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Impact of selected nanoparticles on wastewater treatment efficiency

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ABSTRACT

The widespread application of nanoparticles (NPs) in commercial and industrial products inevitably increases their release into the natural environment which may do harm to human health and cause many environmental problems. The nanoparticles in wastewater treatment systems showed that most NPs are retained in the biological part of the wastewater treatment system. Because some nanoparticles can inhibit or reduce biological activity, nanoparticles trapped in the activated sludge flocs could lessen the effectiveness of wastewater treatment. The most of all laboratory studies regarding of nanoparticles in activated sludge process were carried out with sequencing batch reactors (SBRs) with addition of Ag, ZnO, Cu and TiO_2 nanoparticles. Nanoparticles may be released into surface waters through effluent discharges or into the land through sewage sludge disposal. The treatment efficiency was evaluated based on the total organic carbon (TOC) removal. It was found that at the concentration of 2 mg/L Ag NPs or Cu NPs remained at around 90% of TOC, however increasing the concentration to 3 mg/L led to a decrease in the wastewater treatment. The silver and copper nanoparticles may negatively affect the activity of the activated sludge in SBR.

Keywords: Nanoparticles; Silver; Copper; Wastewater treatment; Bioreactor SBR; Sewage sludge

1. Introduction

Nanotechnology is one of the most important modern and rapidly evolving branches of science that allows us to produce structures that have at least one dimension expressed in nanometers. The dimensions of such nanostructures are usually in the range of 1–100 nm. Because of their very small size, nanostructures possess disparate physicochemical properties, compared with the same materials on the macroscale [1]. The durability, good strength and flexibility associated with nanomaterials have been exploited for many applications. The nanoparticles may be used in the fields of transport, agriculture, medicine, pharmaceuticals and cosmetics. The intense development of nanotechnology being used implies a wide range of applications, but at the same time, it creates the new environmental threats unknown until now [2]. Therefore, the risk of releasing ENPs into the natural environment is potentially threatening to human health and ecosystems [3–7], suggesting that the way and transport of nanoparticles should be investigated to help reduce the risk of their potential negative effect on the elements of environment.

Commonly used articles such as cosmetics including body and hair care products, clothing detergents, cleaning agents, nanotextiles, etc., containing nanoparticles have become a major concern of nanopollution. These products flow into the system of collection sewers and finally flow into wastewater treatment plants (WWTP) [8]. For example, there are large ranging applications of Ag NPs that also increase their risk of potential release into the environment. The studies suggested that a major flow of Ag NPs is from the processing, and consumption to wastewater, as a result

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of Ag NPs applied to textiles and their release during washing [9]. Moreover, with reference to the wider application of CuO NPs in diverse commercial products such as cosmetics, textiles and pigments [10,11], NPs are released to sewer systems and reach WWTPs. Furthermore, the release of nanoparticles could be retained in WWTPs through aggregation, settling, biosorption, precipitation and biomass mediated processes [12], the NPs including CuO NPs and Ag NPs may exhibit potentially toxic or inhibitory effects on the microorganisms in the biological systems. Ag NPs tend to stunt the production of bacterial enzymes which are responsible for metabolism and the cell cycle and influence the structure of the activated sludge flocs [13]. The smaller the size and shape silver nanoparticles become, the more efficient their bactericidal activity is [14]. As a consequence, the nanoparticles negatively influence the performance of WWTPs [15–17].

One of the primary mechanisms for the removal of pollutants is believed to be the adsorption onto the biomass in activated sludge wastewater treatment systems. This reason for this process may be the accumulation of these pollutants in activated sludge [18].

Xu et al. [19] demonstrated that Ag NPs concentration at over 5 mg/L inhibited the chemical oxygen demand (COD), whereas the presence of Ag NPs exerted visible changes in the microbial richness and diversity. Zhang et al. [20] studied the effect of Ag NPs adverse impacts on nutrient removals in sequencing batch reactors (SBRs) and changes of the microbial community structure. It was noted that the presence of 1.0 and 10 mg/L Ag NPs decreased the average removal efficiencies of COD from 95.4% to 85.2% and 68.3% and ammonia nitrogen from 98.8% to 71.2% and 49%, respectively. While Zhang et al. [21] investigated the effect of ZnO and TiO_2 nanoparticles on wastewater treatment showed that both the short and long-term exposure did not adversely affect the pollutant removal of the SBRs. However, it should be taken into account that the oxygen utilization of activated sludge depended on the exposure time on TiO_2 and ZnO NPs [22].

Ganesh et al. [23] investigated the removal of copper nanoparticles (Cu NPs) and copper ions in activated sludge biomass. It was noted that nanoparticles of Cu were removed more effectively at 95% compared with copper ions between 30% and 70% from the wastewater. The dominant mechanisms of copper removal appear to be aggregation and settling (Cu NPs) or precipitation (copper ion) rather than biosorption.

The aim of the study was to determine the effects of silver (Ag) and copper (Cu) nanoparticles on wastewater treatment efficiency in the SBR.

2. Experimental part

2.1. Preparation and characterization of nanoparticles suspensions

Silver (Ag NPs) and copper nanoparticles (Cu NPs) were commercial products purchased from Sigma-Aldrich, USA. The silver nanoparticles used were in the form of nanopowder, about <100 nm particle size, a 99.5% trace metals basis as a dispersant was used PVP (polyvinylpyrrolidone). Also the copper nanoparticles (copper II oxide) were in the form of nanopowder, about <50 nm particle size. A stock suspension of Ag NPs and Cu NPs were prepared by dissolution of 1 g Ag NPs in 1 L of Milli-Q water. Ag NPs and Cu NPs suspension were sonicated for 1 h using Sonics Vibra-Cell VCX 134 (power 130 W, frequency 40 kHz). In the case of the conducted research, the concentration of silver and copper nanoparticles equal 2 mg/L and next 3 mg/L. The above-mentioned doses of nanoparticles were introduced into wastewater.

2.2. Synthetic wastewater

In the experiments was used the synthetic wastewater with a total organic carbon (TOC) value of 150 mg/L. Synthetic wastewater was prepared using glucose, also contained micronutrients including: NH_4Cl – 4.5 g/L, K_2HPO_4 – 4.5 g/L, $MgSO_4$ – 1.95 g/L, NaCl – 9 g/L, $CaCl_2$ – 0.45 g/L and K_2HPO_3 – 4.5 g/L, which was consistent with the composition of synthetic wastewater previously used [24] with a modification. The influent synthetic wastewater was prepared every day.

2.3. Sequencing batch reactor

The SBR with an operating volume of 3.5 L and treating 2.5 L of wastewater per cycle was used in this study. Each cycle consisted of the following steps: 1 h fill and mixing, 8,5 h mixing and aeration, 1 h mixing, 1 h settle and decant, 0.5 h stop zone (Fig. 1). The synthetic wastewater was prepared daily. Aeration was provided to maintain a dissolved oxygen concentration of 5 mg/L during the “aeration” sequence. The air was provided by diffusers at the bottom of the tank. During the stage with the aeration, the opportunity to maintain a dissolved oxygen concentration at the level of 5 mg/L was provided. The study was conducted at room temperature. A reactor working without Ag NPs served as a control.

The activated sludge was taken from a municipal wastewater treatment plant in Częstochowa, Poland. Fresh activated sludge was acclimated by feeding the synthetic wastewater for 1 month.

The SBR was operated with a sludge retention time equalling 12 h and hydraulic retention time (HRT) equalling

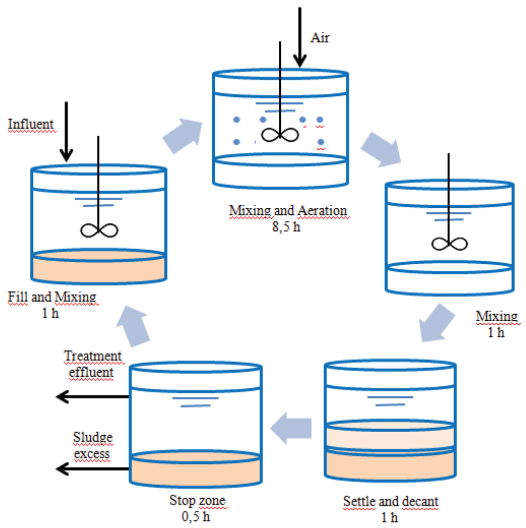

Fig. 1. Schematics of the sequencing batch reactor (SBR) used for the study.

4 h, respectively, and was conducted for 30 d to achieve steady in action. Day 31 to Day 40, 2.0 mg/L of nanoparticles were added to the synthetic wastewater to examine the potential effects of Ag NPs and Cu NPs on wastewater treatment. The influent nanoparticles concentration was further increased to 3.0 mg/L from Day 41 to investigate the response of the wastewater treatment process.

2.4. Analysis

The effluent from the SBRs was analyzed for TOC, suspended solids (SS), volatile suspended solids (VSS), pH and total silver and copper. The filtrate was collected after the SS analysis with cellulose acetate filter pore size of 0.45 μm. SS and VSS was analyzed in accordance to PN-EN-12879 [25], whereas TOC was analyzed by the multi N/C 3100 as organic carbon remaining in an acidified sample after purging the sample with gas. The value of pH was measured with a pH meter called the Elmetron Model CP-411 pH meter according to PN-9/C-04540/05 [26].

The concentration of silver and copper was determined by atomic absorption spectrometry on the Spectro Arcos ICP-OES spectrometer.

Microbial observations of activated sludge were conducted and, based on their results, the sludge biotic index (SBI) proposed by Madoni [27] was determined. The SBI describes on the different sensitivities of each protozoan group sensibility on the physical, chemical and operational conditions. Microscopic observations performed on the basis of appropriate diagnostic keys [28].

In addition, the percent removal efficiency was calculated using the following equation:

$$\text{TOC removal}(\%) = \frac{C_{\text{infl}} - C_{\text{effl}}}{C_{\text{infl}}} 100\% \quad (1)$$

where C_{effl} (mg/L) is the TOC concentration in the effluent after the contact time, and C_{infl} (mg/L) is the initial TOC concentration in the influent.

3. Results and discussion

3.1. Effects of Ag NPs and Cu NPs on treatment efficiencies in SBR

In order to work well, the SBR was operated for 30 d. With an average influent TOC concentration of 163 mg/L, the removal efficiencies were obtained from these pollutants finally at 90.5%. For the reactors on the 31st day operation, 2.0 mg/L of Ag NPs or Cu NPs were added to influent wastewater and then increased to 3.0 mg/L from day 41 (Figs. 2–4).

Fig. 2 shows that the removal efficiencies of TOC indicated a considerable decrease to 88.43% at an influent Ag NPs concentration of 2.0 mg/L, and further to 54.84% at 3.0 mg/L Ag NPs addition. Slightly better results were achieved in the case of Cu NPs that the removal efficiencies of TOC indicated a decrease to 88.91% at an influent Cu NPs concentration of 2.0 mg/L, and further to 67.16% at 3.0 mg/L Cu NPs addition (Fig. 3). Whereas Fig. 4 shows that removal of TOC in the control bioreactor was with a removal efficiency of around 86.5%–92.3%.

A study by Qiu et al. [29] has shown that at 1.0 mg/L Ag NPs and 5.0 mg/L Ag NPs, around 96.7% and 95.1% in COD removal efficiencies was obtained. Zhang et al. [20] showed that 50-d of exposure to 0.1 mg/L Ag NPs exhibited no difference with the controlled one, whereas the presence of 1.0 and 10 mg/L Ag NPs could evidently reduce the average removal efficiencies of COD, NH_4^+-N. During the study conducted by Quan et al. [30], it was shown that 69 d of exposure to 5 and 50 mg/L Ag NPs to two reactors did not influence the removal of COD from wastewater. Wang et al. [31] showed that the COD removal efficiency kept a relatively steady value at about 91% at 0.10 mg/L CuO NPs. But at 30 mg/L CuO NPs, the COD removal efficiency decreased to 89.5%.

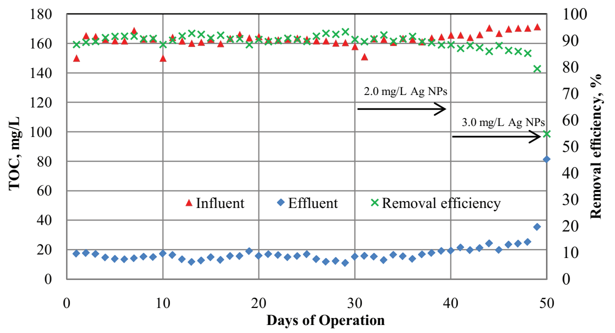

Fig. 2. Effect of Ag NPs on TOC removal in the sequencing batch reactor.

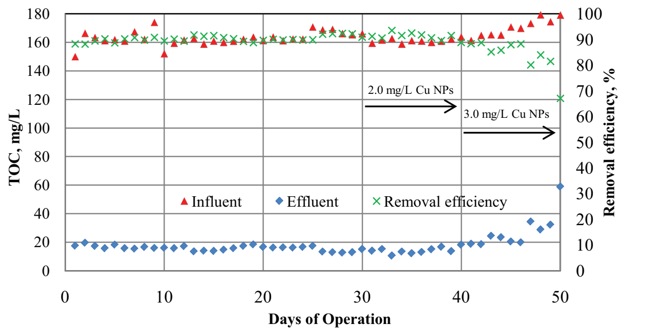

Fig. 3. Effect of Cu NPs on TOC removal in the sequencing batch reactor.

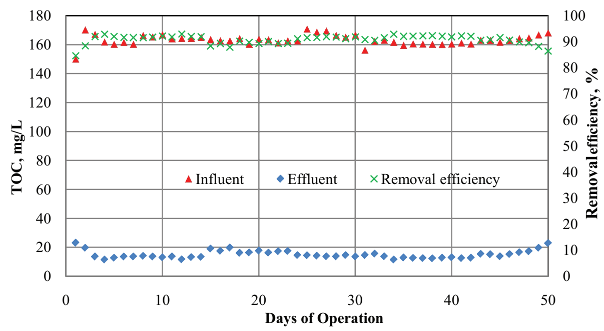

Fig. 4. Changes in TOC removal in the control sequencing batch reactor.

The changes of the VSS in the experiments are illustrated in Fig. 5. In the studies, a marked increase in the concentration of the VSS was followed up to day 20, and then a slight drop on the 30th day. After addition to influent wastewater, 2 mg/L of Ag NPs and Cu NPs did not change notably within the VSS, whereas after increased to 3.0 mg/L a marked decline occurred. In the test control bioreactor, the VSS concentration was similar to the other bioreactors until day 13. A slight drop in the VSS concentration then occurred on day 19. Then, after a slight drop of VSS concentration, in the control reactor remained stable until the end of the experiment. As seen in Fig. 5, a much larger drop in the VSS concentration was recorded for the bioreactor to which was added to the Ag NPs. The presence of the nanoparticles led to a decrease in the rate of organic material elimination throughout the duration of the research.

Table 1 shows how pH values have changed in all three bioreactors. pH measurements in the bioreactor with the Ag NPs showed that the initial average value was 6.51, declined to pH 6.15 in the time wherein at 3.0 mg/L Ag NPs addition. In the case of other bioreactors, the decline of pH was also small.

On the basis of observations on the activated sludge microfauna, the SBI was evaluated [27]. Table 2 shows the results obtained on the activated sludge microfauna in all three bioreactors. The obtained values of SBI allow one to define a quality class of the activated sludge. As seen up to the 47th day of research in all three bioreactors, the first class according to Madoni [27] was indicated. Therefore, the activated sludge was very well colonized and stable sludge, was characterized a good the biological activity. At the end of the experiment, the activated sludge relegated to the second class for the bioreactors with added nanoparticles. The decrease of SBI values was due to the reduced microfauna density, changes in the dominant microorganism communities with which the activated sludge functions properly [32]. In these cases, the activated sludge was well colonized and stable, but indicated the lower biological activated.

Microfauna was widely diversified and mainly composed of crawling and attached ciliates, testate amoebae and small metazoa (Fig. 6). The microfauna of the activated sludge composed of creeping and sessile ciliates, testate amoebae, rotifers and nematodes indicates that the activated sludge is healthy, sufficiently oxygenated, low-loaded, active and ensures the high quality of treated wastewater.

The concentration of nanoparticles in the outflow with SBRs is shown in Fig. 7. The influent Ag NPs concentration of 2.0 mg/L was not detected in the first days, but increased after 10 d to 0.0108 mg/L. Whereas the influent Ag NPs was increased to 3.0 mg/L rose to 0.0508 mg/L at

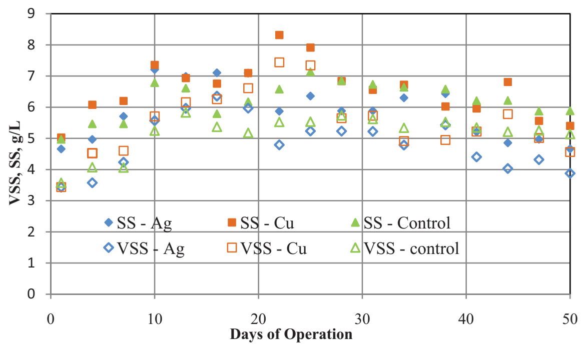

Fig. 5. Effect of Ag NPs and Cu NPs on suspended solids and volatile suspended solids in the SBR effluents.

Table 2
Sludge biotic index for the activated sludge in individual SBRs depending on the added nanoparticles to wastewater

Operation time	Ag NPs, SBI	Cu NPs, SBI	Control, SBI
30	8	9	9
31	8	8	9
34	8	9	9
38	9	9	9
41	8	8	8
44	8	8	8
47	8	8	9
50	7	7	8

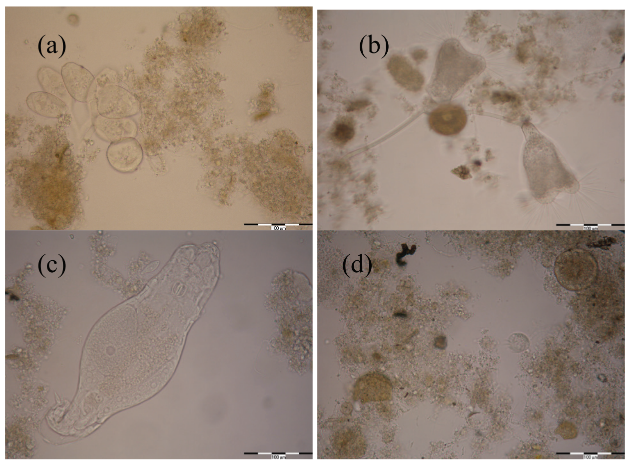

Fig. 6. Control images of activated sludge in the bioreactors: (a) and (b) Ciliata, (c) rotifers, (d) *Arcella* sp.

Table 1
Changes of the pH of the mixed liquor in the SBR

Operation time	Ag NPs		Cu NPs		Control	
D	Average value	Range	Average value	Range	Average value	Range
1–30	6.51	6.15–6.82	6.52	6.10–6.76	6.60	6.27–6.96
31–40	6.21	5.98–6.47	6.22	6.03–6.61	6.32	6.10–6.60
41–50	6.15	5.85–6.45	6.29	6.19–6.48	6.28	6.10–6.67

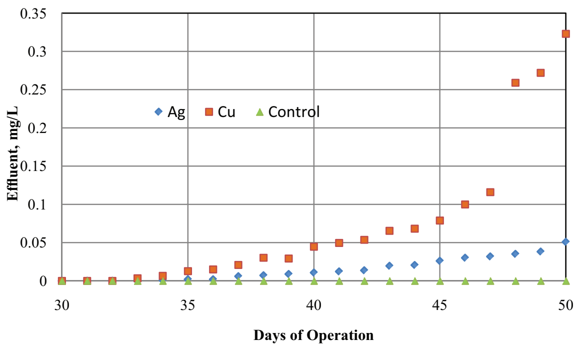

Fig. 7. Total Ag concentration in the SBR effluents.

the end of the experiment. A similar trend was observed when the influent Cu NPs, the concentration of total copper was not detected in the first days, but increased after 10 d to 0.0108 mg/L. Whereas at the end of the experiment rose to 0.323 mg/L. As shown in Fig. 7, the silver nanoparticles were much better removed than the copper nanoparticles. The obtained results showed that treated wastewater included a low concentration of Ag and Cu nanoparticles. This result confirmed previous studies which stated that activated sludge successfully retains Ag NPs during wastewater treatment [33]. The studies which were carried out showed that more than 95% of silver nanoparticles, which penetrated into sewage sludge, were removed during the sewage treatment process. It was estimated that the remaining 5% was retained in the purified sewage [34]. Whereas Park et al. [35] obtained 99% removal of Ag NPs through the activated sludge within 24 h using the concentration of 1 mg/L of Ag NPs.

During the studies, the concentrations of silver and copper nanoparticles at the effluent were determined, which the permissible value for silver is 0.1 mg Ag/L whereas for copper 0.5 mg Cu/L [36]. The silver concentration was exceeded only in final cycles, while the concentration of copper did not exceed the permissible value. However it should be noted that the potential impact of these nanoparticles in the water environment will be a concern.

4. Conclusions

In the study, the effects of silver (Ag) and copper (Cu) nanoparticles on wastewater treatment efficiency in the SBR were investigated. The following conclusions were formulated on the basis of the studies:

1. Ag NPs and Cu NPs at 2.0 mg/L and 3.0 mg/L were found to have remarkable effects on TOC removal in an SBR. The removal efficiencies of TOC indicated a considerable decrease of 88.43% and 54.84%, respectively, in the case of Ag NPs, in the case Cu NPs, respectively, 88.91% and 67.16% after continuous exposure of nanoparticles a specified time.
2. The concentration of Ag NPs and Cu NPs in the outflow with SBRs was not detected in the first days, but increased, however, to 0.0508 mg/L for Ag NPs and to 0.323 mg/L for Cu NPs at the end of the experiment. The obtained results showed that treated wastewater included a low concentration of Ag and Cu nanoparticles.
3. Based on the obtained results, it can be stated that according to the classification given by Madoni [27], the active sludge can be classified as the first class. The activated sludge was stable and properly colonized. However, in the case of the final stage of the experiment, in the bioreactors with added Ag NPs and Cu NPs, active sludge can be qualified for the second grade.
4. The research showed that concentrations of nanoparticles in the range tested only slightly diminished the efficiency of removing pollutants.
5. The main removal pathway of Ag NPs and Cu NPs was via sorption as well as possible aggregation and sedimentation onto the sludge. The silver and copper nanoparticles may negatively affect the activity of the activated sludge in SBR.

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