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Supplementary Treatment of Wastewater by Using Ecological Lime Derived from Eggshell Waste – A New Sustainable Strategy for Safe Reuse

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ABSTRACT

Wastewater from wastewater treatment plants (WWTPs) often requires further treatment before it can be safely reused. Lime is a common and affordable material used for this purpose, but its production can generate significant environmental impacts. This study developed an eco-friendly and effective lime substitute from eggshell waste for wastewater treatment. First, pre-treated wastewater effluent from WWTP El Jadida, Morocco, was collected and characterized. It was found that COD, BOD_5, and TSS values showed non-conformity from Moroccan discharge standards, as well as high concentrations of heavy metals such as cadmium (Cd), zinc (Zn), aluminum (Al), chromium (Cr), manganese (Mn), lead (Pb), silver (Ag), beryllium (Be), copper (Cu) and cobalt (Co). These pollutants represent a potential risk to human health and the environmental ecosystem. To reduce this pollution, the optimal mass of lime powder obtained by thermal treatment of eggshell waste was determined by testing a concentration series of 6, 12, 18, 24, 30, and 36 $g \cdot L^{-1}$. The findings confirmed that the addition of the optimal dose of prepared lime (24 $g \cdot L^{-1}$) resulted in a significant reduction in pollution parameters, with abatement rates of 77% for BOD_5, 63% for COD and 66% for TSS, respectively. Furthermore, the eco-friendly lime substitute also showed promise in reducing the colorization rate for dyes by 84% and removing heavy metals through precipitation. However, the generated by-product loaded with toxic pollutants should be encapsulated in eco-materials to ensure safe operation and contribute to a sustainable management strategy for wastewater treatment.

Keywords: heavy metals, adsorption, eggshell, wastewater, decoloration.

INTRODUCTION

Water is arguably one of the world's most precious resources. Water scarcity is a problem for many countries. In recent years, water shortages have increased due to its use in many areas, wastage and climate change (Pandeya et al., 2021; Yang et al., 2020), particularly in arid and semi-arid countries (Pandeya et al., 2021; Tapsuwan et al., 2022). In the same context, Morocco has recently experienced a huge water deficit, and its water resources are among the lowest in the world (Ouchouia and Chaouki, 2022; Er-Raki et al., 2021). Moreover, the potential of natural water resources is estimated at 700 m^3/capita/year, or 22 billion m^3. This potential is accepted worldwide as a critical threshold indicating the latent onset of a water crisis. Limited water resources are compounded by water contamination, an emerging problem caused by population growth (Gondo et al., 2020; Saravanan et al., 2022). Thus, domestic and industrial effluents represent all liquid discharges resulting from the various industrial processes of transforming raw materials to produce consumer goods and/or manufacture industrial products (Farkas et al., 2020; Prabakar et al., 2018; Wang et al., 2021). Their quantity and quality vary according to their source, the industrial activity and the process used (Xiao et al., 2021). These discharges contain a broad spectrum of chemical contaminants in dissolved or solid form, such as organic and inorganic matter, solvents, polymers, heavy metals and biological pollutants (Kouali et al., 2022; Madhav et al., 2020a; Clarke et al., 2018; Bhat et al., 2022a).

These contaminants can have harmful effects on the environment and human health (Madhav et al., 2020b). Organic matter, such as faecal residues, detergents and food waste, can cause eutrophication, can cause a proliferation of algae that can harm aquatic life (Ahmed et al., 2021). Heavy metals are carcinogenic and toxic to both humans and animals (Fu and Xi, 2020). Chemicals, such as pesticides, herbicides and solvents, can also be harmful to health. Finally, micropollutants, such as hormones, drugs and personal care products, can have adverse effects on health (Sharma et al., 2022). In order to break down pollutants rich in organic matter or micro-pollutants, the use of living organisms is necessary. These organisms are commonly used in processes such as activated sludge purification, lagoons, and planted filters. However, this type of treatment has a number of drawbacks. It requires a vast surface area for its implementation, making it one of the bulkiest treatments. It is also considered the most time-consuming, as it takes a substantial amount of time for living organisms to eliminate all traces of organic pollutants. Moreover, it can generate odors during the activity of the bio-organisms, hence the need to keep these treatment plants away from urban areas (Ma et al., 2021).

The elimination of inorganic pollutants, particularly heavy metals, which are among the most harmful and difficult to remove, can be ensured by several treatment techniques (Hu et al., 2020). These include the use of ion exchangers, which require periodic regeneration of the resins and significant consumption of water and energy. Resins are sensitive to organic pollutants, which can lead to the release of unwanted ions after saturation, thus constituting an additional source of pollution (Bashir et al., 2019). Another technique adopted for the removal of inorganic matter is the use of membranes, which can be effective but costly and require regular maintenance (Obotey Ezugbe and Rathilal, 2020). Photocatalysis is also used to treat water, although it has drawbacks such as the need for a light source, the slowness of the process, and susceptibility to contamination. Electrocoagulation, which uses an electric current to coagulate pollutants, is another method of treating wastewater. The coagulated pollutants are then removed by sedimentation or filtration. However, electrocoagulation can produce contaminated sludge, requiring further treatment and potentially becoming an additional source of pollution (Tahreen et al., 2020). Finally, adsorption is a remarkably effective technique for removing a wide range of contaminants, from organics to heavy metals, pesticides and viruses. Its operational simplicity makes it easy to implement and manage, while its relatively modest cost makes it an economical option. What's more, adsorption is environmentally friendly, contributing to an ecosystem-friendly approach to water treatment (Chai et al., 2021a; Chai et al., 2021b).

As a natural, renewable material, shells offer a promising opportunity for wastewater treatment. Composed mainly of calcium carbonate, an alkaline compound with beneficial properties for wastewater treatment, they offer a number of advantages. Firstly, they are abundantly available at an affordable cost, making them accessible on a large scale. Secondly, they are remarkably effective at eliminating a wide range of pollutants. Thirdly, they are non-toxic and environmentally friendly (Obey et al., 2022). These shells can be used in a variety of ways in wastewater treatment. As a flocculant, they are introduced into raw wastewater to promote the formation of flakes, leading to the precipitation of solids and organic pollutants. As a bacterial substrate, shells can support the bacteria responsible for decomposing organic matter. As filters, they can be used to retain particles and pollutants. Finally, as an adsorbent, they prove to be an effective means of ensuring the elimination of inorganic matter and heavy metals (Chai et al., 2021b).

The use of shells for water treatment is a promising method, characterized by a number of significant advantages. This approach is particularly applicable in developing nations, where resources are often limited. In India, for example, coconut shells have been successfully used to treat wastewater in villages and small towns In China, oyster shells have been put to good use in the treatment of industrial effluents (Sun et al., 2022), while in Africa, crab shells have found use in the treatment of wastewater from hospitals and schools.

In contrast to previous examples, in Morocco, the shampoo, cosmetics, pharmaceutical, and food industries throw away thousands of tonnes of eggshells every year. Global egg production in 2021 reached 77.4 million tonnes, or around 1.9 billion eggs produced daily (Su et al., 2019). The presence of eggshells in nature can have a negative impact on the environment. Eggshells are composed of calcium carbonate, which degrades slowly in nature. This means they can remain intact for a long time, clogging up natural areas. In addition, eggshell degradation can alter soil pH, making it more alkaline. This can have negative effects on plant growth and the health of soil micro-organisms. Finally, broken eggshells can attract opportunistic animals, such as rodents, insects or birds. This can disrupt the local ecosystem and have an impact on other species (Yang et al., 2022).

Eggshells were chosen as ecological lime source due to their high calcium carbonate ($CaCO_3$) content (around 94%) and low organic matter content (around 6%, mainly proteins) (Quina et al., 2017). Additionally, eggshells possess a porous structure and organic compounds, such as proteins, that enhance their adsorption properties by improving pore filling and complex interactions with adsorbates. Numerous studies have demonstrated the effectiveness of eggshells as adsorbents for removing organic and inorganic pollutants from various sources, including water, wastewater, soil, and gas emissions (Hsu et al., 2023).

Lime, as an economical natural substance, has a proven track record in removing various pollutants. In this study, we will focus on its properties for the removal of organic and inorganic matter, including heavy metals, in wastewater treatment. Adding lime to wastewater to neutralize its acidity promotes bacterial growth, which in turn contributes to the decomposition of organic matter. In addition, lime has the ability to coagulate suspended organic particles, facilitating their elimination (Tsai et al., 2006). We will also explore its coagulation and retention potential for eliminating coloration from polluted waters. In addition, lime can form complexes with heavy metals, reducing their solubility and simplifying their elimination. This eco-friendly biochar was selected to treat wastewater from the El Jadida wastewater treatment plant (WWTP) and improve its physicochemical parameters. It is important to note that this biochar has the dual benefit of recovering waste to protect the environment and minimizing heavy metal contamination. The parameters analyzed in this study were COD, BOD5, pH, conductivity, dissolved oxygen, SS, phosphate ions, nitrogen, and heavy metals. To understand the behavior of the ecological lime and evaluate its efficiency, several physicochemical techniques were used, including FT-IR, SEM, EDX, XRD, and ICP-OES.

MATERIALS AND METHODS

Description of the study zone

El Jadida is a coastal city in Morocco, with an estimated population of 199 934, an area of 3357.85 km², and a density of 195 inhabitants/km². It is located on the Atlantic coast and benefits from a temperate climate. The temperature varies between 17 °C and 30 °C, with humidity of up to 90%. The companies located in its industrial zone cover several types of activities of an industrial nature or services related to the industry (food processing, textiles and leather and para-chemistry, etc.). The current industrial zone, created in 1976, covers an area of 117 ha and includes 90 industrial units (Johari et al., 2022).

The pretreatment plant of El Jadida city (Figure 1), addressed in this study, is located in this new area and covers an area of 2 ha, a perimeter of 637.7 m, and an average altitude of 5 m. It is expected to serve 300 000 inhabitants by 2030. The pretreatment plant also covers the wastewater from the industrial area spread at the southern end of the city. It has a treatment capacity of 95 000 PE. The El Jadida WWTP is intended for the pre-treatment of the city's wastewater. The raw water undergoes screening, de-oiling, and desilting operations and then is discharged into the ocean via a 2,086 linear meter marine outfall.

The sampling points retained for the monitoring of the purification performances are:
- WWTP input with a composite sampling mode.
- WWTP output with a composite sampling mode.

Sampling

The raw wastewater samples studied were taken from the main sewer upstream and downstream

Figure 1. Wastewater treatment plant of El Jadida city

of the WWTP, and were taken throughout 2022, in order to characterize the physicochemical parameters, and then determine the month with the most polluted water to be treated with our bio-adsorbent. Subsequently, the samples collected were stored at 4°C in aseptic containers, in accordance with NF EN 25667-1 ISO 5667, protected from light to avoid any possible physicochemical modification (Guvenc et al., 2016).

Adsorbent material

Collection medium

Chicken eggshells were collected from restaurants and bakeries in the city of El Jadida. This biomaterial was collected during their daily use and transported to the laboratory in a sealed plastic bag to be used as a bio-absorbent.

Preparation of the bio-adsorbent

The eggshells are cleaned with demineralized water several times to remove all dirt particles. After removing the eggshell membrane, the eggshells were dried in an oven maintained at 105 °C for 3h, ground manually in a mortar, pulverized in a mechanical grinder into a fine powder, and sieved through a 500 µm sieve. The sample was then calcined in a furnace held at 900 °C for 6h to allow for the total decomposition of the eggshell $CaCO_3$ to CaO (Rohim et al., 2014). Then, the calcined samples were placed in a glass vial, sealed, and labeled appropriately prior to being cooled to room temperature. Finally, the eggshell samples were protected from any contamination (e.g., water, carbon dioxide) and ready for further use.After the adsorption process, the bio-adsorbents were collected for physicochemical characterization. Indeed, they were dried at a temperature of 35°C, and cooled to room temperature before being placed in glass vials, labeled and sealed tightly.

Apparatus

To have a reliable result for COD, a popular method was selected according to the French standard of classification index: T 90-101. For BOD5 we have used the DOB sensor F102B0133. Dissolved oxygen and conductivity were measured with the Multi 3630 from WTW (Germany). Phosphate content was determined using the WTW™ 252076 Combi Check Phosphate $(PO_4)_3$- cell test kits. Nitrogen present is measured using Combi Check Total Nitrogen cell test kits make: WTW™ 251996 (Germany). In addition to the physicochemical parameters determination, X-ray diffraction analysis was also performed to determine the composition of our samples using a BRUKER D8 ADVANCED diffractometer. And the obtained results were supported by FTIR spectroscopy analysis, which were carried out using Nicolet IS10 thermo-fisher spectrometer. Furthermore absorbance measurements were performed using JASCO V- 630 UV/Visible spectrophotometer. Ecological lime analysis was carried out using a scanning electron microscope (SEM) (QUATTRO S-FEG-Thermofisher scientific) to analyze the microstructural morphology of the prepared sample. An energy dispersive X-ray spectrometer (EDX) was coupled to the SEM for elemental analysis of the sample. SEM micrographs are used to analyze the apparent structure of the sample. To finalize these analysis, the retention efficiency of heavy metals on the bio-adsorbent surface were evaluated by using Shimadzu E-9000 ICP-OES. All physicochemical measurements were conducted at room temperature.

RESULTS AND DISCUSSION

Characterization of the discharge before and after treatment

Physicochemical parameters

Table 1 shows the different physicochemical parameters (T, pH, COD, etc.) measured in September 2021. As a result, it can be seen that the pH increased significantly from 7.2 at the WWTP outlet to 11 after the decolorization treatment. This behavior is perhaps attributed to the alkalinity nature of our material containing mainly quicklime. The treatment process showed no effect on both temperature and dissolved oxygen parameters. In contrast, the conductivity of the medium decreased dramatically, which may be due to the fact that the bio-adsorbent used was powerful in removing salinity.

The biodegradability also decreased, which was explained by the decrease in COD and BOD_5 values. Indeed, this decrease could be attributed to the retention of organic compounds at the bio-adsorbent surface and to the consumption of oxygen by the existing bacteria in the water.

X-ray diffraction analysis

X-ray diffraction (XRD) was used to characterize the ecological lime, before and after treatment. This technique enabled us to obtain valuable information on the material's crystalline structure and to identify the different phases present in the two samples. The two XRD diagrams shown in Figure 2a reveal the predominance of two main crystalline minerals: $Ca(OH)_2$ (CaH - portlandite, JCPDS-72-0156), $CaCO_3$ (calcite, JCPDS-47-1743). This predominance is linked to a hydrated version of CaO, which probably results from the adsorption of wastewater. Indeed, the graph shows the partial formation of m-$CaCO_3$ (vaterite) at a temperature of 900°C, which remains stable after cooling to room temperature (Bhat et al., 2022b).

Infrared spectroscopy

As illustrated in Figure 2b, the infrared spectra show the existence of OH in $Ca(OH)_2$ in the peak of about 3642 cm^{-1} (Guvenc et al., 2016). Moreover, two peaks were observed at around 857 cm^{-1} and 874 cm^{-1}, respectively, which were associated to the presence of calcium carbonate. The absorption bands from 1454 cm^{-1} to 3642 cm^{-1} and from 1431 cm^{-1} to 3642 cm^{-1} represent the stretching vibration of CO_3^{2-}, present in the eggshell. The infrared results show that $CaCO_3$ has completely converted to CaO, with the Ca-O bond existing in the calcined eggshell.

Mass effect of the organic load

Adsorption tests were performed in a batch reactor. In Erlenmeyer flasks containing 100 ml of rejection, we introduced increasing masses (0.3 g, 0.6 g, 0.9 g, 1.2 g, 1.5 g, 1.8 g) of the prepared bio-adsorbent under stirring (500 rpm) for 2 H. After the samples taken of the solution exposed to UV radiation, they were filtered on Millipore membrane type 0.45 µm. Subsequently, absorbance measurements were recorded to determine the effects of discoloration on the absorbance of water before and after treatment with the different modes. The results obtained are illustrated on the curve of Figure 3 which shows an abatement of the discoloration in parallel with the addition of the mass of which 1.5 g is the optimal mass with an abatement of 87% of the discoloration.

Table 1. Physicochemical parameters values measured in September 2022 before and after treatment with WWTP

Parameter	Input	Output	After treatment
pH	7.5	7.2	11
T (C°)	17.7	17.4	17
O_2 ($mg.L^{-1}$)	0.01	0	0
Conductivity ($\mu S.cm^{-1}$)	5030	4420	20
COD ($mg.L^{-1}$)	915	816	223
BOD_5 ($mg.L^{-1}$)	700	664	200
COD/BOD_5	1.3	1.2	1.11
NTK ($mg.L^{-1}$)	90	77	37
T.P ($mg.L^{-1}$)	42.1	36.7	0.7
TSS ($mg.L^{-1}$)	1262	1003	500

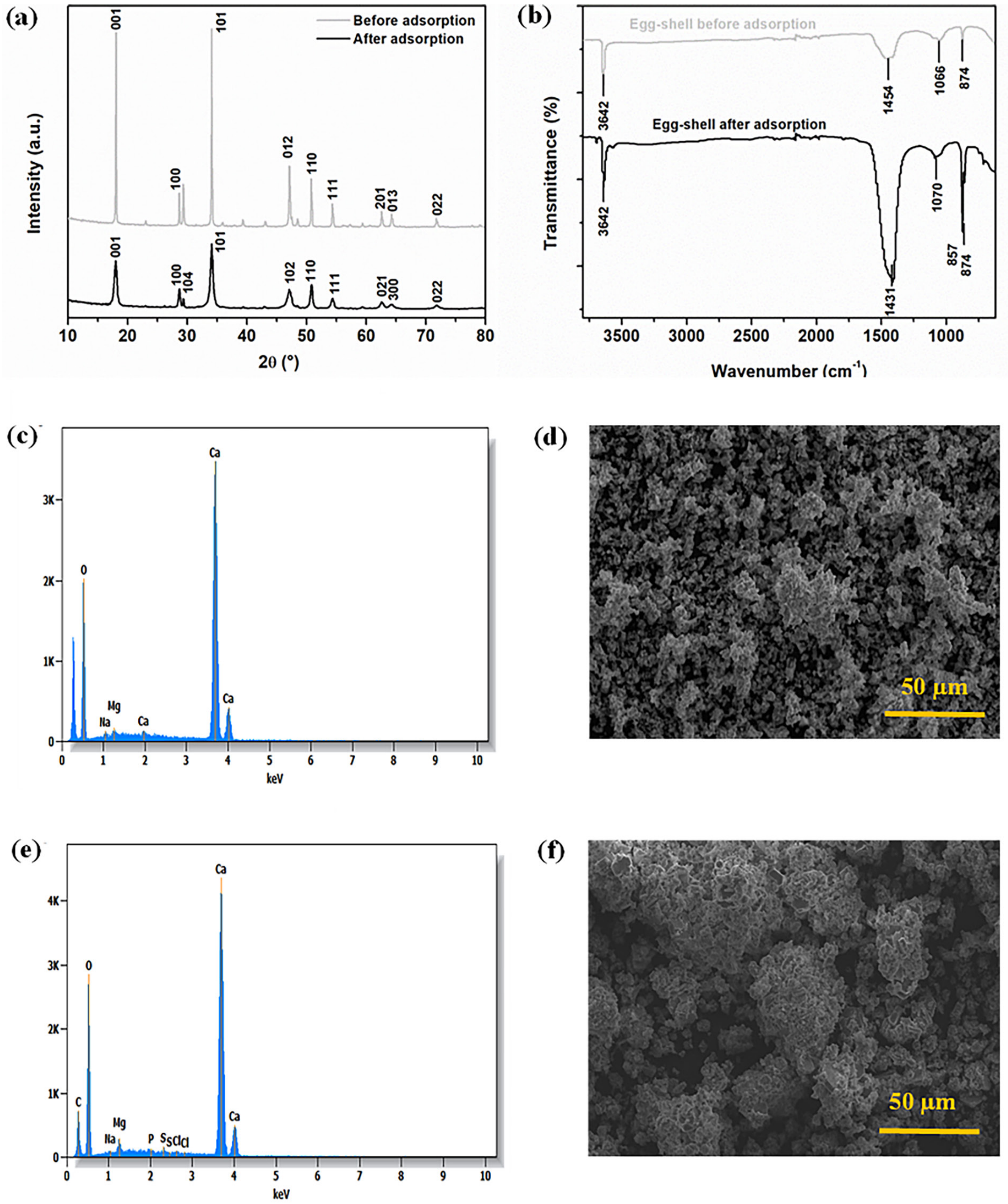

Figure 2. The XRD pattern (a) of eggshell powder calcined at 900°C before and after treatment, enabling us to characterize our bio-adsorbent, in order to monitor the retraction or disappearance of chemical compounds and (b) the FTIR spectra of calcined eggshells at 900°C before and after adsorption, in which it can be seen that the transmittance underwent a significant drop after treatment. In (c) and (d) the SEM and EDX of calcined eggshells before and after adsorption in (e) and (f), showing the appearance of chemical components after treatment. these components are retained by the bio-adsorbent ports

Scanning electron microscopy

Scanning microscopy analysis was used to assess the surface condition of the bioadsorbent before and after adsorption. As a result, the porosity of the bioadsorbent before adsorption was significant Figure 2d. In contrast, Figure 2f shows that after adsorption, porosity decreased, which may be due to pollutant retention. In addition, prior to the adoption process, micrographs clearly indicate the development of irregular, uneven surfaces with well-defined, vacant channels and pores. EDX analysis showed a significant amount of calcium and oxygen present in the bio-adsorbent before Figure 2c or after adsorption Figure 2e, where the weight and atomic percentage are presented in Table 2. Indeed, the EDX result shows 98% oxygen in the form of lime (CaO). However, the EDX shows traces of magnesium oxide (MgO)

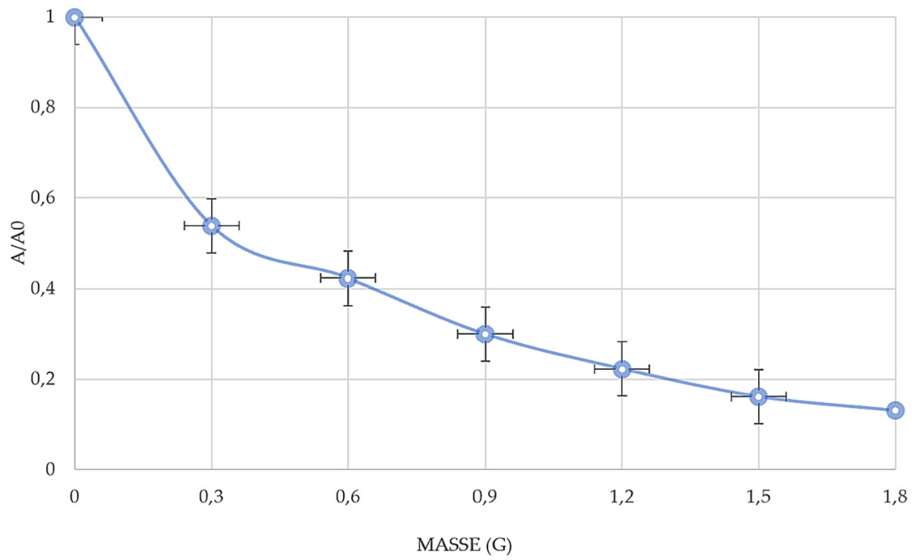

Fig. 3. Mass effect on absorbance compared to raw wastewater

Table 2. Components and chemical composition of prepared lime before and after treatement

Element	Before treatment (wt%)	After treatment (wt%)
C	-	3.6
O	28.6	34.7
Na	0.3	0.0
Mg	0.6	0.9
P	-	0.2
S	-	0.2
Cl	-	0.2
Ca	70.5	60.1
Total	100.0	100.0

and sodium oxide. As illustrated in Table 2, the EDX analysis showed the presence of 13.2% CO_2 after treatment, while this analysis remains semi-quantitative. The Bernard Calcimeter enabled us to accurately determine the amount of CO_2, which amounted to 44%. On the other hand, EDX showed traces of magnesium oxide (MgO), sodium oxide (Na_2O) and phosphorus pentoxide (P_2O_5) on Table 2. After the adoption of wastewater loaded with organic matter and colloidal particles, surface roughness and porosity were reduced, justifying the abatement of discoloration and heavy metals.

Heavy metals retention

Table 3 shows the different values of heavy metal concentrations, which are required by the discharge standard (Abubakar and Usman, 2021). The results obtained showed that a total removal of Co, Be, Ag, Cu, was achieved using eggshell bio-adsorbent. Whereas, a significant reduction of Cd, Pb, Cr, Tin, Mn, Al and Zn. Moreover, this treatment allowed the regulation of Zn in order to be acceptable with respect to the Moroccan standard of water rejections. However, Cd, As and V still require additional treatment to meet water discharge and irrigation requirements (0.25 $mg \cdot L^{-1}$ Cd, 0.1 $mg \cdot L^{-1}$ As, and 0.1 $mg \cdot L^{-1}$ V).

CONCLUSIONS

First of all, the Performance monitoring of the EL JADIDA wastewater treatment plant was carried out to determine the degree of physicochemical pollution of the raw water and to assess the efficiency of the pre-treatment, it was demonstrated

Table 3. Heavy metal concentrations before and after treatment

Metal	Concentration ($mg.L^{-1}$)		% Reduction
	Before	After	
Cd	17.11	8.02	53.1
As	5.66	4.05	28.4
Cr	0.87	0.32	63.2
Pb	0.14	0.07	50
Cu	0.02	0	100
Zn	12.80	2.56	80
Se	ND	ND	ND
Ag	0.11	0	100
Al	3.61	0.77	78.7
Be	0.02	0	100
Co	0.02	0	100
Mn	0.18	0.01	94.4
V	9.39	6.30	32.9
Ni	0.15	0.10	33.3

Note: ND – not detected.

that the wastewater contaminated by various micro-organic and micromineral toxic elements. The present work has demonstrated that ecological lime prepared from eggshells waste is effective to regulate physicochemical parameters, by removing 73%, 69%, 50% and 84% of COD, BOD_5, TSS and discoloration, respectively. Indeed, the present work has demonstrated that eggshells can successfully replace commercially available and non-renewable adsorbents for the effective removal of large quantities of toxic heavy metals and ensures perfect retention. The survival of physicochemical parameters as well as the retention of heavy metals in treated wastewater are essential to provide wastewater that can be used for irrigation in green areas. Nevertheless, further studies are still needed to ensure that the treatment of this water meets a wide range of applications, such as the use of treated water in agricultural irrigation and industrial cooling.

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