, debris, sorption

Introduction and motivation

Sources of industrial wastewater are numerous: agriculture, food and drink processing, iron and steel industry, metal finishers, chemical manufacturing, mines and quarries, industrial laundries, oil fractionation and gas processing, solid hazardous waste disposal, energy production, etc. Industrial wastewaters contain various organic and inorganic pollutants. Heavy metals are among these pollutants. They might be toxic and/or carcinogenic, therefore, harmful to humans and other living species. These ions are not as biodegradable as most organic substances. Some are essential for physiological processes in low concentrations but are considered extremely toxic in higher concentrations (Fe, Zn, Mn, Cr, Cu, and Mo) [1]. However, numerous heavy metals are solely toxic and pose a substantial risk to humans and the environment (Cd, Hg, Pb, Co, and Ni). The most common released heavy metals, that are

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dangerous environmental pollutants, are copper, cobalt, chromium, cadmium, nickel, lead, manganese, and zinc [1].

The high costs of heavy metals removal from industrial wastewater, *e.g.* by sorption on activated carbon [2], stimulate research into the development of cost-effective sorbents, especially those that represent the final waste in production or after use.

Significant quantities of construction and demolition debris (C&DD) are generated annually and are predominantly disposed of in municipal landfills, potentially polluting the environment [3]. The C&DD consists of diverse materials such as concrete, ceramic, *i.e.* clay products (various types of bricks and blocks), asphalt, wood, steel, and other metal products, various plastic types, *etc.* These products include organic and inorganic substances that might be dangerous to the environment and human health if they get into contact with soil, water-courses, or the atmosphere. The EU produces about 900 million tons of C&DD annually, and 50% of generated C&DD is recycled. This might indicate that the construction sector is satisfactorily circular, but the waste management practices surveys reveal that C&DD reuse is mainly based on backfilling operations and low-grade recovery, *e.g.* utilization of recycled aggregates in base and sub-base of road construction. Globally, there is a substantial tendency to reuse recycled construction materials exclusively in the same branch of industry instead of expanding the application to other sectors [4]. Hence, the development of efficient and cost-effective methods for reduction of growing amounts of C&DD and examining the possibilities of C&DD utilization in areas and processes outside the construction industry would be advantageous. The treatment of industrial wastewater by sorption, is therefore seen as a promising and important option.

Industrial wastewater treatment

Industrial wastewater treatment includes processes used to treat water contaminated by industrial or commercial actions before discharging into the environment or further reusing. Wastewater treatment involves processes that include the consumption of large amounts of energy, excessive sludge production, high operating costs, and usually the creation of unpleasant odors. Heavy metal wastewater treatment today is one of the major problems in the environmental protection field. Conventional technologies for removing heavy metal ions from solution include chemical precipitation, ion exchange, reverse osmosis, electrochemical treatment, and sorption processes. Various studies have shown that the sorption process involves one or more mechanisms of interaction between the solid surface and the species present in the solution, based on chemical and physical processes or a combination thereof [5]. These phenomena imply many different mechanisms: surface adsorption caused by attractive molecular forces, ion exchange, ion-pair formation, precipitation, specific sorption (usually a combination of adsorption and precipitation), creation of hydrogen bonds or complex compounds, or their combination. The mechanisms of sorption mostly depend on the physico-chemical properties of the material (sorbent) and ion (sorbate) nature. Depending on the type of sorbent-sorbate interaction, sorption is based on physical or chemical interactions. Physical sorption is characterized by a smaller amount of heat exchange and low activation energy, thus at lower temperatures. On the other hand, sorption can also be caused by chemical factors, such as changes in redox potential, pH of the solution, or concentration of substances. This further affects the process equilibrium in suspension or solution and the formation of chemical bonds between ions in solution with active groups on the surface of the solid phase. Sorption caused by chemical factors has significantly higher activation energy [5]. If the sorbent and ion from the solution came into contact, the ion binds to the sorbent by some

aforementioned mechanism. However, at the same time, the opposite process occurs, *i.e.* the process of desorption. When the speeds of these two processes are equal, an equilibrium state is established. After establishing equilibrium, it is possible to determine the sorption capacity of a given sorbent under specified experimental conditions [6]. The results of sorption experiments are usually represented by sorption isotherms that define the dependence of the sorbed amount in the solid phase Q_e [$\text{mmol}_{\text{ion}}/\text{g}_{\text{sorbent}}$] on the concentration of applied cation C_e [$\text{mmol}_{\text{ion}}/\text{L}_{\text{solution}}$] in the liquid phase after reaching equilibrium at a constant temperature [6].

During the last decade, there has been a significant increase in interest in utilizing waste materials and by-products as sorbents for purifying contaminated wastewaters. The reduction of raw materials and the increasing cost of synthesizing new materials contribute to further research for more economically appropriate, locally available materials, especially materials derived from renewable resources, consuming less natural resources and energy for production or those that represent the final waste after a production process or its useful life [3].

Previous studies with construction industry by-products, the analogs to C&DD components, *e.g.* crushed concrete fines and brick dust, have indicated significant sorption potential for the separation of pollutants from wastewaters. Brick dust, a by-product of the production of ceramic blocks, is proven to be successful for Pb^{2+} , Cd^{2+} , and Cs^+ sorption, as well as for anions As(III) , As(V) , Cr(VI) , and U(VI) [7]. Additionally, the sorption properties comparison of raw clay and broken brick powders revealed that bricks were more effective in Pb^{2+} , Cd^{2+} , and Zn^{2+} removal [8]. Another research [9] found that crushed concrete fines from recycled aggregate production were equally effective in Cu^{2+} , Zn^{2+} , and Pb^{2+} ions immobilization as a range of low-cost by-products. The main objective of this study was to evaluate C&DD components as sorbent materials for the heavy metals removal from industrial wastewater. According to limited data on the utilization of C&DD in sorption processes, it has been concluded that the research should focus on the efficiency of different components of C&DD. Namely, the composition of such materials cannot remain unchanged with the corresponding new materials or by-products due to exploitation in different atmospheric conditions.

Materials and methods overview

Nine composite materials, *i.e.* components of C&DD, were collected at demolished buildings at different locations in the city of Belgrade, Republic of Serbia. The C&DD components used in the experiments were two types of concretes (C1 and C2), and bricks (B1 and B2), hollow brick (B3), facade coating (F), a mixture of crushed roof (RT), ceramic tiles (CT), and asphalt (A). One concrete sample (C1) originates from the ruins of the building, as well as the sample of facade material, while the other concrete sample (C2) comes from the concrete pathway, assuming a different composition and exploitation conditions than the sample of wall concrete (C1) due to their different purposes. Bricks originate from the ruins of buildings built around the 1930s (B1) and the 1970s (B2), while sample B3 is a hollow brick (block) of a more recent period. Bricks from different construction periods were deliberately collected to examine the potential difference in behavior during the experiments due to different ways of brick production over a very long period of production of this traditional building material. The RT and CT samples were selected from five locations as an already partially crushed non-homogenized mixture. Asphalt sample A originates from the paved pathway. The C&DD samples were homogenized and sieved to a fraction with particle size 0.3-0.6 mm [10-12].

The investigations included a detailed waste characterization in order to verify the C&DD sample's effective and safe usage. Therefore, samples were characterized in terms of mineral composition by XRD analysis, surface functional groups by FT-IR spectroscopy, ra-

radioactivity by spectrometry of gamma emitters, stability at different pH values (at 2, 6, and 8), as well as the measurement of pH values of suspension and filtrate, and electrical conductivity (EC) of the filtrate according to modified US EPA 9045D method. The sorption capacities were determined in batch experimental conditions in one- and multi-component solutions, together with mixtures used for simulation of C&DD [10-12]. In sorption experiments, Co^{2+} and Ni^{2+} ions were used, as the particular representatives of heavy metals.

Experimental observations

Sample characterization

The XRD analysis of the samples showed different crystal structures and mineralogical compositions. The XRD spectra of concrete samples C1 and C2 showed that these samples are primarily composed of quartz, while sample F, in addition to quartz, contains muscovite and calcite [10, 11]. Although freshly prepared concrete samples contain by-products of Portland cement hydration processes, such as calcium silicate hydrate (C-S-H) and other cementitious compounds, calcium aluminate hydrate and calcium aluminosilicate hydrate, ageing of concrete under atmospheric conditions causes decalcification and polymerization of silicates [13]. Accordingly, the absence of common peaks for these materials in the tested samples was expected. In addition, the presence of calcium carbonate in cement-based materials can be attributed to the partial carbonation of portlandite, *i.e.* the reaction of CO_2 from the air with calcium hydroxide in concrete [14]. Sample B1 consists mainly of anorthite, calcium silicide, wollastonite, mullite, and elemental iron. Sample B2 contains quartz, sanidine, and anortite. Unlike the previous two samples, B3 contains quartz as the only crystalline phase [10]. The XRD analysis shows that the RT tile sample contains quartz and albite, while quartz and calcite were detected in the CT ceramic tile sample [12]. In addition, peaks originating from silver chromium tin selenide sulfide were observed in the spectrogram of the CT sample. The presence of these phases can be explained by the presence of silver and tin in the colors used, and the use of additives, the so-called chromium and tin-based opacifiers (milky white visual effect) [15]. The main crystal structures in asphalt sample A are calcite, dolomite, and quartz. These minerals are primarily derived from aggregates (sand and gravel).

The FT-IR spectrograms of all samples indicated mostly the presence of Si-O surface groups. Peaks characteristic of carbonate groups [10-12, 16] were detected in the spectra of both concrete, facade, ceramic tiles, and asphalt. In general, the surface groups identified by FT-IR analysis correlated well with the mineral composition of the samples detected by XRD analysis.

Gamma spectrometry demonstrated that the samples were safe for further use from a radiological aspect [10-12].

Equilibration of C&DD samples with solutions with initial pH values of 2, 6, and 8 revealed some similarities in the behavior of the tested materials. The final pH values represent a direct consequence of the mineral composition of C&DD and the period of exploitation of the investigated materials. The increase in pH of the solution during contact with C&DD samples for each initial pH value can be attributed to the dissolution of some of its components [10-12].

Determination of the pH value of C&DD is necessary to assess its corrosive properties and potential negative impact on the environment. Since the reference pH value of the waste class without assigned hazard category (H15), according to Annex 7 of the Regulation on categories, testing, and classification of waste [17], is in the range of 6 and 13, the meas-

ured pH value was within the allowed limits. According to the modified US EPA 9045D method, waste is corrosive if the pH of the suspension is less than two or higher than 12 [18]. Except in the case of suspended sample C1 in a solid/liquid ratio of 1:10 (or greater), disposal and continued use of the tested materials cannot pose a risk to the environment. The EC values were below the maximum allowed values for surface water quality, class I by the Serbian legislature [19]. Based on the results of stability testing and determination of pH and EC values of filtrates, it was concluded that the tested waste materials are suitable for use and do not pose a risk to the environment.

Sorption capacities onto C&DD components

Sorption isotherms of Co^{2+} and Ni^{2+} ions onto C&DD samples are shown in figs. 1 and 2 [10-12].

The highest binding efficiency of Co^{2+} was achieved mainly using cementitious samples C1 and C2, and slightly by sample F [10, 11]. The maximum sorption capacity experimentally determined for the F sample was 0.12 mmol/g, while for samples C1 and C2, it amounted even 0.32 and 0.27 mmol/g, respectively [10, 11]. The RT sample showed a lower sorption capacity of 0.065 mmol/g, while the sorption capacity on the CT sample was more than twice as higher and amounted to 0.17 mmol/g [12]. Samples B1, B2, B3, and A showed the lowest binding tendency to Co^{2+} ions, with maximum sorption capacities of 0.03 mmol/g for sample B3, 0.05 mmol/g for sample B1, and 0.06 mmol/g for samples B2 and A [10], fig. 1.

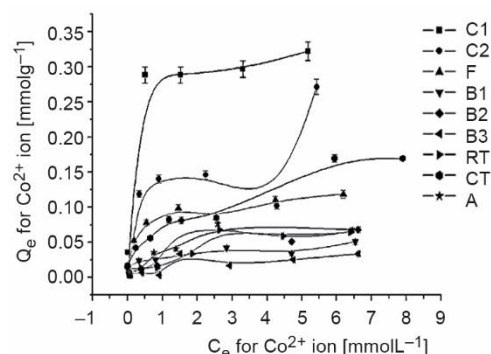


Figure 1. Sorption isotherms of Co^{2+} ion onto C&DD samples

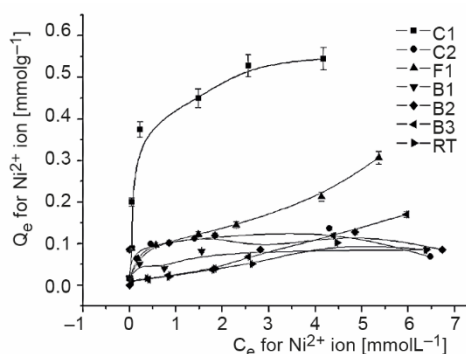


Figure 2. Sorption isotherms of Ni^{2+} ion onto C&DD samples

The most efficient sorption of Ni^{2+} ions was onto sample C1 with the sorption capacity of 0.55 mmol/g, as well as on sample F where the sorption capacity of 0.30 mmol/g was reached, fig. 2. The obtained results are in accordance with the high sorption capacities of C-S-H materials according to Co^{2+} and Ni^{2+} ions [10, 11]. However, sample C2 showed significantly less sorption with a maximum sorbed amount of 0.13 mmol/g [10]. The lowest maximum sorbed amount was observed for samples of bricks, roof and ceramic tiles, and asphalt (B1, B2, RT, CT, and A), with sorption capacities below 0.13 mmol/g [11]. Unlike the Co^{2+} ion, the sorption of Ni^{2+} onto sample B3 proved to be more efficient compared to other brick samples and reached a maximum capacity of about 0.17 mmol/g [10].

The obtained results match with the related studies, which showed that different SiO_2 samples have a variable affinity for Co^{2+} and Ni^{2+} ions. In a study of the sorption of Co^{2+}

and Ni^{2+} ions onto quartz, it was determined that the sorption capacity of both ions was approximately 0.10 mmol/g [20]. Low sorption capacities of $1.68 \cdot 10^{-3}$ mmol/g were determined in the case of hydrated silicates [21], as well as in the case of fly ash (0.1 mmol/g), whose composition dominates silicon dioxide [22].

According to Co^{2+} and Ni^{2+} speciation diagrams against solution pH [23], the formation of insoluble $\text{Ni}(\text{OH})_2$ and $\text{Co}(\text{OH})_2$ starts at pH 8. Therefore, based on obtained final pH values of Co^{2+} and Ni^{2+} ions sorption onto C1, C2, B1, and CT, as well as sample A in the case of Co^{2+} ions, which were close to precipitation threshold, the precipitation process is a possible mechanism of these ions removal [10-12].

Sorption from multi-component solutions with different sorbate ratios was relatively insufficiently investigated. Literature data show that the coexisting cation can cause both antagonistic and synergistic effects in the entire sorption process, depending on the composition of the solution, as well as the type of sorbent used [24]. It was found that the only significant sorbed amount of Co^{2+} ions from the equimolar two-component Co-Ni solution was achieved in the C1 sample with a sorption capacity of 0.18 mmol/g. The Ni^{2+} ion behaved very similarly to the Co^{2+} ion: in an equimolar two-component Co-Ni solution, the Ni^{2+} ion showed satisfactory sorption on sample C1 with a capacity of nearly 0.18 mmol/g.

Cementitious materials showed the highest sorption capacities and the strongest interaction with investigated cations, confirmed by sorption experiments on individual components. Based on the comparison with other waste sorbents, the obtained sorption capacities for the components of C&DD showed a quite satisfying affinity of these materials to the corresponding metal ions [10].

However, it must be taken into account that sorption capacity is not only strongly associated with material properties but also with numerous process parameters (solid/liquid ratio, pH, sorbent particle size, *etc.*) [25], hence the literature values are not straightforwardly comparable. Particles with smaller sizes have more developed specific surface areas and, thus, higher sorption capacity. In particular, it should be noted that in this study, sorbent materials with minimal mechanical treatment in terms of particle homogenization, *i.e.* relatively large granulation 0.3-0.6 mm, were used. Therefore, it can be expected that the specific surface area and, consequently, the sorption efficiency will increase as the particles of the given sorbents decrease. It is also important to emphasize that no additional pre-treatments, thermal or chemical, have been carried out on tested sorbents, which could also increase the sorption capacities of the tested C&DD [10-12, 26].

Sorption capacities onto simulated C&DD

According to the available data, three types of C&DD of the most likely composition are simulated in the sorption experiments, tab. 1. In real C&DD, it is impossible to precisely know its basic composition and the participation of other composite building materials as an ingredient. However, these three types of composites as a mixture with the highest share of the three basic components of C&DD were used to predict the sorption of two-component cation solutions in the case of competitive sorbents. This is especially important because these experiments are closest to realistic conditions.

Samples of wall concrete (C1), older brick (B1), and asphalt (A) with granulation 0.3-0.6 mm were mixed in three different proportions, giving three types of simulated C&DD.

Table 1. Composition of simulated C&DD

C&DD type	C1 sample [%]	B1 sample [%]	A sample [%]
C&DD1	50	40	10
C&DD2	60	30	10
C&DD3	30	60	10

Sorption experiments with simulated C&DD were performed with equimolar two-component solutions of Co^{2+} and Ni^{2+} ions of a total concentration of $2 \cdot 10^{-3}$ mol/L. It follows that the individual concentrations of each cation were $1 \cdot 10^{-3}$ mol/L.

Of all mixtures, the C&DD2 sample showed the most efficient sorption due to the largest amount of wall concrete (60%), which also showed the highest sorption affinity for both tested ions, Co^{2+} and Ni^{2+} in sorption experiments from one-component solutions.

The diagram in fig. 3. shows that the sorption of Co^{2+} and Ni^{2+} ions on all variants of the simulated C&DD is satisfying and ranges from 11.5-18.0%. However, the sorption of Co^{2+} onto C&DD2 that achieved a maximum amount with a sorption capacity of 0.034 mmol/g still was reduced in relation to the sorption from the equimolar two-component solution on the C1 sample, where a satisfactory sorption capacity of Co^{2+} ions was obtained with a value of 0.18 mmol/g. A similar situation was observed in the case of Ni^{2+} ions sorption onto the C&DD2. These ions have the highest sorption capacity of all applied ions sorption on simulated C&DD, approximately 0.041 mmol/g in the case of the C&DD2 sample. However, its sorption is also lower than in the experiments of competitive sorption from a two-component equimolar solution, where the value of nearly 0.18 mmol/g on the C1 sample was obtained. Since the sorption of both investigated ions on all types of simulated C&DD was lower, the potential competitiveness and antagonistic effects of the sorbents themselves in the suspension cannot be ignored, which might be a proposal for further analyses.

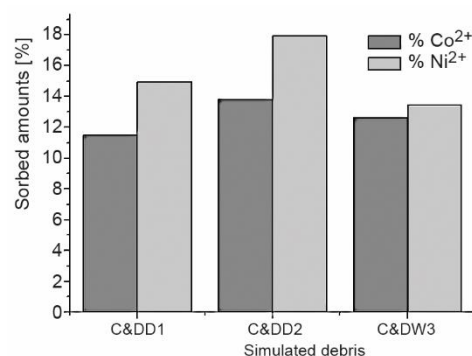


Figure 3. Sorbed amounts [%] of Co^{2+} and Ni^{2+} ions on simulated C&DD

Obtained results significance and conclusion

Based on the obtained results, the use of C&DD and its components in industrial wastewater treatment represents a promising method. Compared with the literature values of sorption capacities for some low-cost sorbents from other studies, tab. 2, the obtained sorption capacities for C&DD components show a satisfactory affinity of these materials for given cations. In general, cement-based materials showed particularly good sorption characteristics.

The highest binding efficiency of Co^{2+} ions was achieved using samples C1, C2, and F. The maximum sorption capacity experimentally determined for sample F was 0.12 mmol/g, while on samples C2 and C1, it was 0.27 and 0.32 mmol/g, respectively. Also, the sorption capacity on the CT sample was substantial and amounted to 0.17 mmol/g.

Ion Ni^{2+} during sorption on sample C1 reached a sorption capacity of 0.55 mmol/g and on F sample 0.30 mmol/g. However, sample C2 showed significantly less sorption with a

maximum sorbed amount of 0.13 mmol/g. Sorption of Ni^{2+} ions on sample B3 was more efficient than on other brick samples, with a maximum capacity of approximately 0.17 mmol/g.

Table 2. Sorption capacities of some investigated waste-based sorbents

Waste-based sorbents	Co^{2+} sorption capacity [mmol g^{-1}]	Ni^{2+} sorption capacity [mmol g^{-1}]	Reference
Fly ash		0.007-0.017	[27]
Zeolite synthesized from fly ash	1.240	1.532	[28]
Activated carbon prepared from apricot stone	0.504		[26]
Rinsed red mud		0.372	[25]
Treated animal bones		0.070-0.490	[16]
Concrete, C1	0.320	0.550	[11]
Concrete, C2	0.270	0.130	[10]
Facade, F	0.120	0.300	[10]
Brick, B1	0.050	<0.130	[10]
Brick, B2	0.060	<0.130	[10]
Hollow brick, B3	0.030	0.170	[10]
Roof tiles, RT	0.065	<0.130	[12]
Ceramic tiles, CT	0.170	<0.130	[12]
Asphalt, A	0.060	<0.130	[10]

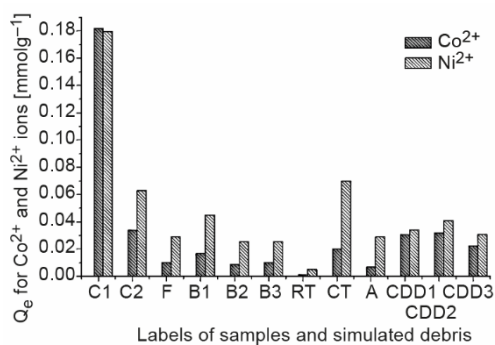


Figure 4. Comparative presentation of Co^{2+} and Ni^{2+} sorbed amounts from two-component solutions on C&DD components and simulated C&DD

4, obtained in sorption experiments from two-component solutions, showed lower values despite a higher concentration of applied ions than in sorption experiments on simulated C&DD. Additionally, bearing in mind that segregation is a demanding and expensive process, the sorption onto C&DD represents a far viable solution.

Sorption experiments of two-component solutions of applied ions on simulated C&DD of different compositions showed lower sorption capacities than those obtained for the same initial concentration of individual cations in competitive sorption experiments. The sorption of both tested ions on all types of simulated C&DD is lower than the sorption from one-component equimolar solutions on samples C1, B1, and A used to prepare simulated C&DD samples. Due to the possible potential competitiveness and antagonistic effects of the sorbents in the suspension, further investigation of the sorption process of these types of suspensions is necessary. However, the sorption capacities of some individual components of C&DD, fig.

The results suggested that cement-based materials, in addition to high affinity for the tested ions, represent a matrix that binds contaminants firmly enough, reduces their mobility and bioavailability, and are suitable for removing Co^{2+} and Ni^{2+} ions. Due to the strong sorbent/sorbate bond, the spent sorbent would not pose a hazard to the environment and could be safely disposed of in a municipal landfill if it has no further use-value. Furthermore, the used sorbent, *i.e.* the sorbent/sorbate system, can be added to freshly prepared concrete or asphalt and, as such, could pose the starting material in civil engineering. Nevertheless, a possible practical application should be preceded by the leaching of contaminants and optimization of the amount of sorbent used in the processes of ion immobilization; thus, the required mechanical properties would not be impaired.

The obtained results should be seen as a basis for further development of technologies and applications that utilize C&DD in the treatment and remediation of contaminated industrial wastewater. Considering the industrial wastewaters represent multicomponent solutions, further research as close as possible to real conditions is needed.

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