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# Optimisation of a hybrid wastewater treatment via full factorial design and its preliminary physicochemical and short-term agronomic assessment for irrigation reuse

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## ABSTRACT

This study develops an economical hybrid treatment process for removing organic compounds from wastewater, along with a preliminary assessment of the treated effluent for potential agricultural reuse. The research first investigated the decontamination of water from phenol, gallic acid, and humic substances through two methods: adsorption onto activated acorn bark and coagulation-flocculation using aluminium sulfate. To maximise treatment efficiency, a hybrid system was developed. Optimal operating conditions were determined using a factorial design methodology. Under optimised parameters conditions, the treatment efficiency achieved a 96.36% rate of organic matter removal. In the context of water scarcity in Algeria, a localised irrigation trial was conducted on green beans to evaluate the physicochemical quality of the treated effluent and its short-term agricultural performance. The preliminary effects of the hybrid process on soil properties and plant growth were evaluated in the short term. Water quality, soil properties, and plant growth were compared across three sources: physicochemically treated wastewater (PCTW), biologically treated wastewater, and conventional water. PCTW was compliant with the relevant international guidelines for agricultural reuse. Furthermore, the irrigation trial demonstrated that PCTW significantly improved soil fertility, while promoting superior vegetative growth compared to conventional water. These findings highlight the potential of this hybrid process to produce treated wastewater with satisfactory physicochemical quality and nutrient content while supporting short-term plant growth under localised irrigation conditions.

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## INTRODUCTION

Water pollution is one of the major environmental problems associated with rapid economic development. Wastewater often contains hazardous organic pollutants, particularly phenolic compounds (Haripriyan et al., 2022). These compounds are characterised by high stability and toxicity, resulting in their classification among the most harmful contaminants by the Environmental Protection Agency (EPA) (Michałowicz and Duda, 2007; Mohd, 2022).

To address this issue, numerous studies have focused on the removal of phenolic compounds using physicochemical and biological treatment methods. Among physicochemical techniques, coagulation–flocculation has proven effective for the removal of organic pollutants (Verma et al., 2012; Karnena and Saritha, 2022). Conventional coagulants, such as aluminium and iron salts, are widely used due to their high efficiency (Cui et al., 2020). However, their performance can be limited when dealing with certain synthetic organic compounds due to their specific physicochemical properties (Zoumpouli et al., 2023).

In recent years, increasing attention has been given to the development of sustainable alternatives (Koul et al., 2022; Alazaiza et al., 2025). In the present study, acorn bark was investigated as a coagulant aid to enhance the removal of phenolic compounds from water. A factorial experimental design was applied to evaluate the principal and interactive effects of operational parameters on removal efficiency of phenolic compounds from water.

Beyond decontamination, the management of these effluents must transition toward a circular economy. In arid and semi-arid regions, such as Algeria, where freshwater availability is severely limited, the reuse of treated wastewater (TWW) for agriculture has become a strategic necessity. TWW is often enriched with organic matter and essential nutrients (including nitrogen and phosphorus), which can improve soil fertility and reduce reliance on synthetic fertilisers (Makhadmeh et al., 2021). However, the safe application of TWW is governed by strict international standards (FAO/WHO) because residual pollutants such as heavy metals, salts, or poorly degraded phenolic traces can negatively impact soil structure and plant health (Lado and Ben-Hur, 2010).

The efficiency of the chosen treatment technology is therefore the determining factor for the safety and productivity of irrigation. While biological processes are effective for standard nutrient reduction, physicochemical methods like optimised coagulation are crucial for removing specific recalcitrant contaminants and suspended solids. It is essential to understand how different treatment chains influence the final quality of the effluent and, subsequently, its impact on the soil-plant system.

In this context, the present study aims to bridge the gap between innovative wastewater treatment and sustainable agriculture. First, we evaluate the performance of a coagulation process enhanced by acorn bark biomaterial for the removal of phenolic compounds. Second, we assess the preliminary short-term agricultural viability of the treated effluent by comparing the effects of physicochemical and biological treatment regimes on the growth of green beans (*Phaseolus vulgaris* L.) and soil properties, using conventional water as a reference.

## MATERIALS AND METHODS

### Preparation of the bio-adsorbent: activated acorn bark

To develop a sustainable treatment process, activated acorn bark was prepared as an adsorbent. The raw bark was first washed with distilled water and dried at 105°C for 24 hours. Chemical activation was performed by immersing 10 g dried bark in 100 mL 40% $H_2SO_4$ solution at 105°C for 2 hours. After treatment, the material was rinsed thoroughly with distilled water until a neutral pH was reached, then dried again at 105°C for 24 hours. The resulting bio-material was calcined at 500°C for 2 hours, ground, and sieved to obtain a particle size ≤ 500 μm.

Activated acorn bark was employed in this study as a coagulant aid within a coagulation-flocculation process. Therefore, the objective was not to investigate the adsorption mechanism, but rather to evaluate its synergistic effect on the coagulation-flocculation performance.

### Organic pollutants

Three phenolic compounds were selected based on their chemical structures to assess the role of carboxylic and hydroxyl functional groups: phenol (PHL), gallic acid (GA), and humic substances (HS). Although not directly identical to the full mixture of real effluents, these model compounds display varying degrees of structural complexity and molecular weights, effectively reflecting the diverse organic matter fractions present in wastewater.

Residual concentrations were determined via UV-Vis spectrophotometry at 270 nm (PHL) and 254 nm (GA and HS).

### Wastewater treatment procedures

The treatment efficiency was evaluated through three distinct treatment processes (adsorption, coagulation and hybrid system) to determine the impact of the organic composition on removal performance.

### Adsorption experiments

Batch adsorption experiments were carried out to assess the affinity of the activated acorn bark for the phenolic compounds. A known mass of the biomaterial was mixed with 500 mL aqueous solution at 150 rpm. Samples were taken at predetermined time intervals, filtered, and analysed to determine the residual concentration of pollutants.

This stage aimed to identify the maximum adsorption capacity (qmax) based on the functional groups (hydroxyl and carboxylic) of the target molecules.

### Coagulation-flocculation experiments

Conventional coagulation tests were performed using aluminium sulfate alone. The process consisted of three successive stages: rapid mixing at 200 rpm for 3 min, slow mixing at 60 rpm for 30 min, and sedimentation for 30 min. This allowed for a baseline measurement of removal efficiency through charge neutralisation and precipitation.

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### Hybrid treatment system

A combined process was developed where the activated acorn bark was used as a coagulant-aid. The biomaterial was added to the 500 mL solution 30 seconds after the aluminium sulfate. This hybrid approach was designed to leverage both the floc-forming properties of the salt and the adsorptive surface area of the activated bark.

In this hybrid system, an adsorbent mass of 1.5 g was used in combination with 50 mg/L aluminium sulfate to treat a mixture of the three organic compounds (PHL, GA, HS). The concentrations of organic compounds before and after treatment were determined by measuring the total organic carbon.

### Optimisation of the hybrid system

To investigate the effects of the operational parameters on the treatment efficiency and optimise the process, a 2-level full factorial design ($2^3$) was implemented. Three independent factors were selected for evaluation: organic matter concentration ($X_1$), coagulant dose ($X_2$), and adsorbent mass ($X_3$). Each factor was investigated at two levels, designated as low (-1) and high (+1), resulting in a total of 8 ($2^3$) standard experimental runs. To assess the reproducibility of the method and estimate the experimental error, two additional replicates were performed at the centre point (0), bringing the total number of experimental runs to 10. The matrix of the experimental design, including the actual and coded levels of the independent variables, is detailed in Table 1.

**Table 1.** Factors and levels used in the full factorial design (*Values represent individual concentrations of gallic acid + phenol + humic substances, respectively)

| Factor | Low level | High level |
|---|---|---|
| $X_1$: Organic matter concentration, mg $L^{-1}$ | 20+20+10* | 50+50+20* |
| $X_2$: Coagulant dose, mg $L^{-1}$ | 10 | 50 |
| $X_3$: Adsorbent mass, g | 0 | 1.5 |

The response variable selected to evaluate the efficiency of the hybrid process was the removal efficiency (Y, %). The relationship between the independent factors and the response was mathematically modelled using a first-order polynomial equation with interaction terms, expressed as follows:

$$Y = a_o + \sum a_i X_i + \sum a_{ij} X_i X_j + \sum a_{ijk} X_i X_j X_k + \ldots, \quad (1)$$

where $Y$ is the response function, $a_0$ is the constant of the model, $a_i$ is the main effect of $a$ factor, $a_{ij}$ and aijk are the interaction effects. $X_i$, $X_j$, and $X_k$ represent the coded independent variables (factors).

Statistical analysis, model development, and surface response plotting were performed using JMP software (version 8). The statistical validation of the generated predictive model was rigorously assessed through analysis of variance (ANOVA). The statistical significance of the model and its terms was evaluated using the Fisher's F-test and its associated probability ($p < 0.05$). The quality of fit and predictive capability of the model were analysed using the coefficient of determination ($R^2$) and the adjusted coefficient of determination ($R^2$adj).

Response optimisation was conducted using the desirability function approach in JMP (version 8). The optimisation objective was set to maximise removal efficiency and to determine the optimal combinations of organic matter concentration, coagulant dose, and adsorbent mass. The overall desirability was computed as the geometric mean of individual desirability values, ranging from 0 (undesirable) to 1 (completely desirable). Experimental conditions yielding a desirability score closest to 1 were identified as the optimal operational settings.

### Irrigation trial design

*Study site and climate*

The irrigation trials were conducted at the experimental station of the National Higher School of Agronomy in Algiers, Algeria. The site is characterised by a Mediterranean climate with a mean annual rainfall of 573 mm and evapotranspiration of 1239 mm.

*Irrigation regimes and water sources*

The experiment was conducted over a period of 60 days following a total randomisation design with a single variation factor (water quality) and 5 replicates per treatment. The setup consisted of pots (25 cm in diameter) with 1 plant per pot of green bean (Tema variety), grown under greenhouse conditions under a natural photoperiod. The experimental substrate was a silty loam soil with pH 7.26, electrical conductivity 730 µS/cm, organic matter content 2.1%, and cation exchange capacity 25.43 meq/100g. No fertilisers or chemical amendments were applied during the study to specifically isolate and evaluate the nutrient contributions of the different wastewater qualities on the soil-plant system.

Three water sources were compared using a localised irrigation method applied every 2 days, which allowed for a controlled assessment of the treated effluent's impact on the soil-plant system:

- Conventional water (CW): groundwater sourced from an agricultural well as the control;
- Biologically treated wastewater (BTW); and
- Physicochemically treated wastewater (PCTW), obtained using the hybrid treatment process described above.

Irrigation requirements were calculated based on crop water needs using the CROPWAT model. Estimations were derived from the crop coefficient (Kc) of green bean and climatic parameters, including temperature, relative humidity, wind speed, and solar radiation. This resulted in a total cumulative irrigation depth of 290 mm applied per treatment over the 60 day experimental period.

*Plant and soil measurements*

Plant growth parameters and yield components, including final plant height, leaf length, leaf width, and pod length, were measured at the end of the crop cycle using a graduated ruler, while the number of seeds per pod was manually counted at harvest. Total nitrogen (N) content in both plant tissues and soil samples was determined using the classic Kjeldahl digestion method. Soil physicochemical properties were analysed according to standard procedures. Soil pH

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and electrical conductivity (EC) were measured in soil-water suspensions using a calibrated pH meter and conductivity meter, respectively. Total calcium carbonate was determined volumetrically using a Bernard calcimeter, and soil organic matter content was quantified using the Anne method. Particle size distribution was analysed by the Robinson pipette sedimentation method according to the AFNOR NF X 31-107 standard. Cation exchange capacity (CEC) along with exchangeable bases were determined following the Metson method (AFNOR NF X 31–130), using atomic absorption spectrometry for exchangeable bases and colorimetry for CEC evaluation.

### Statistical analysis

Experimental data were analysed with ANOVA with a significance level of 0.05, considering irrigation water quality as the main factor. Statistical analyses were performed using the DSAASTAT software (ver 1.0192, A. Onofri, 2008).

## RESULTS AND DISCUSSION

### Application of treatments to remove organic compounds from water

*Adsorption*

Adsorption capacity primarily depends on the chemical structure of the target molecule, which governs its interaction with the adsorbent surface. Fig. 1 shows that phenol, as the organic compound with the simplest structure, exhibits the fastest adsorption, reaching a plateau within 30 minutes that indicates near-complete pollutant removal, corresponding to an adsorbed amount of 9.67 mg/g. In contrast, gallic acid shows slower and partial adsorption, with a plateau of 6.44 mg/g reached after approximately 180 minutes. Humic substances, which have the most complex chemical structure, reach equilibrium in just 10 minutes but with the lowest adsorption capacity of 2.03 mg/g.

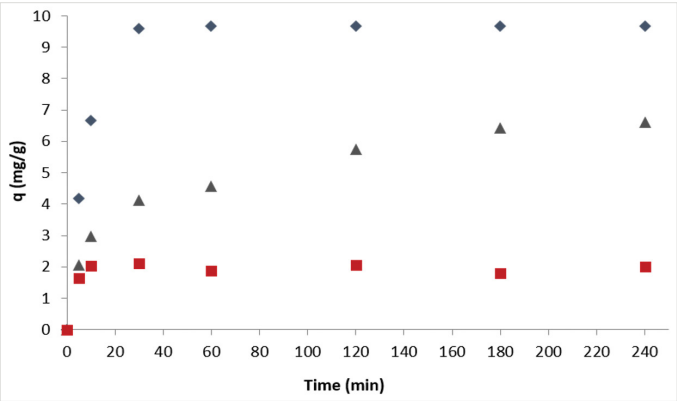



**Figure 1.** ffect of pollutant molecular structure on the adsorption capacity (q) over time for phenol (◆), gallic acid (▲), and humic substances (■) onto activated acorn bark

*Coagulation-flocculation*

The efficiency of coagulation is strongly influenced by the molecular structure of the compound to be removed. The results shown in Fig. 2 indicate that for organic compounds with a simple chemical structure, such as phenol, the removal efficiency by coagulation remains relatively low across the different coagulant doses. Zhao et al. (2016) and Sun et al. (2025) showed that iron and aluminium salts do not result in significant removal of simple organic molecules such as salicylic acid, phthalic acid, and phenol.

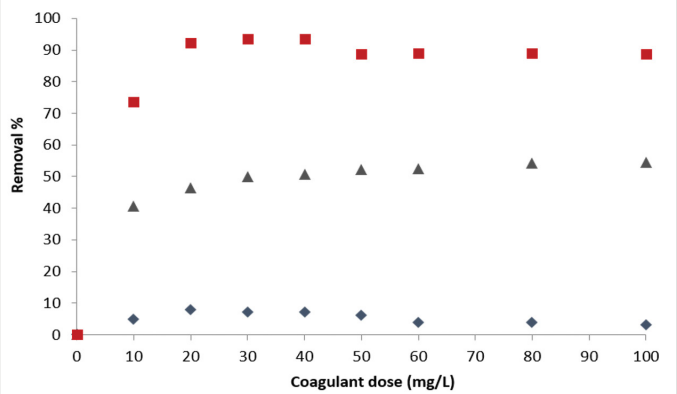



**Figure 2.** Effect of pollutant molecular structure on the coagulation efficiency (removal %) of phenol (◆), gallic acid (▲), and humic substances (■) as a function of coagulant dose

Figure 2 shows high percentages for the removal of gallic acid (50.62%) and humic substances (93.65%), achieved even at low coagulant doses of 40 mg/L. Molecules that contain –COOH functional groups are easily removed by coagulation. The significant differences between the removal percentages of gallic acid and humic substances seem to indicate a strong correlation between the increase in efficiency and the number of –COOH functional groups.

*Hybrid treatment system*

As previously shown, simple organic molecules are not, or only minimally, removed by coagulation. Fig. 3 demonstrates that the use of activated acorn bark as a coagulant-aid significantly enhances the removal of organic matter from water compared to conventional coagulation treatment. The initial TOC concentration dropped from 29.61 to 24.48 mg/L (13.95% removal) with conventional coagulation, whereas the hybrid treatment achieved a residual TOC level of 0.974 mg/L (96.71% removal). This improvement is attributed to the additional contribution of adsorption provided by the coagulant-aid.

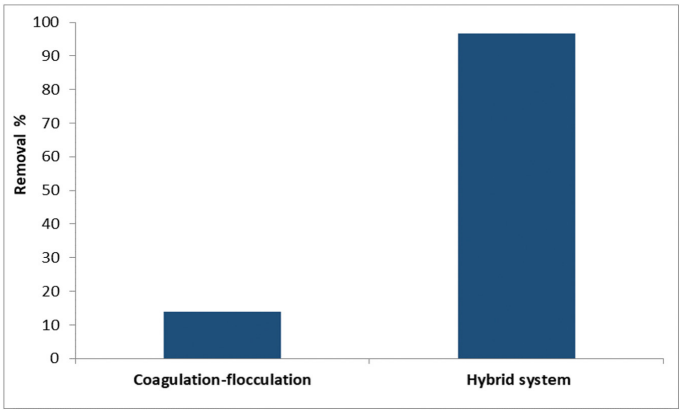



**Figure 3.** OOrganic matter removal efficiency (%) by the coagulation-flocculation process using aluminium sulfate and the hybrid system combining aluminium sulfate with acorn bark

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These results suggest that the hybrid system is effective for the removal of organic compounds with varying chemical structures.

*Optimisation of the hybrid system by full factorial design*

The full factorial design methodology was used to optimise the treatment process and to establish the correlation between organic matter removal percentage and three input variables. Fig. 4 presents the fit between the experimental data and the values simulated using the program JMP (version 8) for organic matter removal. The high determination coefficients ($R^2$ = 0.996 and adjusted $R^2$ = 0.986) confirm that the model was successful in correlating the response to the studied parameters.

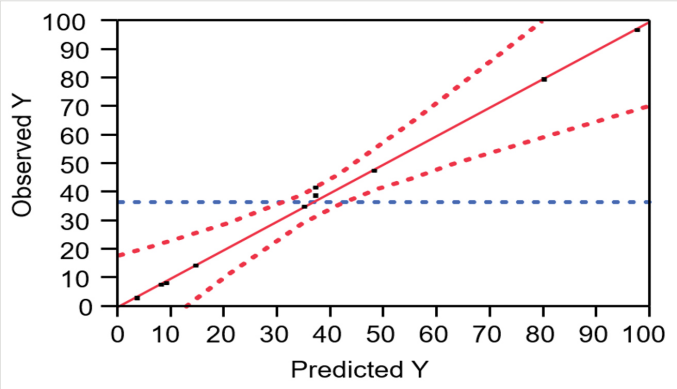



**Figure 4.** Observed versus predicted values for organic matter removal efficiency (%) using the Full Factorial Design model for the optimisation of the treatment process

*Student's t-test*

The Student's t-test determines the significance of the regression coefficients of the different parameters. A large t-value associated with a low p-value (< 0.05) of a variable indicates a high significance of the corresponding model term. After eliminating the insignificant effects using the stepwise method, Table 2 shows the remaining significant effects. The results show that the model is influenced by the concentration of organic compounds ($X_1$), the coagulant dose ($X_2$), the adsorbent mass ($X_3$), the interactions between $X_1X_3$ and $X_2X_3$. The most significant effect is the activated acorn bark with a positive value. This means that the removal of organic matter increases with the activated acorn bark dose. The empirical model for the removal percentage (Y) is:

$$Y = 36.957 - 13.135*X_1 + 5.295*X_2 + 28.2325*X_3 - 10.4825*X_1X_3 + 2.2625*X_2X_3 \quad (2)$$

**Table 2.** Estimated regression significant coefficients for the removal yield (* p <0.05)

| Parameter | Estimate | Standard error | t-value | p-value |
|---|---|---|---|---|
| Constant | 36.957 | 0.912423 | 40.50 | <0.0001* |
| $X_1$ | -13.135 | 1.02012 | -12.88 | 0.0002* |
| $X_2$ | 5.295 | 1.02012 | 5.19 | 0.0066* |
| $X_3$ | 28.2325 | 1.02012 | 27.68 | <0.0001* |
| $X_1*X_3$ | -10.4825 | 1.02012 | -10.28 | 0.0005* |
| $X_2*X_3$ | 2.2625 | 1.020212 | 2.22 | 0.0408* |

*Analysis of variance*

ANOVA was used to validate the adjusted model, and the summary is shown in Table 3. The F-value obtained (213.8367) was statistically significant (p<0.05), indicating that the model is valid for predicting the removal of organic matter with the studied variables.

**Table 3.** Analysis of variance (ANOVA) for the predictive model of organic matter removal efficiency (* p <0.05)

| Source | Degrees of freedom | Sum of squares | Mean square | F-value | p-value |
|---|---|---|---|---|---|
| Model | 5 | 8901.1282 | 1780.23 | 213.8367 | 0.0001* |
| Residual | 4 | 33.3007 | 8.33 | | |
| Total | 9 | 8934.4288 | | | |

*Optimisation design using the desirability function*

The desirability function approach is the most current and strongly suggested method for the optimisation of one or more responses (Mourabet et al., 2012). Fig. 5 shows the combination of organic compound concentrations (PHL 20 mg/L, GA 20 mg/L, and SH 10 mg/L), an aluminium sulfate dose of 50 mg/L, and 1.5 g activated acorn bark allows for an optimal organic matter removal efficiency of 96.364% with a desirability of 0.946. This high desirability indicates that the activated acorn bark effectively complements the aluminium sulfate, likely through the synergistic effect of charge neutralisation and the high adsorption capacity provided by the bark's porous structure and functional groups.

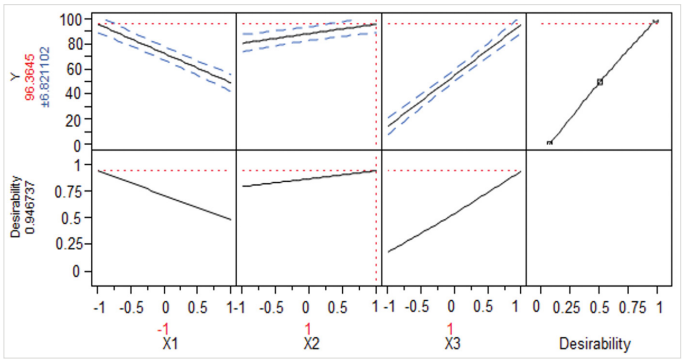



**Figure 5.** Desirability function for removal of organic matter using the Full Factorial Design model

The optimised operating conditions were subsequently applied to actual municipal wastewater. The physicochemical characteristics of the wastewater before and after treatment are presented in Table 4. The treated effluent was then evaluated through a short-term localised irrigation experiment on green beans to assess its physicochemical quality and preliminary agricultural performance.

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**Table 4.** Physicochemical characteristics of municipal wastewater before and after treatment (all values are expressed in mg/L). [BOD : biochemical oxygen demand, COD : chemical oxygen demand]

| Parameters | Before | After |
|---|---|---|
| $BOD_5$ | 180.00 | 28.13 |
| COD | 420.92 | 44.41 |
| Iron | 0.167 | 0.092 |
| Cadmium | 0 | 0 |
| Nickel | 0.015 | 0.004 |
| Zinc | 0 | 0 |
| Manganese | 0.170 | 0.03 |
| Copper | 0 | 0 |
| Lead | 0 | 0 |
| Chromium | 0 | 0 |

## Irrigation experiment

*Irrigation water assessment*

Microbiological quality was not evaluated in this study, as the primary objective was to assess the physicochemical performance and agricultural suitability of the treatment process. According to WHO guidelines (World Health Organization, 2006), when localised drip irrigation is applied to crops whose harvested parts have no contact with the soil, specific microbiological performance targets are not necessary. The quality of the irrigation waters was assessed through the analysis of physicochemical properties (Table 5). The measured values are compared with the national standards for agricultural water reuse in Algeria.

The treated wastewater and conventional water applied in irrigation had a slightly alkaline reaction, with pH values falling within a favourable range of 6.5–8.5. Additionally, the values of electrical conductivity (EC) and sodium adsorption ratio (SAR) indicate that the waters used for irrigation have a moderate salinity, below the allowable limit.

The nitrate concentration in PCTW (14.82 mg/L) is higher than in BTW (7.22 mg/L) and CW (2.35 mg/L). Although none of these concentrations exceed the recommended limit (30 mg/L), it is evident that biological treatment is most effective in reducing nitrate levels. Moreover, biochemical oxygen demand (BOD) and chemical oxygen demand (COD) values are within acceptable standards, further confirming the suitability of the treated wastewater for agricultural use.

Despite the very low concentrations of nickel and manganese observed in the treated wastewater, it can be concluded that heavy metals are virtually absent from the irrigation water.

**Table 5.** Physico-chemical parameters of irrigation waters [CW: Conventional water, BTW: Biologically treated wastewater, PCTW: Physicochemically treated wastewater, EC: electrical conductivity, SAR: Sodium adsorption ratio, BOD: Biochemical oxygen demand, COD: Chemical oxygen demand. Standard : National standard (Official Journal of the Algerian Republic, 2012)]

| Parameters | Unit | CW | PCTW | BTW | Standard |
|---|---|---|---|---|---|
| pH | - | 7.27 | 7.13 | 7.39 | 6.5-8.5 |
| EC | dS $m^{-1}$ | 1.53 | 1.54 | 1.14 | 3 |
| SAR | - | 0.114 | 0.115 | 0.086 | 0.2 |
| $BOD_5$ | mg $L^{-1}$ | - | 28.13 | 15.23 | 30 |
| COD | mg $L^{-1}$ | - | 44.41 | 30.61 | 90 |
| Nitrate | mg $L^{-1}$ | 2.35 | 14.82 | 7.22 | 30 |
| Iron | mg $L^{-1}$ | 0.047 | 0.092 | 0.052 | 20 |
| Cadmium | mg $L^{-1}$ | 0 | 0 | 0 | 0.05 |
| Nickel | mg $L^{-1}$ | 0 | 0.004 | 0.001 | 2 |
| Zinc | mg $L^{-1}$ | 0 | 0 | 0 | 10 |
| Manganese | mg $L^{-1}$ | 0 | 0.03 | 0.01 | 10 |
| Copper | mg $L^{-1}$ | 0 | 0 | 0 | 5 |
| Lead | mg $L^{-1}$ | 0 | 0 | 0 | 10 |
| Chromium | mg $L^{-1}$ | 0 | 0 | 0 | 1 |

*Soil analysis evaluation*

Table 6 shows that there are slight variations in the soil texture after irrigation. Clay and sand fractions remain relatively stable, while minor changes are observed in the silt content.

**Table 6.** Soil texture before and after irrigation (CW: Conventional water, BTW: Biologically treated wastewater, PCTW: Physicochemically treated wastewater)

| Parameters | | Before irrigation | After irrigation | | |
|---|---|---|---|---|---|
| | | | CW | BTW | PCTW |
| Particle size distribution, % | Clay | 20.13 | 20.23 | 20.69 | 20.80 |
| | Fine silt | 36.29 | 38.16 | 38.61 | 39.03 |
| | Coarse silt | 18.96 | 16.01 | 17.74 | 15.60 |
| | Fine sand | 8.18 | 8.51 | 8.63 | 8.50 |
| | Coarse sand | 16.43 | 16.58 | 16.80 | 16.70 |

Fig. 6 shows the variation in chemical properties of the soil samples analysed.

Soil pH increases slightly after irrigation with the three types of water, pH values remaining below the standard limit of approximately 8.5 for agricultural soils. This observation aligns with previous reports (Rusan et al., 2007; Christou et al., 2014; Sdiri et al., 2023) that showed that irrigation with treated wastewater does not cause significant changes in soil pH.

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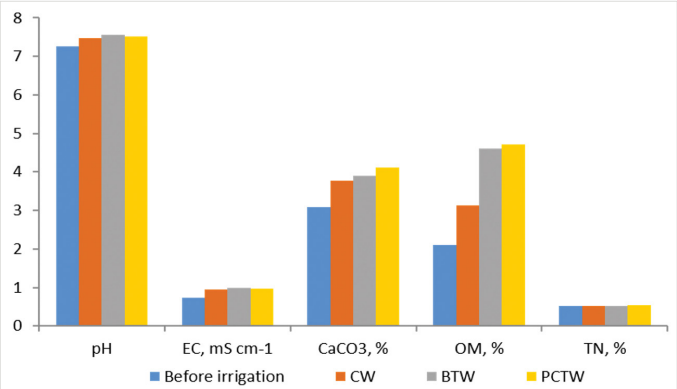



**Figure 6.** Chemical properties of soils before and after irrigation with conventional water (CW), biologically treated wastewater (BTW), and physico-chemically treated wastewater (PCTW)

Soil electrical conductivity (EC) values show a clear increase in irrigated soils, though below the upper limit of 4 dS/m for agricultural soils. Gharaibeh et al. (2016) attribute this increase in electrical conductivity to the high concentration of total dissolved solids in treated wastewater. Furthermore, the test site is characterised by high evapotranspiration during summer, which can lead to the accumulation of salts from irrigation water in the soil, consequently increasing salt concentration in the root zone. However, this salinity buildup can be mitigated through leaching, resulting from frequent water applications via irrigation (Christou et al., 2014).

Organic matter values show an increase in soils irrigated with treated wastewater. The high OM content observed in irrigated soils can be attributed to the substantial organic load in effluents (high BOD and COD), which may contribute to improved soil fertility (Gatta et al., 2016). These results regarding the increase in soil organic matter content due to the application of treated wastewater seem to confirm those reported in the literature (Gharaibeh et al., 2007; Galavi et al., 2010; Albalasmeh et al., 2022).

The results in Fig. 7 show that the treated wastewater supplies the soil with higher amounts of nitrogen and essential nutrients (Na, K, Ca, Mg) necessary for plant growth and development.

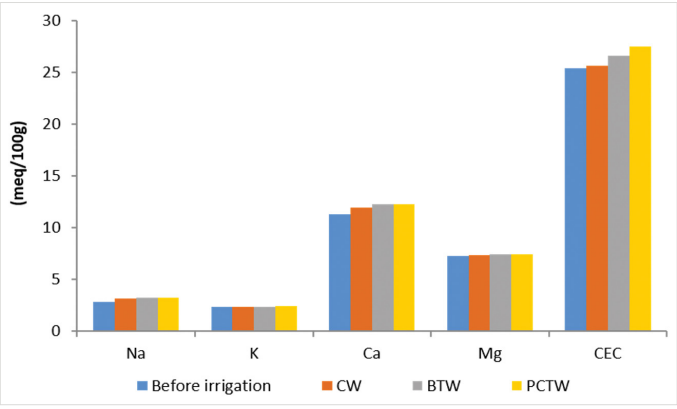



**Figure 7.** Soil exchange complex elements before and after irrigation with conventional water (CW), biologically treated wastewater (BTW), and physico-chemically treated wastewater (PCTW)

This represents an important quality parameter for the agricultural reuse of these waters. Nutrient concentrations are directly related to the origin of the wastewater and to the treatment techniques applied (Etchebarne et al., 2019). This led to a slight increase in CEC values in soils irrigated with wastewater, with noticeably higher levels compared to other water sources. These findings are consistent with the previous reports (Farhadkhani et al., 2018).

The statistical analysis results are summarised in following Table 7. These results show probabilities below the 5% threshold, indicating a statistically significant effect of the factors on the soil physico-chemical parameters, with the exception of total nitrogen. The Fisher's test, used to compare the means of the parameters corresponding to the three water qualities, allowed the grouping of statistically non-different means into homogeneous groups.

**Table 7.** ANOVA analysis of soil physico-chemical parameters (CW: Conventional water, BTW: Biologically treated wastewater, PCTW: Physicochemically treated wastewater, EC: electrical conductivity; $CaCO_3$: calcium carbonate; OM: organic matter; TN: total nitrogen; CEC: cation exchange capacity. Values represent mean (n = 5), Different letters (a, b, c) in the same column indicate statistically significant differences; * p <0.05)

| Parameters | *F*-ration | p-value | Means | | |
|---|---|---|---|---|---|
| | | | CW | BTW | PCTW |
| pH | 12.50 | 0.008** | 7.47$^{b}$ | 7.55$^{a}$ | 7.51$^{a}$ |
| EC, mS cm$^{-1}$ | 211.20 | <0.0001** | 952.00$^{c}$ | 988.33$^{a}$ | 963.67$^{b}$ |
| $CaCO_3$, % | 229.40 | <0.0001** | 3.77$^{c}$ | 3.89$^{b}$ | 4.12$^{a}$ |
| OM, % | 5607.60 | <0.0001** | 3.12$^{c}$ | 4.60$^{b}$ | 4.72$^{a}$ |
| TN, % | 3.70 | 0.089 | 0.51 | 0.52 | 0.54 |
| CEC | 12.7 | 0.007** | 25.43$^{b}$ | 26.59$^{a}$ | 27.48$^{a}$ |

Thus, the wastewater resulted in statistically similar values for pH and CEC, all of which were higher than those associated with EC water. In contrast, significant differences were revealed between soil parameters: EC, calcium carbonate ($CaCO_3$), and organic matter (OM).

*Evaluation of plant growth parameters*

We observed the growth developments of the bean crop at different vegetative stages, as illustrated in Fig. 8. We noted accelerated growth with treated wastewater, resulting in better plant development compared to conventional water.

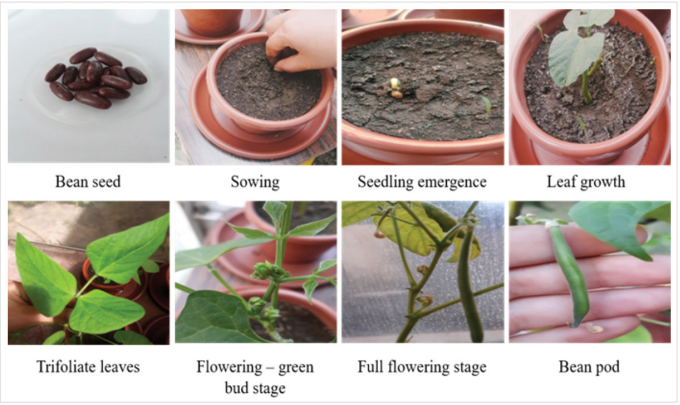



**Figure 8.** Growth stages of green bean (*Phaseolus vulgaris* L.).

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This finding is consistent with previous reports on grapevines (Paranychianakis et al., 2004; Etchebarne et al., 2019). It appears that the physiological and morphological characteristics, as well as crop yields, depend on various factors, including water quality and nutrient availability. (Iqbal et al., 2015) reported that better plant growth can be attributed to the higher availability and uptake of nutrients present in wastewater.

Seedling growth evaluation was performed using the parameters detailed in Table 8. A clear increase is seen in the values of all measured parameters at the end of the cycle for the treated wastewater. This improvement in the characteristics of the bean crop can be attributed to the higher concentration of nutrients and the substantial amount of organic matter present in the wastewater. Moreover, the concentrations of certain essential inorganic ions N, P, K, Ca, and Mg are higher in the wastewater than in the groundwater, which plays a beneficial role for the plants (Hashem and Qi, 2021). Many authors have also reported that adequate nitrogen uptake progressively stimulates plant growth (Pascual et al., 2009; Etchebarne et al., 2019; Seleiman et al., 2021).

**Table 8.** Growth parameters of green bean (*Phaseolus vulgaris* L.) irrigated with treated wastewaters (CW: Conventional water, BTW: Biologically treated wastewater, PCTW: Physicochemically treated wastewater)

| Quality | CW | BTW | PCTW |
|---|---|---|---|
| Height, cm | 49.07 | 51.97 | 55.10 |
| Leaf length, cm | 9.33 | 10.73 | 11.03 |
| Leaf width, cm | 4.90 | 5.27 | 5.47 |
| Pod length, cm | 9.23 | 9.73 | 9.83 |
| Number of seeds/pod | 5.33 | 6.33 | 6.67 |
| Nitrogen, % | 2.93 | 3.40 | 3.48 |

Table 9 summarises the statistical parameters obtained from the ANOVA analysis of the plant characteristics. These results indicate a statistically significant effect of the factors on the parameters related to crop growth, except for the number of seeds per pod.

**Table 9.** ANOVA analysis of growth parameters of green bean (*Phaseolus vulgaris* L.); CW: Conventional water, BTW: Biologically treated wastewater, PCTW: Physicochemically treated wastewater. Values represent mean (n = 5), Different letters (a, b, c) in the same column indicate statistically significant differences; * p <0.05.

| Parameters | *F*-ration | p-value | Means | | |
|---|---|---|---|---|---|
| | | | CW | BTW | PCTW |
| Height, cm | 416.66 | <0.0001** | 49.07$^{c}$ | 51.97$^{b}$ | 55.10$^{a}$ |
| Leaf length, cm | 123.50 | 0.00001** | 9.33$^{c}$ | 10.73$^{b}$ | 11.03$^{a}$ |
| Leaf width, cm | 44.60 | 0.00025** | 4.90$^{c}$ | 5.27$^{b}$ | 5.47$^{a}$ |
| Pod length, cm | 10.33 | 0.011* | 9.23$^{b}$ | 9.73$^{a}$ | 9.83$^{a}$ |
| Number of seeds/pod | 4.33 | 0.068 | 5.33 | 6.33 | 6.67 |
| Nitrogen, % | 409.29 | <0.0001** | 2.93$^{c}$ | 3.40$^{b}$ | 3.48$^{a}$ |

The Fisher test used for comparing the means of the parameters corresponding to the three water qualities allowed for the grouping of statistically non-different means into homogeneous groups. Thus, the treated wastewater (BTW and PCTW) resulted in statistically similar pod lengths, with higher values compared to those obtained with CW. In contrast, the analysis revealed significant differences between the means of the other growth parameters, which were grouped into distinct homogeneous groups (groups a, b, and c).

## CONCLUSION

This study highlights both the optimisation of a hybrid wastewater treatment process and its preliminary physicochemical and short-term agricultural assessment under localised irrigation conditions. The use of activated acorn bark as a coagulant aid significantly enhanced the coagulation process, achieving up to 96.36% removal of organic matter under optimal conditions (50 mg/L aluminium sulfate and 1.5 g activated acorn bark). This high treatment efficiency led to a substantial improvement in the physicochemical quality of the treated wastewater.

Short-term irrigation with wastewater treated by different processes (PCTW and BTW) demonstrated that the evaluated physicochemical parameters complied with the relevant irrigation water quality standards and contributed positively to soil fertility by increasing organic matter and essential nutrients. Among the two, PCTW showed slightly superior performance in terms of nitrate content, soil quality, and plant growth under the tested conditions. Overall, these short-term results provide meaningful preliminary insights into the agricultural performance of the treated wastewater under localised irrigation conditions. However, long-term field studies, together with microbiological and contaminant assessments, remain necessary before large-scale agricultural reuse can be fully established.

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## CRediT AUTHORSHIP CONTRIBUTION STATEMENT

**Nadjiba Boulahia:** Methodology, Formal analysis, Writing-original draft. **Dalila Hank**: Supervision, Methodology, Validation. **Fouad Sellam:** Methodology, Formal analysis, Validation. **Abdelmalek Chergui:** Supervision, Project administration.

## DECLARATION OF COMPETING INTEREST

The authors declare that they have no competing interests.

## ETHICS AND PERMIT APPROVALS

The authors declare that this study did not require any ethical approval or specific research permits.

## DATA AVAILABILITY STATEMENT

All relevant data supporting the findings of this study are included within the manuscript.

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## AI USE STATEMENT

None.

## SAŽETAK

### Optimizacija hibridnog postupka obrade otpadnih voda primjenom potpunoga faktorijelnog dizajna te preliminarna fizikalno-kemijska i kratkoročna agronomska procjena za ponovnu uporabu u navodnjavanju

U ovom je istraživanju razvijen ekonomičan hibridni postupak obrade za uklanjanje organskih spojeva iz otpadnih voda, uz preliminarnu procjenu mogućnosti poljoprivredne ponovne uporabe obrađenog efluenta. Istražena je dekontaminacija vode onečišćene fenolom, galnom kiselinom i huminskim tvarima primjenom dviju metoda: adsorpcije na aktiviranu koru žira i koagulacije–flokulacije aluminijevim sulfatom. Radi postizanja najveće učinkovitosti obrade razvijen je hibridni sustav, a optimalni radni uvjeti određeni su metodologijom potpunoga faktorijelnog dizajna.

Pri optimiziranim uvjetima, uz primjenu 50 mg/L aluminijeva sulfata i 1,5 g aktivirane kore žira, postignuta je visoka učinkovitost uklanjanja organske tvari od 96,36%. U kontekstu nestašice vode u Alžiru provedeno je pokusno navodnjavanje graha (*Phaseolus vulgaris* L.) lokaliziranim sustavom navodnjavanja kako bi se procijenila fizikalno-kemijska kakvoća obrađenog efluenta i njegova kratkoročna agronomska učinkovitost. Pokusom su procijenjeni preliminarni učinci hibridnog postupka na svojstva tla i rast biljaka u kratkoročnom razdoblju.

Eksperimentalni dizajn obuhvatio je usporedbu kvalitete vode, svojstva tla i rast biljaka pri primjeni triju izvora vode: fizikalno-kemijski obrađene otpadne vode, biološki obrađene otpadne vode i konvencionalne vode. Rezultati su pokazali da analizirani fizikalno-kemijski pokazatelji fizikalno-kemijski obrađene otpadne vode udovoljavaju relevantnim međunarodnim smjernicama za poljoprivrednu ponovnu uporabu. Nadalje, pokus navodnjavanja pokazao je da su fizikalno-kemijski obrađene otpadne vode značajno poboljšale plodnost tla te potaknule izraženiji vegetativni rast u usporedbi s konvencionalnom vodom. Dobiveni rezultati ističu potencijal ovog hibridnog postupka za proizvodnju obrađene otpadne vode zadovoljavajuće fizikalno-kemijske kvalitete i odgovarajućeg sadržaja hraniva, uz istodobno poticanje kratkoročnog rasta biljaka u uvjetima lokaliziranog navodnjavanja.

**Ključne riječi:** obrada otpadnih voda; koagulacija–flokulacija; adsorpcija; potpuni faktorijelni dizajn; održiva poljoprivreda.

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