

Hybrid Leachate Treatment with Date Pit Tannins and Persulfate AOP: A Statistical Approach

Shabib Al Rashdi

Center of Excellence in Environmental Science & Technology (CEEST), National University of Science and Technology, Oman
shabibalrashdi@nu.edu.om

Nageswara Rao Lakkimsetty

Department of Chemical and Petroleum Engineering, School of Engineering & Computing, American University of Ras Al Khaimah, Ras al Khaimah, UAE
lakkimsetty.rao@aurak.ac.ae (corresponding author)

Hala Ali Al Zaabi

Department of Mechanical and Industrial Engineering, National University of Science and Technology, Oman
hala200489@nu.edu.om

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ABSTRACT

The treatment of stabilized landfill leachate using a hybrid approach combining coagulation-flocculation and Persulfate Advanced Oxidation Process (P-AOP) is studied in this article. Catechin tannins were extracted from domestically sourced Omani date pits (Fardh and Khalas) using Soxhlet extraction with 50% ethanol and were subsequently characterized via Fourier Transform Infrared (FTIR) and ferric tests. Leachate samples collected from the Barka Engineered Landfill underwent treatment under varying operational parameters. Coagulation-flocculation was optimized at 70 mg/L tannin dosage, pH = 5.8, and 60 minute reaction time, while P-AOP was performed at a 1:6 sodium persulfate-to-zinc sulfate heptahydrate ratio, pH = 11, 200 RPM, for 140 min. Spectrophotometric analysis confirmed removals of 74% COD, 60% ammonia, and 96% color, with a 12,433% increase in TSS. Design Expert software was utilized to develop a statistical model and optimize treatment conditions. The efficacy of this hybrid method was highlighted by the obtained results, exhibiting the potential of agricultural waste-derived tannins in sustainable leachate management, aligned with Oman Vision 2040 and the UN Sustainable Development Goals.

Keywords-hybrid treatment; Persulfate Advanced Oxidation (P-AOP); date pit tannins; landfill leachate; sustainable waste management

I. INTRODUCTION

Sanitary landfilling remains globally the most widely implemented method for municipal solid waste disposal. Conversely, it generates Landfill Leachate (LL), an effluent containing recalcitrant organic matter, ammonia, and heavy metals and is considered a severe environmental threat if inadequately treated. The characteristics of LL vary with landfill age, precipitation, and biochemical conditions. Stabilized leachate common in mature landfills—exhibits elevated ammonia levels, Chemical Oxygen Demand (COD) and low biodegradability [1]. A substantial quantity of solid waste is produced annually in the Sultanate of Oman, with projections revealing a continued upward trend. This escalation has made

the management of stabilized landfill leachate an emerging national priority, particularly in the context of limited access to advanced treatment infrastructure, dependence on imported treatment chemicals, and the widespread use of sludge-generating biological treatment processes.

Coagulation-Flocculation (C-F) and Advanced Oxidation Processes (AOPs) are traditional physicochemical methods employed to treat LL. Conventionally, metal-based coagulants and oxidants like ozone or hydrogen peroxide are employed, often resulting in operational complexities, increased sludge volumes, and secondary pollution [2, 3]. Recent studies have addressed these limitations by highlighting the potential of natural, plant-derived polyphenolics as viable substitutes in

wastewater treatment [4, 5] due to their biodegradability, high phenolic content, and flocculant activity [6, 7].

This study introduces a hybrid treatment system that combines C-F using catechin tannins extracted from Omani date pits as an abundant, locally sourced, agricultural byproduct with Persulfate Advanced Oxidation Process (P-AOP) as an alternate and sustainable methodology for the treatment of stabilized LL. Integrating these two processes leverages the strong flocculating capabilities of polyphenolic compounds and the high oxidative potential of persulfate radicals activated by zinc sulfate under alkaline conditions. Bio-based tannins and zinc-activated P-AOP aid the treatment system in enhancing the removal of critical pollutant factors such as COD, ammonia, color, and Total Suspended Solids (TSS), while reducing the chemical footprint and sludge generation. Furthermore, the utilization of date pits offers a low-cost resource for environmental remediation in resource-limited countries, promoting circular economy principles within the Sultanate and possibly other arid climates [8].

The dual process executed was optimized using the Response Surface Methodology (RSM) via the Design Expert software. This was done to investigate the interaction between key operational reaction parameters including pH, tannin dosage, and reaction time. The statistical model was validated with independent experimental. The proposed system supports Oman Vision 2040 objectives and United Nations Sustainable Development Goals (SDGs 6, 9, and 12).

II. MATERIALS AND METHODS

The treatment of stabilized LL conducted in this study followed a multi-phase experimental approach to assess and optimize the hybrid system and its effectiveness. Raw leachate samples were collected from Barka Engineered Landfill, Oman. Sampling was conducted using sterilized 1000 mL polypropylene bottles, under the supervision of landfill engineers, adhering to HSE protocols and utilizing appropriate Personal Protective Equipment (PPE). The samples were stored and transported to the Waste-to-Energy laboratory at the National University of Science and Technology in insulated coolers at $\pm 1^\circ\text{C}$ preserving sample integrity. The leachate samples were then characterized in accordance with APHA and USEPA protocols, measuring key parameters including COD, ammonia, TSS, and color.

Catechin tannins were extracted from the Fardh and Khalas date pits, located in the governorate of Ad Dakhiliyah. The samples were washed, dried for 72 h, dried in an oven at 105°C for 20 h, and ground to a 1 mm particle size. The extraction was done using Soxhlet with 50 % ethanol (250 mL ethanol: 250 mL water) at a temperature of 80°C and running time of 19.5 hr/batch. The evaporated solution, crystallized, ground, and then stored, was used to carry out experiments of C-F. The initial specification of tannins was done through the phytochemical ferric test wherein 1 mL of extract was combined with 2-3 drops of 5% ferric chloride to produce an instant color shift of brown-to-dark green/blue-black, which suggested the presence of hydrolysable tannins. Additional characterization through Fourier Transform Infrared (FTIR) proved the existence of functional groups of catechin tannins. The Soxhlet apparatus

made it possible to have a solvent cycling to recover polyphenol efficiently [9].

C-F were designed using Design-Expert ® software in the scope of a Central Composite Design (CCD) for the three independent variables (tannin dose ranging from 70 to 110mg/L, pH from 4 to 8, and reaction time from 60 to 180 min). The pH of the raw leachate samples was adjusted with 5 M sulfuric acid (H_2SO_4) or sodium hydroxide (NaOH). For each run, coagulant mixing was set rapidly at 150 RPM for 2 min, followed by flocculation at 60 RPM for 30 min and sedimentation for 60 min. The samples were filtered through 125 mm Whatman filter paper, transferred to sterile 50 mL conical tubes, and stored at 2°C for further treatment [10-11]. Table I shows the design matrix of the experimental runs generated via CCD. The independent variables are the tannin dosage (mg/L), pH, and reaction time (min). A total of 18 experimental runs were designed.

TABLE I. DESIGN MATRIX OF EXPERIMENTAL RUNS GENERATED VIA CCD

Run	Factor 1	Factor 2	Factor 3
	A: Tannin dosage (mg/L)	B: pH	C: Time (min)
1	110	4	60
2	110	8	180
3	90	8	120
4	70	8	180
5	90	6	120
6	90	4	120
7	90	6	180
8	70	6	120
9	110	4	180
10	90	6	60
11	70	4	180
12	110	8	60
13	70	4	60
14	90	6	120
15	70	8	60
16	110	6	120
17	90	6	120
18	90	6	120

Each of the 18 LL samples was exposed to persulfate advanced oxidation where 1 g sodium persulfate ($\text{Na}_2\text{S}_2\text{O}_8$) was added in 250 mL of leachate and activated by the addition of 6 g zinc sulfate heptahydrate ($\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$, 1:6 molar ratio). The mixture was stirred at 200 RPM for 140 min and left to settle for 60 min to maximize the degradation of the recalcitrant compounds. Response Surface Methodology (RSM) was employed to analyze all 18 runs. Comparing the experimental results with expectations, the models were statistically confirmed, which allowed us to optimize the hybrid system and evaluate its reliability in practice.

III. RESULTS AND DISCUSSION

The raw leachate of Barka Engineered Landfill was characterized by dark brown color (33,600 Pt-Co), high ammonia (4,170 mg/L), COD (1,280 mg/L), and TSS (260 mg/L), showing exceedance of Omani discharge standards, and indicating a strongly stabilized recalcitrant leachate. To solve this, a hybrid method using tannin-based C-F was used along

with P-AOP. The modeling and optimization of tannin dosage, pH, and reaction time using Design-Expert V12.1 with CCD was used to determine the responses, i.e. the percentage removal of COD, ammonia, and color, and the TSS increase indicating the formation of a floc. The removal efficiency and the increase in TSS outcome of the 18 experimental runs is summarized in Table II. The optimum treatment performance occurred at 70 mg/L tannin, pH = 5.8, and 60 min, with 96% color removal, 74% COD reduction, 60% ammonia removal and 12.433% TSS increase, which means that the floc was formed successfully. The increased TSS indicates that dissolved and colloidal pollutants are flocculated to allow easy sedimentation and to ensure the hybrid system has effective results. Run 13, which was performed at the lowest pH and tannin dose (70 mg/L at pH 4), removed 96% of the color, 82% of the COD, and 77% of the ammonia, with a moderate TSS increase of only 4.238%. In comparison, run 3 (90 mg/L tannin, pH 8, 120 min) showed 94% color and 63% COD removal and an extremely high increase of TSS 28.789%, indicating very low floc stability at higher pH and dosage combinations [12].

TABLE II. EXPERIMENTAL RESULT SUMMARY

Run	1	2	3	4
	Color removal	COD removal	TSS increase	NH ₃ removal
	% Pt-Co	% ppm	% ppm	% mg/L
1	95.22	80.28	5103.07	76.07
2	94.90	61.85	28038.46	48.92
3	94.10	62.69	28789.23	45.80
4	94.80	63.52	27921.54	47.48
5	93.32	61.48	20166.15	43.88
6	92.95	79.33	7027.69	62.59
7	94.25	64.91	16507.69	49.64
8	95.20	71.49	15798.46	50.12
9	94.55	79.93	6533.85	67.15
10	95.82	64.15	18046.15	46.76
11	94.43	80.64	6355.38	66.43
12	96.98	60.12	23390.77	51.08
13	95.89	81.71	4238.46	76.76
14	95.71	70.77	17380	48.20
15	97.01	71.30	21156.92	50.84
16	94.45	68.22	13173.84	47.72
17	95.38	70.16	16566.15	46.52
18	96.04	78.45	8223.08	72.18

As shown in Figure 1, the tube on the right represents the raw stabilized landfill leachate after C-F using catechin tannins, characterized by a dark brown color resulting from residual organic matter. In contrast, the tube on the left depicts the same sample following treatment with the P-AOP, displaying a markedly lighter yellow-orange hue, indicating substantial pollutant degradation.

Figure 2 shows the deposited flocs retained on a filter paper following tannin-based C-F of the stabilized LL, indicating effective particle aggregation and sedimentation. One of the main innovations of the hybrid treatment system is the extraction of the catechin tannin in Fardh and Khalas date pits. The agricultural wastes are underutilized and offer a sustainable, biodegradable substitute for traditional ferric and aluminum

salts. A combination of plant-based coagulants with persulfate advanced oxidation caused the high removal of pollutants, low consumption of chemicals, and low production of sludge. FTIR analysis (Figures 3-4) confirmed the composition and purity of the extracted tannins. Both Fardh & Khalas extracts contained a broad O-H stretch at 3300 cm^{-1} , indicating the presence of hydrogen-bonded hydroxyl groups in polyphenols, C=C aromatic stretch at 1600 cm^{-1} , indicating the presence of benzene rings in flavonoids, and C-H bending vibrations at 1400 cm^{-1} that are characteristic of tannin-like catechins [13-14].



Fig. 1. Leachate color before and after treatment.



Fig. 2. Floc formation observed after C-F.

Although the spectral profiles of the extracts aligned well with those of high-purity catechin, slight differences were noted in peak intensity & width. The O-H stretches in the extracted samples, while present at a similar wavenumber ($\sim 3300\text{ cm}^{-1}$), showed slightly broader and less intense signals compared to the reference.

These variations may result from the extraction matrix, source material heterogeneity, or the duration and solvent conditions used in the Soxhlet process. Such discrepancies suggest opportunities to refine the extraction protocol to improve the purity, yield, and functional consistency of the tannin product [15-16].

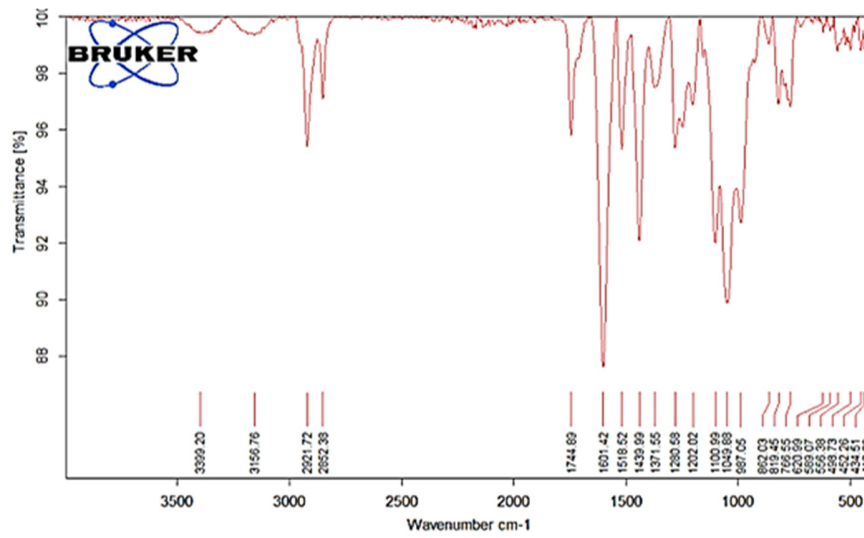


Fig. 3. FTIR of catechin tannins extracted from Khalas Date pits.

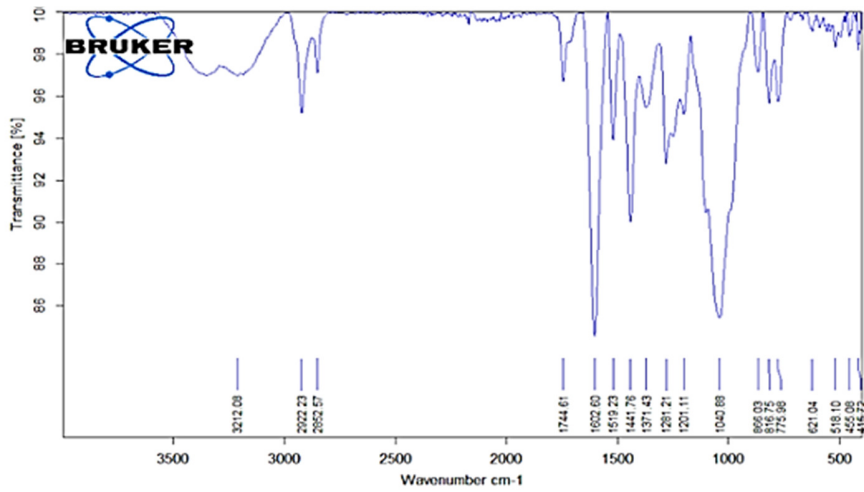


Fig. 4. FTIR of catechin tannins extracted from Fardh Date pits.

Design-Expert® Software

Factor Coding: Actual

Colour (Pt-Co)

● Design points above predicted value

○ Design points below predicted value

93 97

X1 = C: Time

X2 = A: Tannin Dosage

Actual Factor

B: pH = 8

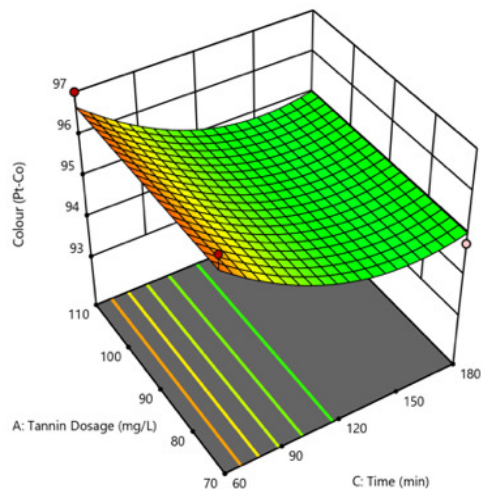


Fig. 5. RSP for color removal efficiency.

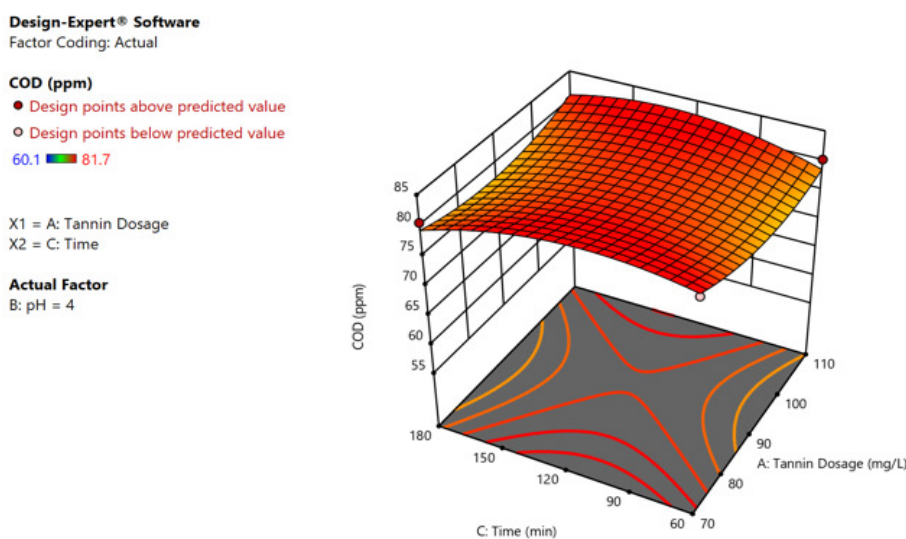


Fig. 6. RSP for COD removal efficiency.

Table III shows the comparison of FTIR peaks for high-purity catechin and tannin extracts from Fardh and Khalas date pits with standard high-purity catechin [7]. The similarity in peak positions confirms the successful extraction of polyphenolic structures. FTIR analysis indicates that major functional groups such as O-H, C=C, and C-H are maintained in the tannin extracts, confirming the suitability of tannin extracts as natural coagulants. The extraction parameters, such as temperature, solvent polarity, and time could be further optimized to improve performance. The appearance of dark complexes on agitating ferric chloride with Khalas and Fardh extracts (greenish black and olive green, respectively) proves the existence of the catechol-type polyphenols, which is affirmed by the phytochemical ferric test.

TABLE III. FTIR PEAK COMPARISON OF HIGH-PURITY CATECHIN AND OMANI DATE PIT TANNIN EXTRACTS

Functional Group	Peak (cm ⁻¹) – high-purity catechin [7]	Fardh extract	Khalas extract
O – H stretch (hydroxyl)	3317.90	~3300	~3300
C = C stretch (aromatic)	~1600	~1600	~1600
C – H bending (aliphatic)	~1400	~1400	~1400

Figure 5 illustrates a 3-D Response Surface Plot (RSP) of the impact of the dosage of tannin (70–110 mg/L) and reaction time (60–180 min) on color removal at pH 8 and how these variables affect residual color in Pt-Co units. The surface reveals a concave profile, with the lowest color values (highest removal) concentrated in the region of lower dosage and shorter time, suggesting a region of optimal efficiency. Overall, the Omani date pit catechin tannins present a promising eco-friendly and effective method of LL treatment, with high coagulation efficiency, spectral similarities to the standard, and compatibility with the principles of the circular economy, which can be used in a wider range of applications in sustainable water treatment.

The coded equation possesses a positive pH coefficient (B), meaning that the removal of color decreases with an increase in pH, which is in line with the tannins of polyphenolic nature having an optimum working range in the mildly acidic-neutral

range. The time (C) linear coefficient is negative and C² was positive, which means reducing returns after an optimal time (approx. 120 min). This trend is confirmed by the actual factor equation, which indicates that as time goes by, the removal of color rises to some point, and the extra benefit of even more reaction time reduces, which agrees with the floc formation and settling kinetics [17]. The coded regression equation and the actual term model are described by (1) and (2), respectively:

$$\text{Color} = 94.64 + 0.4800 B - 0.8 C + 0.7425 C^2 \quad (1)$$

$$\text{Color} = 97.767 + 0.540 \text{ pH} - 0.0628 \text{ Time} + 0.000206 \text{ Time}^2 \quad (2)$$

The system's efficiency is reflected in the input of minimal reagent, where the predicted minimum color concentration in the treated leachate occurs at lower tannin dosages (~70 mg/L) and shorter reaction times (~60–90 min). This is considered an important finding for a sustainable process design, where the excessive use of the coagulant or longer contact time beyond the optimal region have no significant effect on color removal and may lead to process inefficiencies or increased sludge volume. The plot also confirms good model fitness, as design points cluster closely around the surface with minimal deviation, indicating accurate predictions by the regression model.

Figure 6 shows a 3D RSP of the influence of tannin dosage (70–110 mg/L) and reaction time (60–180 min) on COD removal at a constant pH of 4. The surface demonstrates a moderately curved profile, indicating that COD removal efficiency improves with both increased time and dosage up to a threshold, beyond which further increases yield diminishing returns. The coded regression model is:

$$\text{COD} = 68.8 - 1.83 A - 8.24 B - 0.68 C - 1.35 AB + 1.28 AC - 0.575 BC + 2.48 A^2 + 3.63 B^2 - 2.87 C^2 \quad (3)$$

The actual term model is:

$$\begin{aligned} \text{COD(ppm)} = & 164.39268 \\ & -1.13346 \text{ TanninDosage} \\ & -11.40393 \text{ pH} + 0.112982 \text{ Time} \\ & -0.033750 \text{ TanninDosage} \times \text{pH} \\ & +0.001062 \text{ TanninDosage} \times \text{Time} \\ & -0.004792 \text{ pH} \times \text{Time} \\ & +0.006205 \text{ TanninDosage}^2 \\ & +0.908036 \text{ pH}^2 - 0.000797 \text{ Time}^2 \end{aligned} \quad (4)$$

The equations demonstrate that tannin dosage and pH are highly linear, where an increase in the COD removal results in a decrease in pH and an increase in tannin dosage which demonstrates an increase in the radical activity as well as the coagulant activity. The dosage (A^2) and pH (B^2) positive quadratic points depict that the COD removal advances to an optimum after which the efficiency ceases or slightly decreases. The negative quadratic term for time (C^2) proposes that exceptionally long reaction times offer little additional COD removal, likely due to the reduction of oxidizable organics or radical recombination. The actual term model reflects the complexity of real-world processes in terms of interaction (e.g., AB). The negative AB coefficient recommends that high tannin dosage linked with low pH can on occasion reduce performance, potentially due to the over-coagulation or floc re-stabilization. RSM analysis establishes that the removal of COD tends to increase with dosage and reaction time, however the plot becomes a straight line in the upper-right quadrant, which shows diminishing returns and high costs of operation at excessive treatment. The similarity of design points to the fitted surface and the correlation of predicted and experimental data testify to the strength of the model [18]. As these results show, COD removal is a nonlinear process that is extremely sensitive to the interaction of critical operating parameters that were successfully optimized with the use of RSM.

Figure 7 shows a 3D RSP of the influence of tannin dosage (70–110 mg/L) and pH (4–8) on ammonia concentration at a fixed reaction time of 60 min. In contrast to the non-planar, curved response of COD and color plots, this surface is flat and straight, indicating that there is a linear relationship between response and singled-out variables. The coded factor equation is described by (5) and the actual factor equation by (6):

$$\text{NH}_3 = 55.47 - 0.06 A - 10.46 B - 2.2 C \quad (5)$$

$$\begin{aligned} \text{NH}_3 = & 91.52 - 0.003 \text{ TanninDosage} \\ & - 5.23 \text{ pH} - 0.0367 \text{ Time} \end{aligned} \quad (6)$$

Both equations show that pH has the most significant effect, the negative coefficient in both equations indicates that ammonia removal efficiency improves notably as pH decreases. Furthermore, it is evident (by their smaller coefficient) that time and tannin dosage have less contribution to ammonia removal, suggesting that lower pH is the primary driver for ammonia reduction. The straight-line trend in the RSP shows that the system behaves in a linear way. Since there are no curved

(quadratic) effects or combined (interaction) effects between variables, the relationship is simple and direct. This corresponds the smooth and simple shape seen in the response surface. This model proves a trend consistent with the chemical behavior of ammonia in aqueous systems, where at lower acidic pH conditions, NH_3 is to form ammonium ions (NH_4^+), which are readily removed via the C-F processes. In contrast, at higher pH values, the presence of free NH_3 increases, making removal less efficient due to its volatility and poor reactivity with sulfate radicals or tannins.

The response plot demonstrates that the gradient to be followed by increasing tannin dosage and decreasing pH is gradual, and therefore, ammonia removal is only possible under acidic conditions, whereas ammonium capture cannot happen under tannin dosage conditions alone. The lack of curvature or saturation indicates that further addition of dosage or reaction time would further reduce ammonia, yielding less returns as the process nears a plateau in the experimental range. In general, these findings support the fact that acidification is the primary driving factor, and tannin dosage and contact time are secondary supportive factors. The design points that overlap with the regression surface are a sign of the high predictive reliability of the model under the studied conditions [19].

Figure 8 shows the 3D RSP of the effect of tannin dosage (70–110 mg/L) and reaction time (60–180 min) on TSS at a constant pH of 8. The elevation of TSS serves as an indirect measure of effective floc development, indicating the grouping of colloidal and dissolved particles into measurable suspended solids. The coded factor equation is described by (7) and the actual factor equation by (8):

$$\begin{aligned} \text{TSS} = & 15800.95 + 76.92A \\ & + 10003.84B + 1342.14C \end{aligned} \quad (7)$$

$$\begin{aligned} \text{TSS(ppm)} = & -17240.99000 \\ & + 3.84600 \text{ TanninDosage} \\ & + 5001.92000 \text{ pH} + 22.369000 \text{ Time} \end{aligned} \quad (8)$$

Tannin dosage is the main influence on TSS increase, as indicated by the high linear coefficients. Higher concentrations of coagulant promote the high extent of particle aggregation, indicating that slightly alkaline conditions (pH 8) will lead to an increase in floc. A smaller but positive effect has been found on reaction time, showing cumulative and time-dependent floc formation. The response surface indicates a linear, stepwise rise in TSS along the axes, and no leveling off, meaning that there is no tannin saturation or destabilization of a floc in the conditions in which the experiment was carried out. The significant TSS rise (4238.5 - 28789.2 ppm) indicates that dissolved and colloidal material is converted to flocs that can be filtered, something that is beneficial in the downstream solid-liquid separation. Nevertheless, the unending increase in TSS highlights a trade-off, where beyond a certain point a surplus coagulant may create a greater sludge thereby driving up the disposal needs [20]. Overall, the results prove the usefulness of tannins in strong flocculation, the predictive capacity of the model, and offer an empirical foundation of dosage and contact time optimization in full-scale processes to balance the treatment quality and the sludge control [21].

Design-Expert® Software
Factor Coding: Actual

NH3 (mg/L)

- Design points above predicted value
 - Design points below predicted value
- 43.9 76.8

X1 = A: Tannin Dosage
X2 = B: pH

Actual Factor
C: Time = 60

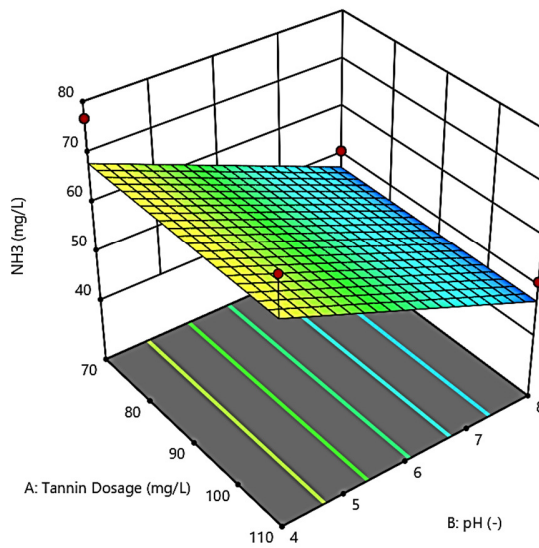


Fig. 7. RSP for NH₃ removal efficiency.

Design-Expert® Software
Factor Coding: Actual

TSS (ppm)

- Design points above predicted value
 - Design points below predicted value
- 4238.5 28789.2

X1 = A: Tannin Dosage
X2 = C: Time

Actual Factor
B: pH = 8

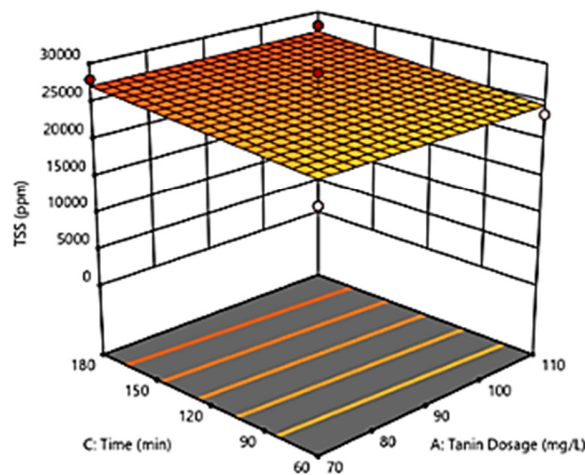


Fig. 8. RSP of TSS for flocc formation.

Table V shows the RSM-determined conditions of optimum treatment performance of the combined catechin tannin-based C-F and P-AOP system. The statistical soundness and predictive capability of the system were proved analyzing the regression models and the response surface, which identified the pH and tannin dosage as the most significant factors. The fact that locally found date pits were utilized decreases the level of chemical addiction and validates circular economy principles, with local agricultural waste being utilized. These findings reveal that the system can be used in practical applications for the treatment of leachate in real-world conditions, particularly in arid areas with minimal access to traditional chemicals [21].

TABLE IV. OPTIMIZED OPERATIONAL PARAMETERS FOR HYBRID LANDFILL LEACHATE TREATMENT

Parameter	Optimized value
Tannin dosage	70 mg/L
pH	5.8
Reaction time	60 mins
ZnSO ₄ · 7H ₂ O	6 g
Na ₂ S ₂ O ₈	1 g
Agitation speed (C-F)	150 / 60 RPM
Agitation speed (P-AOP)	200 RPM
Temperature	Room temperature

IV. CONCLUSIONS

The study shows a successful hybrid treatment of stabilized Landfill Leachate (LL) through the combination of a bio-based Coagulation-Flocculation (C-F) system with catechin tannins in date pits in Oman and Persulfate Advanced Oxidation Process (P-AOP). The process demonstrated a good capability to remove color (96%), reduce COD (74%), eliminate ammonia (60%), and increase TSS (12.433%) under optimized conditions (70 mg/L tannin concentration, pH equal to 5.8, and 60 min reaction time) which implies that the process could form an effective floc. RSM and the regression analysis were utilized to prove the robustness of the model and defined the pH and tannin dosage as the most significant factors. The treatment provides a low-cost, eco-friendly solution to the traditional coagulants, lessens the utilization of chemicals, minimizes sludge, and offers a degree of flexibility in operation to arid regions. These findings highlight the viability of the system in practice and environment and the prospects of its full-scale utilization in the future.

DECLARATION OF COMPETING INTERESTS

The authors declare no competing interests.

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DATA AVAILABILITY

Data will be made available from the corresponding author upon request.

AI USE AND DECLARATION OF GENERATIVE AI USE

Grammarly and Quill Bot tools were used to polish the paper and ensure it was grammatically correct. The authors reviewed and edited the outcome and take full responsibility for the content of the published article.

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