Original Research Article **CODEN: PJZSAZ e-ISSN: 1331-7776**

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

Nadjiba Boulahia[1,2,3], Dalila Hank[1,2,3,*], Fouad Sellam[1], Abdelmalek Chergui[3,4]

[1]*Ecole Nationale Supérieure Agronomique (ENSA), Hacen Badi, El Harrach Alger, Algérie*

[2]*Laboratoire de Maitrise de l'Eau en Agriculture, Ecole Nationale Supérieure Agronomique (ENSA), Hacen Badi, El Harrach Alger, Algérie*

[3]*Laboratoire des Sciences et Techniques de l'Environnement, Ecole Nationale Polytechnique, Hacen Badi, El Harrach Alger, Algérie*

[4]*Ecole Nationale Polytechnique, Hacen Badi, El Harrach Alger, Algérie*

*Correspodence: Dalila Hank (dalila.hank@edu.ensa.dz)

Article info:

Received: 5 June 2026

Revised: 6 August 2026

Accepted: 7 August 2026

Available online: 7 August 2026

Academic Editor: Prof. Marina Piria, University of Zagreb Faculty of Agriculture, Croatia

How to Cite:

Boulahia N., Hank D., Sellam F., Chergui A. (2026). Optimization of a hybrid wastewater treatment via full factorial design and its preliminary physicochemical and short-term agronomic assessment for irrigation reuse. Agric Conspec Sci 91: e011.

This article is an accepted manuscript and an uncorrected proof that has not undergone copyediting, language editing, or technical checks. The final version of the record may differ from this version.

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 aluminum sulfate. To maximize treatment efficiency, a hybrid system was developed. The optimal operating conditions were determined using a factorial design methodology.

Under optimized parameters conditions (50 mg/L of aluminum sulfate and 1.5 g of activated acorn bark) the treatment efficiency achieved a high organic matter removal rate of 96.36%. In the context of water scarcity in Algeria, a localized irrigation trial was conducted on green beans (*Phaseolus vulgaris*) to evaluate the physicochemical quality of the treated effluent and its short-term agronomic performance under localized irrigation conditions. This experiment evaluated the preliminary effects of the hybrid process on soil properties and plant growth in the short term.

The experimental design compared water quality, soil properties, and plant growth across three sources: physicochemically treated wastewater (PCTW), biologically treated wastewater, and conventional water. Results showed that the analyzed physicochemical parameters of PCTW complied 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 localized irrigation conditions.

Keywords: Wastewater treatment, coagulation-flocculation, adsorption, factorial design, sustainable agriculture.

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 characterized by high stability and toxicity, which has led the Environmental Protection Agency (EPA) to classify them among the most harmful contaminants (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 aluminum 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 because of 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 (Nitrogen, Phosphorus, ...), which can improve soil fertility and reduce reliance on synthetic fertilizers (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 optimized 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 agronomic viability of the treated effluent by comparing the effects of physicochemical and biological treatment regimes on the growth of green beans (*Phaseolus vulgaris*) 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 of dried bark in 100 mL of a 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.

It should be emphasized that 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 of aqueous solution at 150 rpm. Samples were taken at predetermined time intervals, filtered, and analyzed to determine the residual concentration of pollutants.

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

Coagulation–Flocculation experiments

Conventional coagulation tests were performed using aluminum 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 neutralization and precipitation.

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 aluminum 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 of aluminum sulfate to treat a mixture of three organic compounds (gallic acid, phenol, and humic substances). The concentrations of organic compounds before and after treatment were determined by measuring the total organic carbon.

Optimization of hybrid system

To investigate the effects of the operational parameters on the treatment efficiency and optimize 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 center 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.

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 modeled 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 a_{ijk} are the interaction effects.

Statistical analysis, model development, and surface response plotting were performed using JMP software. The statistical validation of the generated predictive model was rigorously assessed through the 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 analyzed using the coefficient of determination (R^2) and the adjusted coefficient of determination (R^2adj).

A response optimization was conducted using the desirability function approach. The optimization objective was set to maximize the removal efficiency to determine the optimal combinations of organic matter concentration, coagulant dose, and adsorbent mass.

Agronomic experimental design

Study site and climate

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

Irrigation regimes and water sources

The experiment was conducted over a period of 60 days following a total randomization 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 a pH of 7.26, an electrical conductivity of 730 µS/cm, an organic matter content of 2.1%, and a CEC of 25.43 meq/100g. No fertilizers 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 localized 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, used as the control treatment;
- 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 analyzed according to standard procedures. Soil pH 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 analyzed 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), utilizing atomic absorption spectrometry for exchangeable bases and colorimetry for CEC evaluation.

Statistical analysis

The experimental data were analyzed using analysis of variance (ANOVA) with a significance level of 0.05, considering irrigation water quality as the main factor. Statistical analyses were performed using the DSAASTAT software.

RESULTS AND DISCUSSION

Application of different treatment for the removal of 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 the organic compound with the simplest structure, phenol 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.

Coagulation-flocculation

The efficiency of coagulation is strongly influenced by the molecular structure of the compound to be removed. The results 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; Sun et al., 2025) have shown that iron and aluminum salts do not result in significant removal of simple organic molecules such as salicylic acid, phthalic acid, and phenol.

Furthermore, the graph 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 mg/L 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.

These results lead us to conclude that the hybrid system is effective for the removal of organic compounds with varying chemical structures.

Optimization of hybrid system by full factorial design

The full factorial design methodology was used to optimize 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 JMP8, resulting in high determination coefficients ($R^2 = 0.996$ and adjusted $R^2 = 0.986$), which confirm that the model was successful in correlating the response to the studied parameters.

Student's t-test

The Student's 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 by step-by-step method, Table 2 gives only the 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. It means that the removal of organic matter increases with the activated acorn bark dose. The empirical model for the removal percentage is:

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

Analysis of variance (ANOVA)

Analysis of variance was used to validate the adjusted model. The ANOVA summary is shown in Table 3. The F- -value obtained (213.8367) and the P-value inferior to 0.05 indicate that the model is valid to predict the removal of organic matter with the studied variables.

Optimization design using the desirability function

The desirability function approach is the most current and strongly suggested method for the optimization of one or more responses (Mourabet et al., 2012).

As shown in Fig.5, The combination of organic compound concentrations (PHL 20 mg/L, GA 20 mg/L, and SH 10 mg/L), an aluminum sulfate dose of 50 mg/L, and 1.5 g of activated acorn bark allows for an optimal response of 96.364% with a desirability of 0.946. This high desirability indicates that the activated acorn bark effectively complements the aluminum sulfate, likely through the synergistic effect of charge neutralization and the high adsorption capacity provided by the bark's porous structure and functional groups.

The optimized 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 localized irrigation experiment on green beans to assess its physicochemical quality and preliminary agronomic performance.

Agronomic experimental

Irrigation water assessment

Microbiological quality was not evaluated in this study, as the primary objective was to assess the physicochemical performance and agronomic suitability of the treatment process. According to WHO guidelines (World Health Organization, 2006), when localized 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 favorable range of 6.5-8.5. Additionally, the values of electrical conductivity (EC) and SAR indicate that the waters used for irrigation have a moderate salinity, below the allowable limit.

The nitrate concentration in PCTW at 14.82 mg/L is higher than those in the BTW (7.22 mg/L) and CW (2.35 mg/L). Although all concentrations do not exceed the recommended limit of 30 mg/L, it is evident that biological treatment is the most effective in reducing nitrate levels. Moreover, the 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.

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.

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

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 that reported in the studies of (Rusan et al., 2007; Christou et al., 2014; Sdiri et al., 2023) which have shown that irrigation with treated wastewater does not cause any significant change in soil pH.

Soil electrical conductivity (EC) values show a clear increase in irrigated soils, however, they remain below the upper limit of 4 dS/m for agricultural soils. (M. A. Gharaibeh et al., 2016) attribute this increase in electrical conductivity to the high concentration of total dissolved solids in the treated wastewater. Furthermore, the test site is characterized by high evapotranspiration rates during summer, which can lead to the accumulation of salts from the 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 the 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 obtained in the studies by (M. 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. This represents an important quality parameter for the agricultural reuse of these waters. Nutrient concentrations are directly related to the origin of the wastewater as well as 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 results reported in the study by (Farhadkhani et al., 2018).

The statistical analysis results are summarized in the 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, except for 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. Thus, the wastewater resulted in statistically similar values for pH and cation exchange capacity (CEC), all of which were higher than those associated with EC water. In contrast, the analysis revealed significant differences between the means of the soil parameters: electrical conductivity (EC), calcium carbonate ($CaCO_3$), and organic matter (OM).

Evaluation of plant growth parameters

First, we observed the growth developments of the bean crop at different vegetative stages, illustrated in Fig.8 We noted accelerated growth with treated wastewater, resulting in better plant development compared to conventional water. This finding is consistent with the work of (Paranychianakis et al., 2004) and (Etchebarne et al., 2019) who reported similar results on grapevines. 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.

The results show a clear increase 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 9 below summarizes the statistical parameters obtained from the ANOVA analysis of the plant characteristics.

These results show p-values below the 5% threshold, indicating a statistically significant effect of the factors on the parameters related to crop growth, except for the number of seeds per pod.

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 labeled a, b, and c.

CONCLUSION

This study highlights both the optimization of a hybrid wastewater treatment process and its preliminary physicochemical and short-term agronomic assessment under localized 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 aluminum 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.

Furthermore, 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 agronomic performance of the treated wastewater under localized irrigation conditions. However, long-term field studies, together with microbiological and contaminant assessments, remain necessary before large-scale agricultural reuse can be fully established.

ACKNOWLEDGEMENTS

The authors declare that there are no acknowledgements for this manuscript.

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.

FUNDING STATEMENT

This research received no specific grant from any funding agency in the public, commercial, or not-for-profit sectors.

AI USE STATEMENT: None.

REFERENCES

Alazaiza, M. Y., Alzghoul, T. M., Nassani, D. E., & Bashir, M. J. (2025). Natural coagulants for sustainable wastewater treatment: current global research trends. *Processes 13*(6): 1754. https://doi.org/10.3390/pr13061754

Albalasmeh, A. A., Alghzawi, M. i. Z., Gharaibeh, M. A., & Mohawesh, O. (2022). Assessment of the effect of irrigation with treated wastewater on soil properties and on the performance of infiltration models. *Water14*(9): 1520. https://doi.org/10.3390/w14091520

Christou, A., Eliadou, E., Michael, C., Hapeshi, E., & Fatta-Kassinos, D. (2014). Assessment of long-term wastewater irrigation impacts on the soil geochemical properties and the bioaccumulation of heavy metals to the agricultural products. *Environmental monitoring and assessment 186*(8): 4857-4870. https://doi.org/10.1007/s10661-014-3743-4

Cui, H., Huang, X., Yu, Z., Chen, P., & Cao, X. (2020). Application progress of enhanced coagulation in water treatment. *RSC advances10*(34): 20231-20244. https://doi.org/10.1039/d0ra02979c
Etchebarne, F., Aveni, P., Escudier, J.-L., & Ojeda, H. (2019). *Reuse of treated wastewater in viticulture: Can it be an alternative source of nutrient-rich water?* Paper presented at the BIO Web of Conferences.https://doi.org/10.1051/bioconf/20191201009
Farhadkhani, M., Nikaeen, M., Yadegarfar, G., Hatamzadeh, M., Pourmohammadbagher, H., Sahbaei, Z., & Rahmani, H. R. (2018). Effects of irrigation with secondary treated wastewater on physicochemical and microbial properties of soil and produce safety in a semi-arid area. *Water research144*: 356-364. https://doi.org/10.1016/j.watres.2018.07.047
Galavi, M., Jalali, A., Ramroodi, M., Mousavi, S. R., & Galavi, H. (2010). Effects of treated municipal wastewater on soil chemical properties and heavy metal uptake by sorghum (Sorghum bicolor L.). *Journal of Agricultural Science2*(3): 235. https://doi.org/10.5539/jas.v2n3p235
Gatta, G., Libutti, A., Beneduce, L., Gagliardi, A., Disciglio, G., Lonigro, A., & Tarantino, E. (2016). Reuse of treated municipal wastewater for globe artichoke irrigation: Assessment of effects on morpho-quantitative parameters and microbial safety of yield. *Scientia horticulturae213*: 55-65. https://doi.org/10.1016/j.scienta.2016.10.011
Gharaibeh, M., Eltaif, N., & Al-Abdullah, B. (2007). Impact of field application of treated wastewater on hydraulic properties of vertisols. *Water, Air, and Soil Pollution184*(1): 347-353. https://doi.org/10.1007/s11270-007-9423-z
Gharaibeh, M. A., Ghezzehei, T. A., Albalasmeh, A. A., & Ma'in, Z. A. (2016). Alteration of physical and chemical characteristics of clayey soils by irrigation with treated waste water. *Geoderma276*: 33-40. https://doi.org/10.1016/j.geoderma.2016.04.011
Haripriyan, U., Gopinath, K., Arun, J., & Govarthanan, M. (2022). Bioremediation of organic pollutants: a mini review on current and critical strategies for wastewater treatment. *Archives of Microbiology204*(5): 286. https://doi.org/10.1007/s00203-022-02907-9
Hashem, M. S., & Qi, X. (2021). Treated wastewater irrigation—A review. *Water13*(11): 1527. https://doi.org/10.3390/w13111527
Iqbal, S., Tak, H. I., Inam, A., Inam, A., Sahay, S., & Chalkoo, S. (2015). Comparative effect of wastewater and groundwater irrigation along with nitrogenous fertilizer on growth, photosynthesis and productivity of chilli (Capsicum annuum L.). *Journal of Plant Nutrition38*(7): 1006-1021. https://doi.org/10.1080/01904167.2014.991032
Karnena, M. K., & Saritha, V. (2022). Contemplations and investigations on green coagulants in treatment of surface water: a critical review. *Applied Water Science12*(7): 150. https://doi.org/10.1007/s13201-022-01670-y
Koul, B., Bhat, N., Abubakar, M., Mishra, M., Arukha, A. P., & Yadav, D. (2022). Application of natural coagulants in water treatment: a sustainable alternative to chemicals. *Water14*(22): 3751. https://doi.org/10.3390/w14223751
Lado, M., & Ben-Hur, M. (2010). Effects of irrigation with different effluents on saturated hydraulic conductivity of arid and semiarid soils. *Soil Science Society of America Journal74*(1): 23-32. https://doi.org/10.2136/sssaj2009.0114
Makhadmeh, I. M., Gharaiebeh, S. F., & Albalasmeh, A. A. (2021). Impact of irrigation with treated domestic wastewater on Squash (Cucurbita pepo L.) fruit and seed under semi-arid Conditions. *Horticulturae7*(8): 226. https://doi.org/10.3390/horticulturae7080226
Michałowicz, J., & Duda, W. (2007). Phenols--Sources and Toxicity. *Polish Journal of Environmental Studies16*(3)
Mohd, A. (2022). Presence of phenol in wastewater effluent and its removal: an overview. *International Journal of Environmental Analytical Chemistry102*(6): 1362-1384. https://doi.org/10.1080/03067319.2020.1738412
Mourabet, M., El Rhilassi, A., El Boujaady, H., Bennani-Ziatni, M., El Hamri, R., & Taitai, A. (2012). Removal of fluoride from aqueous solution by adsorption on Apatitic tricalcium phosphate

using Box–Behnken design and desirability function. *Applied Surface Science258*(10): 4402-4410. 10.1016/j.apsusc.2011.12.125

Official Journal of the Algerian Republic. (2012). Specifications of treated wastewater used for irrigation purposes. (41). https://www.joradp.dz/ftp/jo-francais/2012/f2012041.pdf [In French]

Paranychianakis, N. V., Aggelides, S., & Angelakis, A. N. (2004). Influence of rootstock, irrigation level and recycled water on growth and yield of Soultanina grapevines. *Agricultural water management69*(1): 13-27. https://doi.org/10.1016/j.agwat.2004.03.012

Pascual, I., Azcona, I., Morales, F., Aguirreolea, J., & Sánchez-Díaz, M. (2009). Growth, yield and physiology of Verticillium-inoculated pepper plants treated with ATAD and composted sewage sludge. *Plant and soil319*(1): 291-306. https://doi.org/10.1007/s11104-008-9870-3

Rusan, M. J. M., Hinnawi, S., & Rousan, L. (2007). Long term effect of wastewater irrigation of forage crops on soil and plant quality parameters. *Desalination215*(1-3): 143-152. https://doi.org/10.1016/j.desal.2006.10.032

Sdiri, W., AlSalem, H. S., Al-Goul, S. T., Binkadem, M. S., & Ben Mansour, H. (2023). Assessing the effects of treated wastewater irrigation on soil physico-chemical properties. *Sustainability15*(7): 5793. https://doi.org/10.3390/su15075793

Seleiman, M. F., Al-Suhaibani, N., El-Hendawy, S., Abdella, K., Alotaibi, M., & Alderfasi, A. (2021). Impacts of long-and short-term of irrigation with treated wastewater and synthetic fertilizers on the growth, biomass, heavy metal content, and energy traits of three potential bioenergy crops in arid regions. *Energies14*(11): 3037. https://doi.org/10.3390/en14113037

Sun, H., Lin, H., Shi, J., & Kuang, Z. (2025). Insights into the Interaction Between Coagulants and Natural Organic Matter (NOM) in Drinking Water Treatment: A Review of Floc Formation and Floc Aging. *Water17*(21): 3124. https://doi.org/10.3390/w17213124

Verma, A. K., Dash, R. R., & Bhunia, P. (2012). A review on chemical coagulation/flocculation technologies for removal of colour from textile wastewaters. *Journal of environmental management93*(1): 154-168. https://doi.org/10.1016/j.jenvman.2011.09.012

World Health Organization. (2006). *Guidelines for the safe use of wastewater, excreta and greywater* (Vol. 4): World Health Organization.

Zhao, H., Wang, L., Hanigan, D., Westerhoff, P., & Ni, J. (2016). Novel ion-exchange coagulants remove more low molecular weight organics than traditional coagulants. *Environmental science & technology50*(7): 3897-3904. https://doi.org/10.1021/acs.est.6b00635

Zoumpouli, G. A., Herron, D., Thornton, A., Jefferson, B., & Campo, P. (2023). The role of coagulation on the fate of PFAS, brominated flame retardants and other trace contaminants in tertiary wastewater treatment for phosphorus control. *Science of the Total Environment887*: 163982. https://doi.org/10.1016/j.scitotenv.2023.163982

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

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	<.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	<.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*

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			

520 Table 4. Physicochemical characteristics of municipal wastewater before and after treatment

Parameters	Unit	Before	After
BOD_5	mg L^{-1}	180.00	28.13
COD	mg L^{-1}	420.92	44.41
Iron	mg L^{-1}	0.167	0.092
Cadmium	mg L^{-1}	0	0
Nickel	mg L^{-1}	0.015	0.004
Zinc	mg L^{-1}	0	0
Manganese	mg L^{-1}	0.170	0.03
Copper	mg L^{-1}	0	0
Lead	mg L^{-1}	0	0
Chromium	mg L^{-1}	0	0

Table 5. Physico-chemical parameters of irrigation waters [CW: Conventional water, BTW: Biologically treated wastewater, PCTW: Physicochemically treated wastewater, 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

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

Parametres		Before irrigation	After irrigation		
			CW	BTW	PCTW
Particle size distribution, %	Clay	20.13	20.23	20.69	20.80
	Fine silts	36.29	38.16	38.61	39.03
	Coarse silts	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

Table 7. ANOVA analysis of soil physico-chemical parameters (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)

Parametres	*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	2.749E-06**	952.00^{c}	988.33^{a}	963.67^{b}
$CaCO_3$, %	229.40	2.152E-06**	3.77^{c}	3.89^{b}	4.12^{a}
OM, %	5607.60	1.529E-10**	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}

Table 8. Plant growth parameters (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. ANOVA analysis of plant growth parameters 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.

Parametres	*F*-ration	p-value	Means		
			CW	BTW	PCTW
Height, cm	416.66	3.653E-07**	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	3.853E-07**	2.93[c]	3.40[b]	3.48[a]

Figure Captions:

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

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 3. Organic matter removal efficiency (%) by the coagulation-flocculation process using aluminum sulfate and the hybrid system combining aluminum sulfate with acorn bark.

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

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

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

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

Figure 8. Growth stages of green bean.

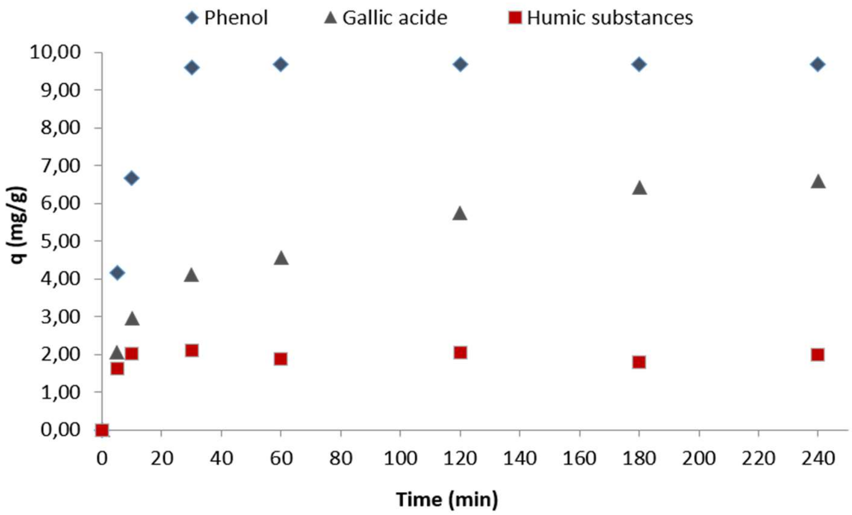

Figure 1. Effect of pollutant molecular structure on the adsorption capacity (q) over time for phenol, gallic acid, and humic substances onto activated acorn bark.

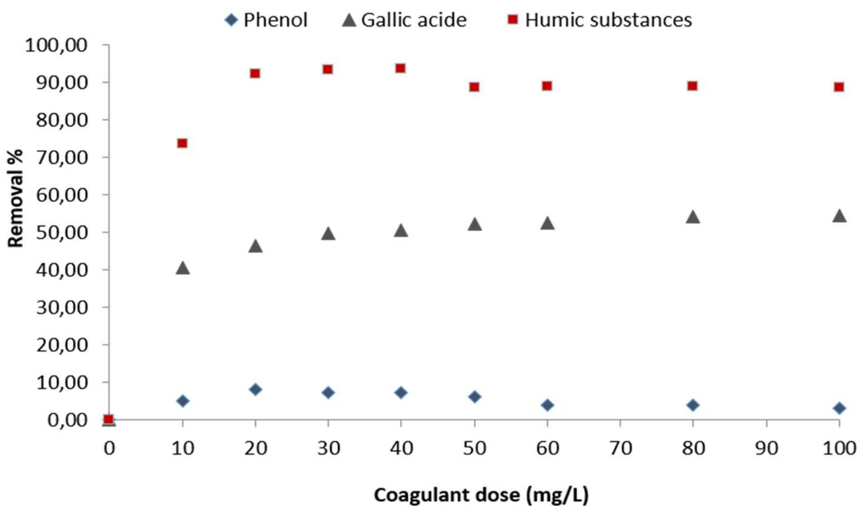

Figure 2. Effect of pollutant molecular structure on the coagulation efficiency (Removal %) of phenol, gallic acid, and humic substances as a function of coagulant dose.

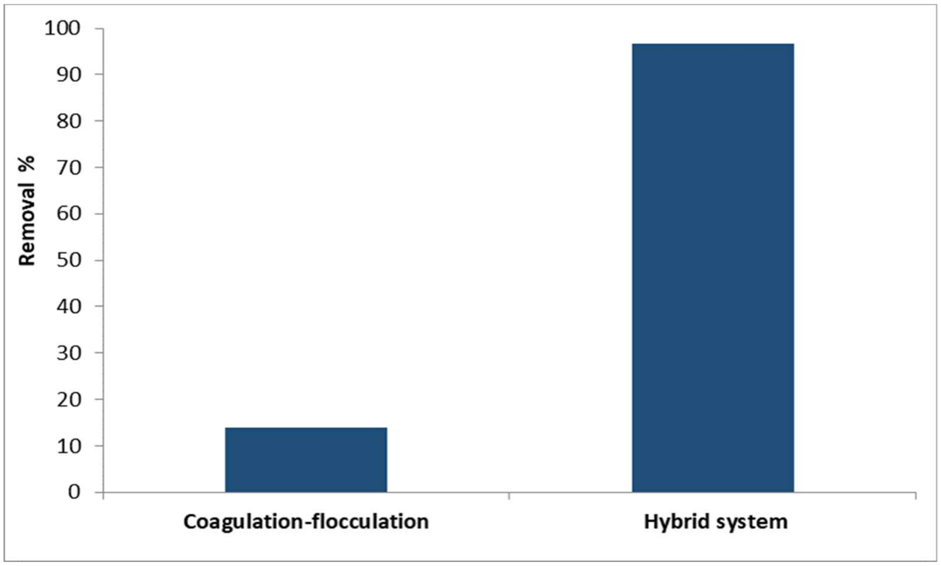

Figure 3. Organic matter removal efficiency (%) by the coagulation-flocculation process using aluminum sulfate and the hybrid system combining aluminum sulfate with acorn bark.

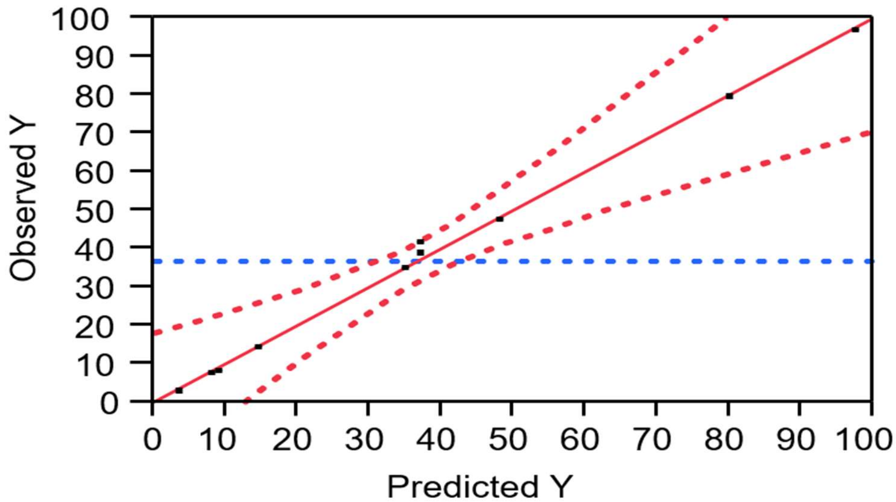

Figure 4. Observed versus predicted values for organic matter removal efficiency (%) using the Full Factorial Design model for the optimization of the treatment process.

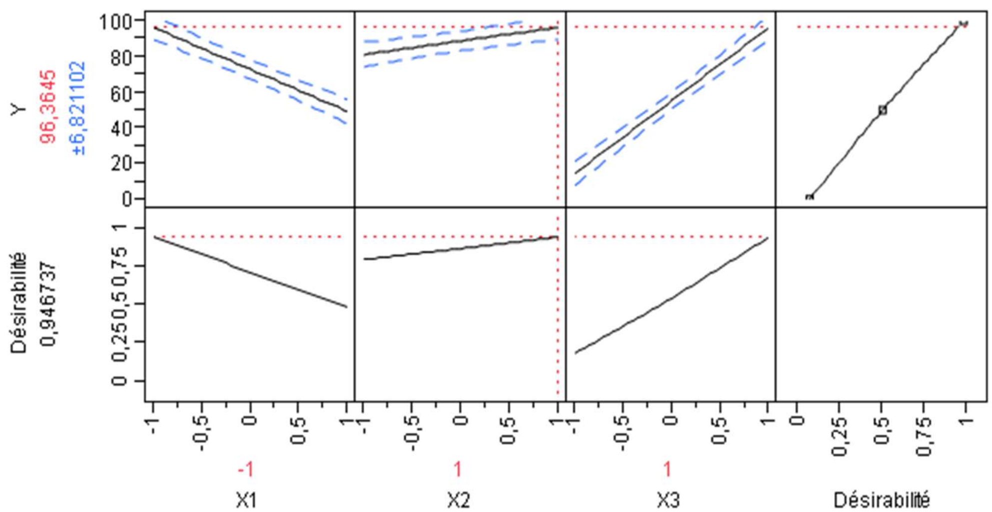

Figure 5. Desirability function for removal of organic matter using the Full Factorial Design model.

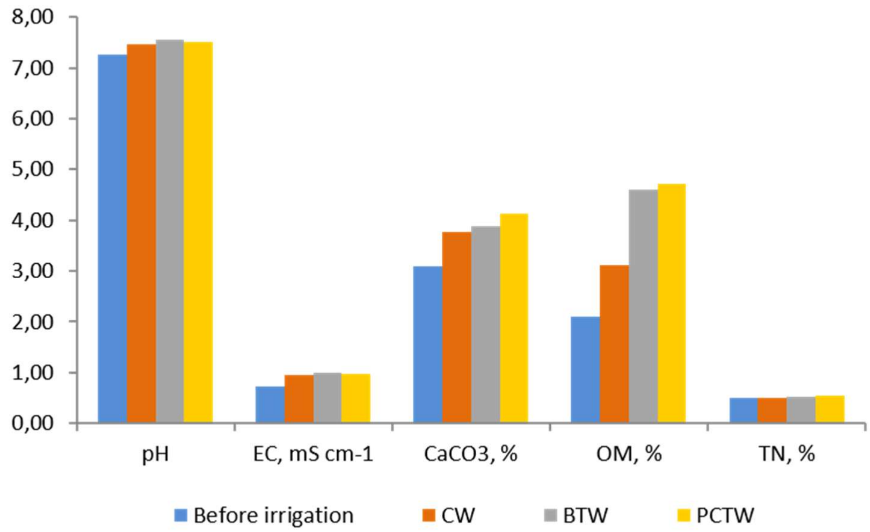

Figure 6. Chemical properties of soils before irrigation and after irrigation with conventional water (CW), biologically treated wastewater (BTW), and physicochemically treated wastewater (PCTW).

https://doi.org/10.5513/NPFB3849

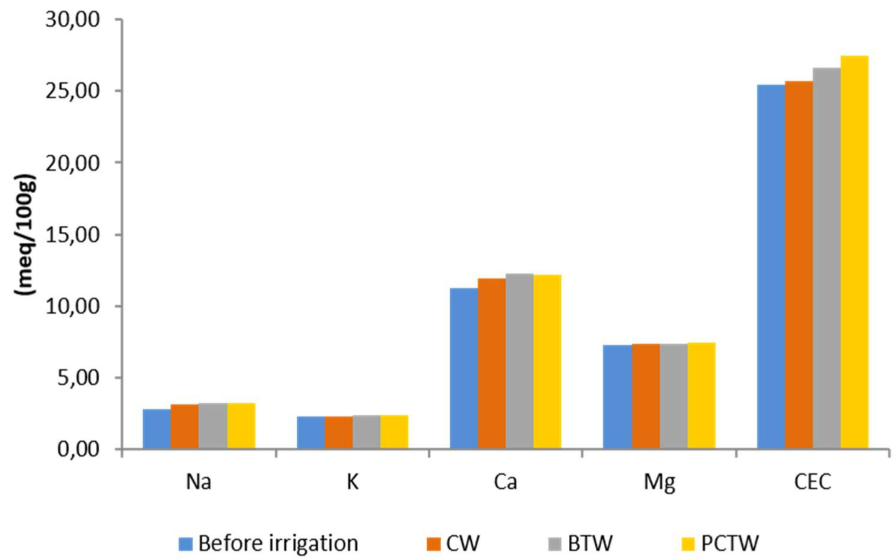

Figure 7. Soil exchange complex elements before irrigation and after irrigation with conventional water (CW), biologically treated wastewater (BTW), and physicochemically treated wastewater (PCTW).

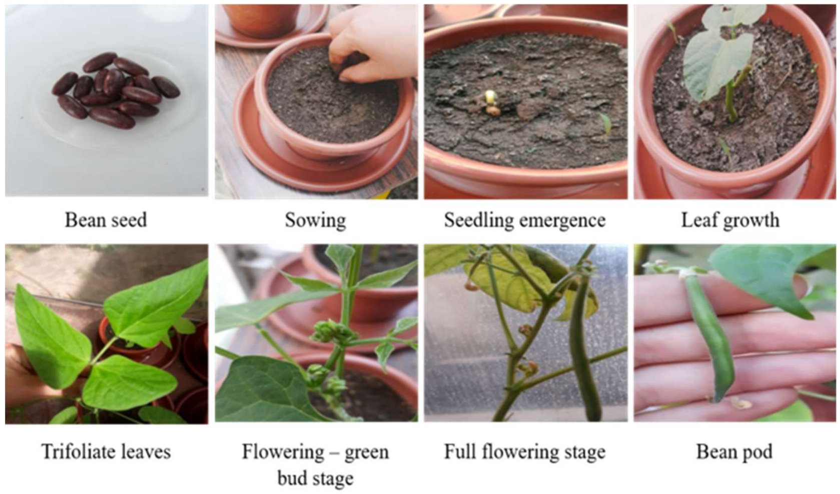

Fig.8. Growth stages of green bean.