

Treatment of contaminated well water with coagulants of different Opuntia *ficus-indica* L. populations

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Abstract

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1. INTRODUCTION

Water is a natural resource essential to the life of all living beings. Global freshwater use has grown by a factor of six over the 100 years past and continues to increase at a rate of about 1% per year since the 1980s (United Nations, 2021). It is estimated that if unsustainable pressures on global water resources continue, 52% of the world's population, 45% of global gross domestic product, and 40% of global grain production will be at risk by 2050 (WWAP, 2019). The global water situation is also marked by the problem of water pollution which has worsened in Africa, Latin America, and Asia since the 1990s (WWDR, 2018). Tunisia also faces a delicate water situation where water is increasingly scarce and less and less renewable

Opuntia ficus-indica (OFI) is a high added value species which different organs are used in several fields, including water purification. The present study intended to treat contaminated well water using naturally prepared coagulant from the cladodes of four local OFI populations in Tunisia. Physicochemical characterization of the water was carried out before and after treatment. The treatment applied allowed to decontaminate the well water studied. Most of the parameters tested were significantly influenced by the "population used" factor. The treatment by the "Amless" population of OFI resulted in the best reduction rates of the contents of Mg, Na, Cu, and Pb. "Loki" population allowed obtaining the best pH and EC reductions. The "Violet" population resulted in the highest COD, BOD₅, and TSS reductions. The effectiveness and simplicity of this treatment method would encourage farmers to use it to improve the quality of the well water that is used for irrigation.

> (Chahed et al., 2010). The deterioration of water quality will increase over the next decades, which would increase the threats to humans, the environment, and sustainable development (Boretti and Lorenzo, 2019). Contaminated water should not be consumed directly due to the presence of toxic contaminants. Globally diseases related to water pollution are responsible for the death of more than 5 million people annually (Malik et al., 2012). The major problem of water scarcity has piqued the interest of researchers around the world to discover new efficient and cost-effective means and techniques for the treatment of contaminated water (Marston et al., 2021). Water purification is a set of techniques that purify water for the transformation of natural

water into drinking water. Conventional metal

methods (chemical precipitation, removal activated carbon adsorption, ion exchange, evaporation, and membrane processes) are becoming either inadequate to meet the current strict regulatory efficiency limits or very expensive (Rajasulochana and Preethy, 2016). While purification techniques using biological materials such as microorganisms or plants are increasingly used and are now becoming an interesting and profitable alternative (Sharma, 2021). These techniques are increasingly used to remove toxic metals from contaminated water due to their increased performance and availability (Ahluwalia and Goval, 2007). They are less expensive to achieve (Alsarayreh et al., 2022), which is very important for the sustainability of these processes in developing countries such as Tunisia. The use of more ecological processes to treat contaminated water is more and more topical because these processes are respectful of the environment and offer many other advantages, such as the reduction of by-products, better biodegradability, and low energy consumption (Villaseñor-Basulto et al., 2018).

The cactus is a tree native to the arid and semiarid regions of Mexico (Tenorio-Escandón et al., 2022). It belongs to the genus *Opuntia*, it is a succulent xerophytic plant capable of storing a large amount of water and does not present any danger to human health (Shetty et al., 2012). As part of this work, we were interested in studying the purifying performance of four different populations of *Opuntia ficus-indica* (*OFI*) on contaminated well water and selecting the population with the best purifying performance.

2. MATERIAL AND METHODS

2.1. Plant materials

Samples of four *OFI* populations were collected from farmers of the Nabeul region in January 2020 in hermetically sealed bags under ambient conditions with temperatures varying from 20 to 28°C. The *OFI* populations used in this study are the "Amless" population characterized by large cladodes devoid of spines, the "Loki" population which has small spiny cladodes, the "Chaouky" population having large cladodes and large spines, and the "Violet" population presenting large cladodes without spines (Fig. 1).



Figure 1. Cladodes of the used populations of *Opuntia ficus-indica* L.: (P1) Amless population, (P2) Loki population, (P3) Chaouky population, and (P4) Violet population.

2.2. Water samples

Contaminated well water was collected from a well located at the National Research Institute for Rural Engineering, Water and Forests experimental station in "Oued Souhil", Nabeul governorate (Fig. 2). The well in question is supplied by collecting surface water. The well structure is permeable; rainwater seeps into the ground and then fills the well through its walls. The water is then extracted, via a surface pump. The water samples were placed in hermetic and sterilized plastic bottles, and then transported to the laboratory where they were stored at 4°C.



Fig. 2. Location of well water sampling in Tunisia (source: ArcGIS 10.3).

2.3. Coagulant preparation

The cladodes were rinsed with tap water followed by distilled water. They were stripped of thorns and external envelopes. The mucilage was cut before being dried at 60°C in the oven for 24 hours. The dried cladodes were finally ground to a fine powder which was placed in a sterile vial and stored at 4°C in order to be used as a coagulant for contaminated well water (Deshmukh and Hedaoo, 2018).

2.4. Water treatment

The water samples were thoroughly mixed with 70 mg·L⁻¹ of coagulant of each *OFI* population (Deshmukh and Hedaoo, 2018), and agitated at high speed (1000 rpm) for coagulation; the speed was then reduced (800 rpm) for flocculation. The water samples were then decanted for 2 h, and finally, the supernatant forming the purified water was collected.

2.5. Water samples characterization

The physicochemical characterization of the contaminated well water samples, before and after treatment, was based on the parameters given in Table 1.

2.6. Statistical analysis

Statistical analyses were performed based on the independent factor "OFI population", which were used to explain variations in the dependent quantitative variables: chemical oxygen demand (COD), five-day biochemical oxygen demand (BOD₅), total suspended solids (TSS), electrical conductivity (EC), mean hvdrogen ion concentration (pH), Turbidity, and concentrations of chloride (Cl), bicarbonate (HCO_3) , calcium (Ca), magnesium (Mg), potassium (K), sodium (Na), cadmium (Cd), lead (Pb), copper (Cu) and iron (Fe). The normality of the quantitative variables was assessed using the Shapiro-Wilk test. An analysis of variance (ANOVA) was performed using SPSS software

Table 1. The studied parameters and analysis methods.

(IBM SPSS statistics, version 20), and this was complemented by a Duncan's test of the OFI populations concentration groups to compare their means using a pairwise comparison. Values of p < 0.05 were considered to be statistically significant. Principal component analysis (PCA) performed using XLSTAT software was (Adinosoft, version 2014.5.03), based on the removal efficiency (H %) of the physicochemical parameters of the well water that was treated by the OFI coagulant (Marzougui et al., 2021). The H value was calculated according to the formula:

$$\% (H) = \left[\frac{Ci - Cf}{Ci}\right] \times 100$$

where *Ci* and *Cf* are the initial and final concentrations of corresponding parameters (Nhut et al., 2020).

3. RESULTS AND DISCUSSION

3.1. pH change

The pH is a parameter that greatly affects coagulation. Depending on the characteristics of the water, there is an optimal pH, which leads to the best coagulation when the solubility of the coagulant is minimal and gives better precipitation (Sahu and Chaudhar, 2013). The results showed that the pH values of the well water samples tested were in the pH normal range limited for irrigation water (FAO, 1985). Treatment with the different *OFI* populations did not obviously change the pH values of the well water compared to the control (Table 2); the variation of this parameter was non-significant depending on the population used (p=0.1). Control samples showed an average pH value of 7.77. After treatment with the different OFI

Parameter	Methodology	Unit	Source
рН	HANAA 2210 pH meter (U. K.)	-	NF T 90-008, 2001
EC	WTW LF 330 conductometer (Germany)	(dS⋅m ⁻¹)	Rodier et al., 2016
Turbidity	Spectrophotometer HACH DR/4000 (U.S.A.)	NTU	Rodier et al., 2016
COD	Method by oxidation with KMnO ₄	(mg O ₂ ·L ⁻¹)	Baird et al., 2017
BOD ₅	Instrumental method	$(mg 0_2 \cdot L^{-1})$	Baird et al., 2017
TSS	Filtration on filter paper	(mg·L ⁻¹)	Rodier et al., 2016
Na, K	Spectrometric method (flame photometer Jenway PFP7, U.K.)	(mg·L ⁻¹)	Rodier et al., 2016
Cl	Mohr method	(mg·L ⁻¹)	Belcher et al., 1957
HCO ₃	Volumetric titration	(mg·L ⁻¹)	Rodier et al., 2016
Ca, Mg	Complexometric EDTA (Chem-Lab) titration with basic medium	(mg·L ⁻¹)	Ringbom et al., 1958
MTE: Cd, Pb, Cu, Fe	Atomic absorption spectroscopy with PerkinElmer PinAAcle 900 T, USA.	(mg·L ⁻¹)	Sleimi et al., 2021

(pH: Mean hydrogen ion concentration, EC: conductivity, COD: Chemical oxygen demand, BOD₅: Five-days biochemical oxygen demand, TSS: Total suspended solids, Na: Sodium, K: potassium, Cl: Chloride, HCO₃: Bicarbonate, Ca: Calcium, Mg: magnesium, MTE: Metal trace elements, Cd: Cadmium, Cu: Copper, Pb: lead, Fe: Iron).

populations, the average pH value reached 7.63. "Loki" population allowed obtaining the lowest pH value (7.1). These results are consistent with the work of Mukhar et al. (2015) on the effect of Opuntia stricta coagulant on surface waters. They noted that the water pH remains constant during coagulation and pH adjustment was subsequent unnecessary for treatment processes, which is often needed when metal coagulants, such as sulfate ammonium, are used. However, Miller et al. (2008) state that there is a relationship between pH and coagulation activity of *Opuntia* spp. They found that species of this genus operated with over 98% removal of turbidity from pH 8–10. Similarly, Zhang et al. (2006) have reported that Opuntia spp. is greater effective at pH 10 and is lowest effective at pH 6.

3.2. EC change

The EC reflects the degree of global

mineralization of provides water and information on its salinity rate (Belghyti et al., 2009). Table 2 showed that the EC value of control well water studied (3.81 dS·m⁻¹) exceeds the limit indicated in the guidelines for interpretation of water quality for irrigation, showing that it is water with severe salinity (FAO, 1985). EC variations were significant according to the population used (p < 0.05). The value of this parameter increased slightly compared to the control following treatment with the "Amless", "Chaouky" and "Violet" populations. The percentage increases were 0.34%, 2.04% and 1.62%, respectively (Table 2). These increased percentages of the EC are clearly lower than those recorded by Gandiwa et al. (2020) when they tested the effectiveness of OFI extracts coagulants in the treatment of raw wastewater. They recorded a 36% increase of EC as the OFI coagulant dosage increased from 0 to 60 mg·L⁻¹. The increase in EC is a result of the

Table 2. Physicochemical characterization of well water before and after treatment with four varieties of *Opuntia ficus-indica* L.

Parameter	Before treatment	After treatment				Restriction	References
		Amless	Loki	Chaouky	Violet		
рН	7.77 ±0.07 ^a	7.82 ±0.01 ^b	7.10 ±2.2 ^b	7.81 ±0.01 ^b	7.81 ±0.005b	6.5-8.4	FAO (1994)
EC (dS⋅m ⁻¹)	3.81 ±0.01ª	3.85 ± 0.01^{ab}	3.5 ±1.1 ^{ab}	3.91 ±0.02¢	3.9 ±0.02 ^{bc}	0.7-3	FAO (1994)
Turbidity (NTU)	2.6 ± 0.01^{a}	6 ± 1.5 ^b	5.5 ± 3.2°	$10.8 \pm 0.9^{\circ}$	8.6± 1.5°	-	-
COD (mgO ₂ ·L ⁻¹)	697.4 ±77¢	667.2 ±101 ^d	676.4 ±16.9ª	589.8 ±62.8e	533.4 ±57 ^b	90	INNORPI (1989)
BOD ₅ (mgO ₂ ·L ⁻¹)	50.33 ±1.5ª	19 ±1 ^b	20 ±1 ^b	6.73 ±0.25¢	2.46 ±0.35ª	30	INNORPI (1989)
TSS (mg·L ⁻¹)	96.6 ±0.19 ^a	79.5 ±0.27 ^a	80.2 ± 0.25^{a}	72.3 ±0.32 ^a	69.6 ± 0.14^{a}	30	INNORPI (1989)
Cl (mg·L ⁻¹)	779.75 ±58 ^a	766.4 ±14.4 ^a	731.42 ±76 ^a	804.74 ±52.9ª	721.44 ±5.7 ^a	141.81-354.53	FAO (1994)
HCO ₃ (mg·L ⁻¹)	446.76 ±1.9ª	454.9 ±0.06ª	459.75 ±11ª	458.25 ±27.6 ^a	461.04 ±1.7 ^a	91.52-518.65	FAO (1994)
K (mg·L ⁻¹)	140.21 ±0.7ª	138.21 ±0.4 ^b	150.5 ±9.2 ^b	147.54 ±2.08 ^b	146.5 ±2.3 ^b	-	-
Ca (mg·L ⁻¹)	114.29 ±0.23ª	55.46 ±0.04 ^b	76.85 ±17.5 ^b	61.15 ±1ª	60.81 ±12.7ª	-	-
Mg (mg·L ⁻¹)	2.8 ± 0.08^{d}	1.9 ± 0.19^{a}	3.56 ± 0.85^{ab}	3.40 ±0.43°	3.29 ±1.3 ^{bc}	-	-
Na (mg·L ⁻¹)	406.23 ±7.07ª	376.9 ±1.7ª	385.3 ±7.2ª	381.9 ±6.6ª	391.9 ±13.2ª	-	-
SAR	2.32	3.07	2.65	2.93	3.02	3-9	FAO (1994)
Cd (mg·L ⁻¹)	0	0	0	0	0	0.01	FAO (1994)
Fe (mg·L ⁻¹)	0.71 ± 0.006^{a}	0.58 ± 0.2^{a}	0.52 ±0.01 ^b	0.67 ±0.01¢	0.59 ±0.21ª	5.00	FAO (1994)
Pb (mg·L ^{−1})	0.47 ± 0.05^{a}	0.39 ±0.02ª	0.34 ±0.02 ^b	0.41 ± 0.05^{ab}	0.38 ± 0.06^{a}	5.00	FAO (1994)
Cu (mg·L ⁻¹)	0.104 ± 0.005^{ab}	0.082 ± 0.003^{a}	0.074 ±0.007¢	0.092 ±0.001 ^b	0.082 ±0.03 ^b	0.20	FAO (1994)

Values are mean \pm standard deviation (n = 3); The different letters within the same row indicate a difference between the treatments at p < 0.05 level (Duncan's test).

coagulant dissociation, which produces ions that raise EC. Since raw wastewater is often loaded with pollutants than surface water (Edokpayi et al., 2017), the dissociation of the coagulant will be higher there.

3.3. Influence of the treatment on turbidity

Turbidity is a physical phenomenon inversely proportional to water transparency (Bin Omar and Bin MatJafri, 2009). It is the main pollution parameter indicating the presence of suspended solids (organic or mineral) in the water and varies according to their presence (Metahri, 2012). The results showed significant variations of well water samples turbidity according to the population used (p < 0.05). The values of this parameter tend to rise: the average value recorded in the control is 2.66 NTU, it increased to reach a maximum of 10.3 NTU (an increase of 75.38%) following treatment with the "Chaouky" population. Contrary, Zhang et al. (2006) found that the coagulant prepared from the cactus cladode is very effective in reducing turbidity in conventional waters. The turbidity increase of the treated samples can be explained by the phenomenon of system restabilization when there is an excess of added coagulant, this effect can disappear by increasing the settling time (Dihang, 2007). Miller et al. (2008) studied the effect of *Opuntia* spp. cladode on synthetic water turbidity. They found that *Opuntia* spp. operates essentially through a bridging coagulation mechanism. The cladode mucilage may be contributing to the observed coagulation behavior, specifically its galacturonic acid component, which may explain some of the turbidity reduction by *Opuntia* spp. by reducing turbidity by more than 50%.

3.4. Ion-forming salts removal

The results showed that the variations in Ca^{2+} , Mg²⁺, and K⁺ concentrations were significant based on the population used (p < 0.05). On the other hand, the concentrations of Na⁺, Cl⁻ and HCO₃showed non-significant variations according to this same factor (p>0.05). Table 2 after treatment. showed that Mg²⁺ concentrations increased by 2.8 mg·L⁻¹ in the control to reach a maximum of 3.56 mg·L⁻¹ with the "Loki" population (47.36% increase). In contrast, Ca²⁺ levels decreased after treatment from 114.2 mg·L⁻¹ in the control to 55.46 mg·L⁻¹ after treatment with "Amless" population (106.07%) decrease). The Na⁺ ion levels decreased slightly from 406.2 mg·L⁻¹ in the

control, to 376.9 mg·L⁻¹ following treatment with "Amless" population (7.77% decrease). By determining the Sodium Adsorption Ratio (SAR) which assesses water sodicity and the potential for infiltration problems due to a sodium imbalance in irrigation water (Bauder et al., 2014), it turns out that the control well water was within the standards indicated by the FAO for irrigation water quality (FAO, 1994), it is also even after the treatment by the different OFI populations (Table 2). The contents of K⁺ ions decreased slightly in the samples treated with "Amless" population compared to the control (1.44% decrease); whereas by treating the contaminated well water with the other populations, slight increases in the contents of this cation were noted, a maximum of 6.86% increase was recorded after treatment with "Loki" population. About the Cl- ions, the contents slightly decreased following the treatment with OFI coagulant above all the "Violet" population which allowed obtaining a decreasing percentage of 8%. These results are consistent with the work of Cherif (2012) for who the treatment with the OFI coagulant made it possible to reduce the levels of chloride ions present in brackish water and partially neutralize their negative charges by the cations of the OFI coagulant. However, the well water tested does not meet the standards for irrigation water (FAO, 1994) concerning to the Cl concentration and the treatment by the different OFI populations did not allow circumventing this problem (Table 2). For the HCO_3^- contents, the results showed that they increased slightly following treatment with four the the populations tested, the maximum concentration of 461.04 mg·L⁻¹ was obtained with the "Violet" population while that of the control is 446.76 $mg \cdot L^{-1}$ (3% increase). Starting with the fact that *Opuntia* spp. operates essentially through a bridging coagulation mechanism, Miller et al. (2008) considered that natural electrolytes present in the cladodes, particularly the divalent cations such as Ca2+ and Mg2+, which are important for coagulation with anionic polymers, facilitate adsorption.

3.5. Influence of the treatment on COD, BOD₅, and TSS

Table 2 showed that TSS values tend to decrease following the treatment with the coagulants of the 4 *OFI* studied populations. However, the variations of this parameter turn out to be non-significant according to the "population used" factor (p=0.31). The average value decreases by

96.25 mg·L⁻¹ in the control to reach 69.6 mg·L⁻¹ (38.24% decrease) thanks to the treatment with the "Violet" population while remaining above the recommended TSS value (30 mg·L⁻¹) for the agricultural reuse of treated wastewater, according to the Tunisian standards NT 106.03 (INNORPI, 1989). Contrarily, the COD of the tested well water samples recorded significant variations based on the "population used" (p<0.05). Its value fell from 697.4 mgO₂·L⁻¹ in the control to 533.4 mgO₂·L⁻¹ in the sample treated with the "violet" population (30.74% decrease). These reductions were not sufficient for the well water considered to meet the irrigation water quality Tunisian standards with respect to COD (INNORPI, 1989). Whereas, OFI treatment allowed to obtain BOD₅ values complied with the standards as showed in Table 2 (p < 0.05). They spectacularly decreased in the treated well water samples compared to the control (50.3 mgO₂·L⁻¹). The maximum decrease of BOD₅ value (2.46) $mgO_2 \cdot L^{-1}$) was obtained following treatment with the "violet" population (1940% decrease). Sethu et al. (2019) showed when they treated palm oil mill effluent with Opuntia coagulant reductions of 91.2% in COD and 94.4% in TSS. They reported that the coagulation mechanism for the removal of the contaminants may involve, in addition to the natural polyelectrolytes and the galacturonic acid localized in the mucilage, complex carbohydrates within the cladodes such as L-arabinose, Dgalactose, L-rhamnose, D-xylose. These last are considered as possible active ingredients supporting the coagulation capability of *Opuntia* spp.

3.6. Removal of Metallic Trace Element (MTE)

For the MTE, it was noted that the well water tested was compliant with the irrigation water quality standards before and after the treatments with the coagulants of the four OFI populations (FAO, 1994). The Pb, Fe, and Cu contents in the contaminated well water tested tend to drop after the treatments (Table 2). The Pb, Fe, and Cu contents showed significant variations according to the "population used" factor (p < 0.05). On the other hand, no traces of Cd were detected in well water before and after the treatments were carried out. The average values recorded for Pb, Fe, and Cu decreased by 0.47 mg·L⁻¹ in the control to reach the minimum value of 0.34 mg·L⁻¹ (40.49% decrease), by 0.71 $mg\cdot L^{-1}$ in the control to 0. 52 $mg\cdot L^{-1}$ (35.25%) decrease), and from 0.104 mg·L⁻¹ to 0.07 mg·L⁻¹

(40.54% decrease), respectively after treatment with "Loki" population. Vargas-Solano et al. (2022) confirmed the MTE removal efficiency of OFI mucilage during their river water decontamination trials. They recorded a removal capacity of the OFI mucilage greater than 90% for Fe and less than 40% for Cd, and lead Pb. By identifying the functional groups of the OFI mucilage by Fourier transform infrared spectroscopy (FTIR) before and after the removal process, these authors stated that the acting groups in the removal of the heavy metals were the carbonyl, carboxyl, and hydroxyl groups.

3.7. Structuring of *Opuntia ficus-indica* populations

A PCA, based on the removal efficiencies of the parameters that we considered here, was established to analyze the similarities between and the variations of the treatment efficiency. The first two PCAs described 89.47% of the total variability (Fig. 3). PCA1 (51.34% of the total variation) was positively correlated with Mg, Na, Cu, and Pb contents and was negatively correlated with Cl content, TSS, COD, and BOD₅. PCA2 (38.13% of total variation) was positively correlated with EC and pH and was negatively correlated with Ca content.

Biplot (PCAs 1 and 2 : 89.47 %)



Fig. 3. Identification of the groups of contaminated well water treated with four *Opuntia ficus-indica* populations and drawn up by the PCA based on the removal efficiencies of the parameters analyzed: (P1) Amless population, (P2) Loki population, (P3) Chaouky population, and (P4) Violet population.

The first two PCAs allowed structuring the well water samples into four groups:

• The first group was represented by the positive aspects of PCA1 and the negative aspects of PCA2. It was formed by the well water samples treated with "Amless" population. This treatment resulted in the highest removal efficiencies of Mg, Na, Cu, and Pb contents.

• The second group had the negative aspects of PCA1 and the positive aspects of PCA2. It constituted the well water samples treated with "Loki" population which had the highest removal efficiencies of pH and EC.

• The third group, located near the intersection of PCA1 and PCA2, presented the well water samples that had been treated by "Chaouky" population. This treatment did not exhibit considerable removals for all the parameters analyzed.

• The fourth group, with the negative aspects of PCA1 and PCA2, included the well water samples treated by "Violet" population. It was characterized by the highest TSS, COD, and BOD₅ removal efficiencies.

PCA1 and PCA2 show that the "Amless" and "Violet" populations have presented satisfactory results in treating contaminated well water. It can therefore be used as an alternative for the chemical agents such as aluminum sulfate that are commonly used in the treatment of conventional water (Young et al., 2006), because of its low cost in treatment and handling, low amount of residual sludge generation, inherently renewable character, and low toxicity (Dai et al., 2018; Joseph et al., 2019). The effectiveness and simplicity of this treatment method of the OFItreated water would encourage farmers to use it to improve the quality of the well water that is used for irrigation.

4. CONCLUSIONS

This work was carried out with the aim of evaluating the purifying power of contaminated well water by the powder of the cladodes of four different OFI populations. The treatment was carried out with a concentration of 70 mg·L⁻¹ of coagulant by adopting coagulation at 1000 rpm for 3 min, followed by flocculation at 800 rpm for 20 min, and finally decantation for 2 hours. The treatment applied allowed to decontaminate the well water. The majority of parameters tested (62.5% of these parameters) were significantly influenced by the "population used" factor. The PCA showed that the treatment by the "Amless" population of OFI resulted in the best reduction rates of the contents of Mg, Na, Cu, and Pb. "Loki" population allowed to obtain the best reductions for pH and EC. "Chaouky" population did not allow significant reductions to be recorded and the "Violet" population resulted in the highest reductions for the COD, BOD₅, and TSS parameters. Further investigations will be conducted about the effect of the mixture of the four *OFI* populations tested on contaminated well water.

Tunisia is marked by serious problems affecting the water quality and availability in addition to the energy shortage. Developing alternative and low-cost treatment methods is therefore of great importance.

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