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Removal of detergents in car wash wastewater by sub-surface flow constructed wetland

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Abstract
Suspended substances in car washes can often be easily removed by physicochemical processes. The main problem is removing dissolved substances such as detergents from the water. In this study, the biodegradable substance Sodium Lauryl Sulfate (SLS) was removed from the car wash wastewater by the use of *Phragmites australis* in the subsurface constructed wetland. For this study, 4 plexiglass reactors having an effective volume of 10.8 L with the dimensions of 15 cm × 45 cm × 20 cm were used. The experiments were conducted with vegetation, which was called SCW, and without vegetation, which was named the control group (CG), as two groups. A serial connection of two reactors was performed for each group. Up to 90%, detergent removal was observed with the vegetation in the SCW with a loading rate of 75 L/(m^2.d) The effluent quality showed that the treated water can be reused car washing or irrigation for landscaping.

1. Introduction

The car wash industry uses a huge amount of freshwater such as 200 L-900 L depending on the car wash type. The chemical composition of car wash wastewater contains greases, detergents, waxes, salts, dust, metals, and organic matter [1]. Sand, dust, and detergent are reported as common contaminants in wastewater [2].

Detergents, which may consist of surfactants, adjuvants, bleaches, and several additives functionality, counteract the external tension of the aquatic to form micelles and eradicate grime. Surfactants are in control of the cleaner influence in washing products, and they can be categorized into four groups: anionic, cationic, non-ionic, and amphoteric [3]. For many years, Sodium Lauryl Sulfate (SLS), an anionic detergent, has been utilized as an antibacterial surfactant [4], [5]. It accumulates in seawater and sediments because of untreated wastewater discharges. In 2001, a study by Della Croce et al. reported that an average of 60,000 tons of cleansers are discharged into the Mediterranean annually [6].

Some car wash wastewater is discharged directly into municipal wastewater systems [7], while others are treated using a variety of technologies, including primary sedimentation or filtration techniques for the removal of suspended solids and oil. These are followed by the application of detergent removal processes such as membrane bioreactors or coagulation processes. Finally, the treated water is reused or discharged into sewer systems. The detergent in the wastewater can be filtered by hydrophobic interaction chromatography, size exclusion chromatography, ion-exchange chromatography, dialysis, and ethyl acetate extraction [8]. However, these promising technologies are generally expensive and require a skilled workforce. Car wash services are usually small in size, and it is difficult to employ qualified operators for the above-mentioned high-tech treatment units. Therefore, a system that does not produce sludge and does not require chemicals

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should be established with a low initial investment and operating costs for sustainable operation.

As stated in recent works, the wetland process has shown promising applications for treating grey wastewater with a high detergent concentration. For example, in linear alkylbenzene sulfonate (LAS), which is another common anionic surfactant, the removal efficiency was measured at 77% after 15 days of HRT and the treated water was used for gardening [3]. Another research was focused on horizontal flow wetlands cultivated with helophytes, graminoids, tropical, and subtropical plants. The removal efficiency of chemical oxygen demand (COD), biochemical oxygen demand (BOD), and total suspended solids (TSS) were reported more than 85% [9].

As a result of contracted wetlands (CWs) being considered as an economical and sustainable alternative for wastewater treatment [10], reusing technology is being investigated and improved in most districts due to serious water shortages in several countries. Some research on CW effluents indicates that these effluents can be used for irrigation, flushing toilets, and industrial purposes [11]. With this technique, if approved for the car wash industry, water problems of scarcity and contamination might be solved. So, the results of the experimental study were also evaluated based on reusability.

In the study, the objectives were; (1) to evaluate the performance of the subsurface constructed wetland for removal of SLS based on COD, turbidity, alkalinity, and conductivity parameters, (2) to assess the subsurface constructed wetland operation condition base on vegetated or non-vegetated, (3) to evaluate the effect of hydraulic retention time on the removal efficiency, and (4) to evaluate the reusability of the effluents regarding feasible option or not.

2. Materials and Method

2.1. Synthetic Wastewater Preparation

Synthetic wastewater was prepared by adding 1 ml of detergent taken from Shell Petrol A.Ş. to 1 L of tap water. It is a biodegradable detergent based on SLS. The chemical structure of SLS and its properties are given in detail in the literature [12]. The characterization of synthetic wastewater is given in Table 1.

Table 1. The characterization of t synthetic wastewater

Parameter	Value
pH	8.55 ± 0.3
Turbidity (NTU)	0.64±0.4
Alkalinity (mg $CaCO_3$/L)	129±7
Conductivity (µS/cm)	402.3±48.6
COD (mg/L)	142.2±19
Loading Rate (L/(m^2.d))	75

2.2. Experimental Set-Up and Procedure

Subsurface constructed wetland experiments were conducted in batch mode using 4 Plexiglas reactors having an effective volume of 10.8 L with the dimensions of 15 cm × 45 cm × 20 cm (Figure 1). Two reactors were used as main experimental groups (SCW), and the others were used as control groups (CG). The two reactors in the group were connected by pipeline so that they could be connectedin series. Those pipelines were mounting influences and effluents of the reactors which opened a diameter of 1 cm spherical geometry nozzle. The first reactor of each group was fed synthetic wastewater by a peristaltic pump (Perimax, SPETEC, Germany).

A media (pebble stone and soil) was put in all reactors. Because its size affects the system's ability to function, this particle size distribution was designed following numerous recommendations for tolerable hydraulic conductivity; the declining magnitude along the stream route decreased the risk of clogging [13]–[15], which is crucial for the long-term stabilization of SCWs. The depth of media was set at 7 cm of pebble stone (sizes ranging between 15-80 mm) and 9 cm of red soil in whole reactors. Reed plants (*Phragmites australis*) were planted only in two reactors, called the experimental SCW reactors. The reed plants were collected from the natural area side of the creek, flowing around the Harran University Campus, Turkey. Their roots were carefully put inside the pebble stone in the SCW.

At the beginning of the research, the whole reactors in each group were tested by feeding 5 L of tap water daily to observe the stability of the reactors. To understand the stabilization condition of the reactors, turbidity values of the influent wastewater and the effluent water were observed daily until they had stabilized.

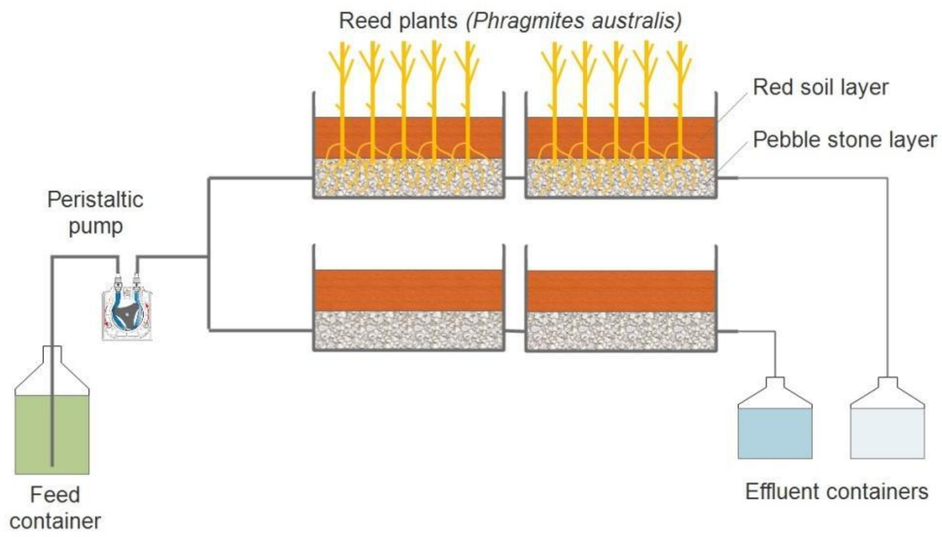

Figure 1. The configuration of the lab-scale subsurface constructed wetland with vegetation (SCW) and without vegetation (control group (CG))

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2.3. Analytical methots

Samples were centrifuged at 3000 RPM (3024 RFC) for 10 minutes prior to analysis. The conductivity and the temperature in the aqua were measured by WTW equipment (Multi (3620 IDS) probe, Weilheim, Germany), and the pH was determined using a pH-meter (pH 211 Microprocessor, Hanna Instruments, Woonsocket, RI, USA). The alkalinity and COD analyses were carried out according to 2320-B, and 5220-A (closed reflux method), respectively [16].

Concentrations of detergents in aqueous solutions can be determined by different measurement methods according to their types. It can be measured as total phosphate by measuring the phosphate in their structure, as well as mass spectrophotometry or chromatographic methods. A study by Lau *et al.* mentions that detergents can also be measured as COD equivalents [17]. In the study, detergent was assessed by COD analysis.

The removal efficiencies of COD and alkalinity were determined as a percentage and calculated as:

$$R\ (\%) = (1 - (Ce/Ci)) * 100 \quad (1)$$

initial days of operation, only tap water was fed to the reactors to clean initial impurities based on reactor media and red soil. After 13 days of operation, it was observed that each reactor stabilized (influent and

where Ci and Ce represent the concentration of a particular component in the influent and in the effluent flow of the reactor, respectively.

2.4. Evaluation of reusability

Overall, reclaimed wastewater transportation and dissemination denotes a higher charge in an application project and subsequently limits its economic sustainability [18]. SCW applications are more appropriate for the countryside where self-serve car washes have generally. It is important to understand that its effluents are applicable to the car wash again or irrigation for landscaping. Therefore, the reclaimed wastewater quality was evaluated in Cyprus (KDP 379/2015), Italy (Ministry Decree (DM) 185/2003), and USEPA (2012) guidelines, as done before by Arden and Ma, (2018) and Otter et al., (2020).

3. Results and Discussion

3.1. Treatment Performance

The stability of the reactors was monitored by influent and effluent turbidity measurements. During the effluent turbidity were equalized). After this stabilization period, the experiments were performed at a wastewater loading rate of 75 L/(m^2.day) by feeding with synthetic wastewater.

The electrical conductivity (EC) indicates the total amount of ions (nitrogen, phosphorus, etc.). In our study, synthetic water made with dissolved detergent in tap water was used. Therefore, the influent wastewater included mainly phosphate coming from detergent and other ions in tap water. To observe detergent changes in the study, the EC value was preferred mainly because its analysis simply and rapidly uses inexpensive sensors. Hence, to find a correlation between the detergent concentration (DC) in the solution and the EC, correlation experiments were carried out. The result of the correlation experiments indicated as

$$EC= 17.164 \times DC \quad (2)$$

The correlation coefficient of 0.98 indicates a strong positive correlation (Eq (2)). Although the correlation between them in the synthetic wastewater was found well, a similar correlation between the DC in the effluent and the EC value in the effluent water was not observed**.** Moreover, the EC value was observed to be more affluent than the influent at the same reactors (Figure 2). Besides, the EC value change of each reactor was also observed to be dissimilar. While the average EC value in the influent wastewater was measured as 402.3±48.6 µS/cm (Table 1), the values from both the SCW effluent and the CG effluent were measured as 464.2±31.8 µS/cm and 454.6±34.1 µS/cm, respectively. This situation might be caused by the presence of some impurities in the effluent water. The impurities may originate from biochemical or chemical products such as bacterial and/or fungal products, organic substances, some ions getting from the stone surfaces, and organic leakages of photosynthetic activity production from the root in the reactors [20].

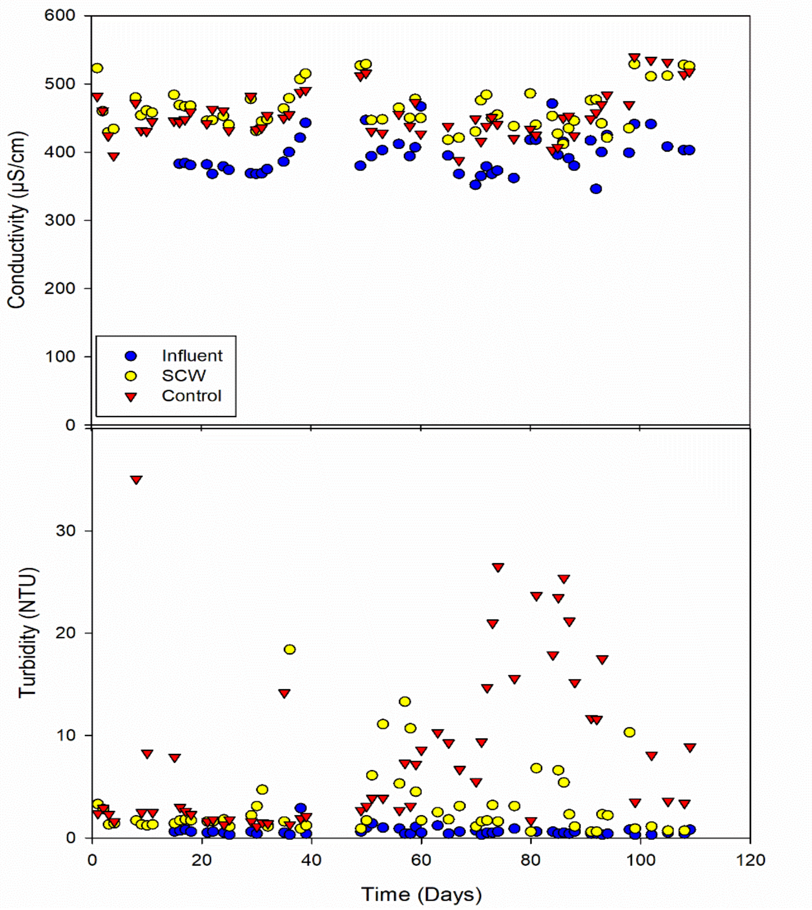

Figure 2. Conductivity and turbidity variations.

In the first 40-day start-up period, the effluent turbidity values of the SCW and the CS were determined at the levels of 2.50±3.5 and 4.3±7.2 NTU, respectively (Figure 2). However, between 40 and 108 days, the turbidity values in the effluent waters of the SCW and the effluents of the control reactors were measured averagely as 3.55±3.46 and 10.8±7.56 NTU, respectively. An almost similar trend was observed for the COD removal efficiencies in each group, as shown in Figure 3.

Pebbles used as reactor filter material are adsorption surfaces in the system. Therefore, some of the particles, such as microorganisms, their products, and other impurities, can be adsorbed onto pebbles. These microorganisms make microbial biofilms, communities of microorganisms attached to a living or inert surface in an aqueous environment and surrounded by a matrix of extracellular polymeric substances [21]. When these attachments on the surface of the pebbles reach a certain size, such as in trickling filters (regeneration of the filter material), microbial flocks can be observed in the effluent water [15]. Therefore, if these microorganisms in SCW grow without control, the SCW process may block and create short circuits. In the study, as mentioned above, the turbidity value between 40 and 108 days was determined to be higher than that in the feed. The reason for the increase may be microorganisms detached from the sorbent. On the other hand, Figure 2 indicated that the turbidity value of the SCW became stable more quickly than the CG. The probable reason for the difference between the two systems could be thatthe SCW system has a natural filtration mechanism due to the presence of plant roots.

The concentrations of detergents in aqueous solutions can also be determined by different measurement methods according to their types. It can be measured as total phosphate by measuring the phosphate in its structure as well as mass spectrophotometry or chromatographic methods. A study by Lau *et al.* reported that detergents can also be measured as COD equivalents [17]. Correlation experiments were performed to find a correlation between the detergent concentration (DC) in the solution and the COD value. The result of the experiments is indicated as

$$COD= 161.93 \times DC \quad (3)$$

The correlation coefficient of 0.99 indicates a strong positive correlation (Eq (3)). As mentioned above, a similar correlation was observed between DC and EC. However, the correlation between the DC in the effluent and the EC value in the effluent water was not determined as suitable. Hence, to observe the change of the DC in systems, the COD parameter was preferred in the study. The COD analyses were started after the 13th day since the systems were fed only with tap water for the first 13 days. Figure 3 shows that the treatment mechanisms reached a steady state in the first 40 days and their components adapted to the ambient conditions. In this period, the average COD removal efficiencies in the SCW effluent and the CG effluent were calculated as 52% and 47%, respectively. Figure 3 indicates that the variance between the effluent COD concentrations of the SCW and the CG was growing after the 40^{th} day. This situation can be interpreted with the adaptation of the reeds placed in the SCW to the environment and the increase of biochemical reactions in these reactors. Between the 40^{th} and 108^{th} days of the experiments, the effluent COD concentrations of the SCW and the CG were measured as 36.9±12.9 mg/L and 80.9±17 mg/L, respectively, and the calculated removal efficiencies, 74%, and 44%, respectively.

Throughout the study, the average effluent COD value of the SCW and the CG was determined as 45.8±22.1 mg/L, and 77.5±19.8 mg/L, respectively. The possible reasons for the removal of the CG are (1) the use of detergent in oxidation/reduction reactions by the microorganisms in the environment, or (2) adsorption on stone surfaces. Dhouib et al., (2003) reported that *Citrobacter braakii* species can reduce sodium lauryl ether sulfate-based surfactants at a rate of 0.065 g/(L.h) under 20-hour HRT conditions. Other studies reveal that microorganisms can reduce anionic surfactants at various rates [23]–[25]. Another removal of surfactants from the water environment may also be adsorbed by the adsorption process. Some of the adsorbent materials, such as peach kernels, olive seeds, natural asphaltite, and coal tar pitch, have been reported in the literature for this purpose [26].

The COD removal efficiency was observed more in the SCW than in the CG. It can be interpreted as the reed roots in this reactor helping the biodegradation process or directly taking the SLS into the plants. Various CW applications in the treatment of greywater and industrial wastewater (paper industry, petrochemical, textile, metal processing, alcohol production, fish and seafood processing, dairy wastewater, and food industries, etc.) in the literature were reported similarly [10], [27]. Moreover, the existence of vegetation upturns water retention time, so the contact time between substrate materials and contaminants is extended. It could cause the adsorption of impurities onto sorbent resources in the CWs to be enhanced. According to P-sorption capacity investigation in the literature, the vegetated batch reactor demonstrated better performance than the non-vegetated ones [28]. This study shows that the detergents in car washing wastewater can be removed with SCW (including vegetation) and non-vegetated ones. On the other hand, if the SCW (including vegetation) system is preferred, its removal efficiency can provide more than a non-vegetated system.

No significant difference was observed in pH between the SCW and the control group. The pH values of influents and effluents from both the SCW and the CG were measured as 8.55 ± 0.3, 8.18 ± 0.48, and 8.18 ± 0.47, respectively (Figure 4).

During the first 40 days in the experiments, start-up period, the alkalinity value of the effluent from the SCW and the CG was observed to be almost the same value, 174.7±23.9, and 158.6±16.3 mg $CaCO_3$/L, respectively (Figure 4). After the start-up

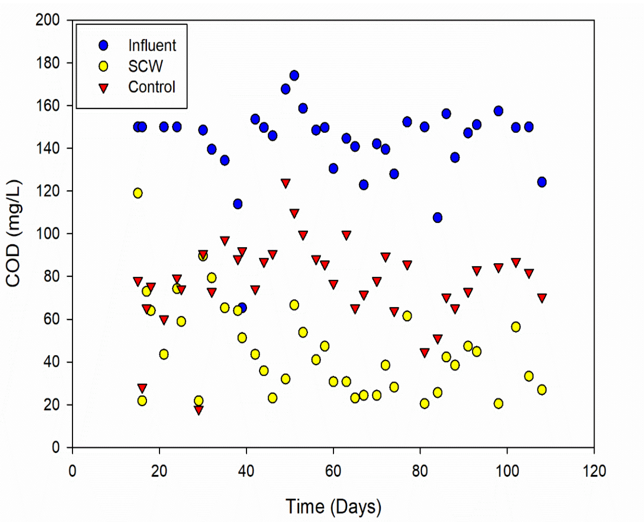

Figure 3. Chemical oxygen demand variations throughout the study

period, the alkalinity value of each group was shown to be uptrend. The alkalinity value between the 40th days to 108th days both in the SCW and in the CG increased from 174.7±23.9 to 220.7±21.2 mg $CaCO_3$/L (the increasing ratio is 71%), from 158.6±16.3 to 189.4±22.1 mg $CaCO_3$/L (the increasing ratio is 46%), respectively. The reason for these uptrends might be an influence of biochemical reactions in the reactors. There are several possible predictions for biochemical growth within the reactor. One of them may be the reduction of both approximately 35 mg/L sulphate in the tap water and sulphate in the detergent structure via the oxidation process due to having organic substances in the soil. Another possible reaction might be the transformation of existing microorganisms into organisms in particulate form and then into dissolved and biodegradable forms of organic matter according to the Dead Regeneration Model [29]. The conversion of these organics to volatile fatty acids in an oxygen-free environment may be another factor that affects alkalinity concentrations.

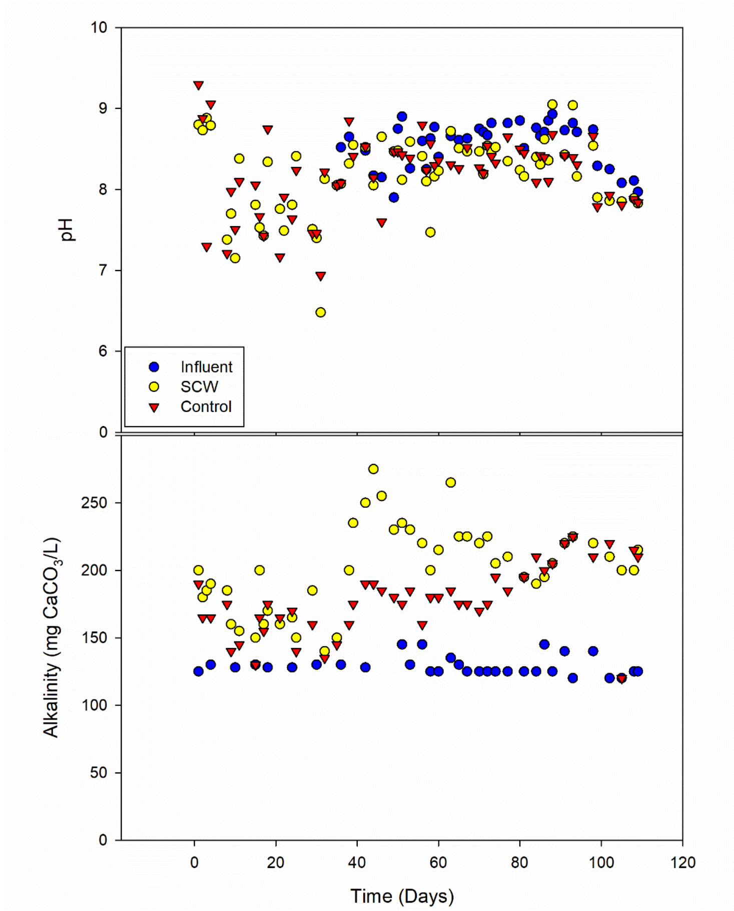

Figure 4. pH and alkalinity variations throughout the study.

3.2. Treatment Performance

A few studies reported car wash wastewater treatment for reuse in the literature. In the study, regarding the United States Environmental Protection Agency guidelines and European Country Standards, the water quality of end-uses of recycled water was evaluated as done by previous researchers [10], [19].

During the study, no significant difference was observed in the pH between influent and effluent for each group. The pH levels were found limit within the unrestricted and also restricted levels of the USEPA guidelines (2012). Besides, in terms of turbidity, although feeding water was prepared with tap water, therefore the turbidity in influent water was very low, and turbidity levels of both effluents are measured <10 NTU. The values are not suitable because the turbidity guideline for unrestricted reuse is 2 NTU [30].

While the load of the influent COD value was 142.2±19 mg/L, the average COD effluent values in the SCW effluent and the CG were measured as 63.5±26.3 mg/L and 70.8±23.9 mg/L, respectively.

The study of Otter et al., (2020) reviewed wastewater reuse quality requirements. Regarding their study, the effluents based on COD value were archives of Cyprus (KDP 379/2015) and Italy (Ministry Decree (DM) 185/2003) standards. Before using the treated water for irrigation of the landscape or reuse for the car wash industry, the treated water should be investigated in detail and diminish environmental risks.

4. Conclusion and Suggestions

Using a high amount of wastewater with a low pollution load produced in the car wash industry is important to save water, and money and to protect environmental health. Dissolved detergent is the most important component in the car wash water. Since oil and suspended solids do not dissolve, they can be easily removed from the water by a simple precipitation/filtration and flotation process. SCW has a high potential for the treatment of car wash water due to its advantages such as low installation cost, low maintenance requirement, and not needing a qualified person. During the study, the average COD removal efficiency was determined to be around 68%, and the maximum observed COD removal was calculated to be 87%. The operation of the SCW with vegetation (*Phragmites australis*) showed a higher removal ratio than without the vegetation group. Even though the effluent qualities were achieved by some European Standards for reuse, we did not focus on bacterial conditions, and therefore, a disinfection process should be installed in the system before the application of the system.

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Contributions of the Authors

The authors confirm that the contribution is equally
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Conflict of Interest Statement

There is no conflict of interest between the authors.

Statement of Research and Publication Ethics

The study is complied with research and publication ethics

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