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Comparative evaluation of an organic and an inorganic coagulant in the clarification of vinasse: dose optimization for BOD_5 and turbidity reduction

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Abstract: Environmental pollution caused by vinasse, a byproduct of alcohol distillation characterized by high organic load and turbidity, presents a significant challenge to the sustainability of distilleries and the quality of aquatic ecosystems. This study aimed to determine the optimal doses of organic and inorganic coagulants for reducing Biochemical Oxygen Demand (BOD_5) and turbidity in vinasse through clarification. The Response Surface Methodology (RSM) was used to evaluate combinations of coagulants and flocculants in a standard coagulation–flocculation system. The main findings were: (1) the combination of the organic coagulant Lipesa 1700 and the flocculant Lipesa 1587A was the most effective, achieving a coefficient of determination (R^2) of 96.07% for both BOD_5 and turbidity reduction; (2) optimization identified optimal doses of 2347.47 mg/L of Lipesa 1700 and 20 mg/L of Lipesa 1587A, with adjusted BOD_5 of 17121.0 mg O_2/L and adjusted turbidity of 7084.32 NTU; and (3) the inorganic coagulant Lipesa AC005 showed lower predictive accuracy (R^2 = 62.24% for BOD_5 and R^2 = 62.25% for turbidity), indicating lower effectiveness in vinasse clarification. In conclusion, the use of organic coagulants provides greater efficiency in contaminant reduction, although their industrial application should account for operational costs and residue management.

Keywords: Vinasse, clarification, BOD_5 reduction, turbidity removal, polyaluminum chloride.

Resumo: A poluição ambiental causada pela vinhaça, um subproduto da destilação de álcool caracterizado por alta carga orgânica e turbidez, representa um desafio significativo para a sustentabilidade das destilarias e a qualidade dos ecossistemas aquáticos. O objetivo deste estudo foi determinar as doses ótimas de coagulantes orgânicos e inorgânicos para a redução da Demanda Bioquímica de Oxigênio (DBO_5) e turbidez na vinhaça por meio da clarificação. A Metodologia de Superfície de Resposta (MSR) foi utilizada para avaliar combinações de coagulantes e floculantes em um sistema padrão de coagulação–floculação. Os principais achados foram: (1) a combinação do coagulante orgânico Lipesa 1700 com o floculante Lipesa 1587A foi a mais eficaz, alcançando um coeficiente de determinação (R^2) de 96,07% para a redução tanto da DBO_5 quanto da turbidez; (2) a otimização identificou as doses ótimas de 2347,47 mg/L de Lipesa 1700 e 20 mg/L de Lipesa 1587A, com DBO_5 ajustada de 17121,0 mg O_2/L e turbidez ajustada de 7084,32 NTU; e (3) o coagulante inorgânico Lipesa AC005 apresentou menor precisão preditiva (R^2 = 62,24% para DBO_5 e R^2 = 62,25% para turbidez), indicando menor eficácia na clarificação da vinhaça. Em conclusão, o uso de coagulantes orgânicos proporciona maior eficiência na redução de contaminantes, embora sua aplicação industrial deva considerar os custos operacionais e o manejo dos resíduos gerados.

Palavras-chave: Vinhaça, clarificação, redução de DBO_5, remoção de turbidez, policloreto de alumínio.

1. Introduction

Vinasse is a major environmental challenge due to its high organic load, measured as Biochemical Oxygen Demand (BOD_5) [1], and turbidity [2], caused by its abundant organic compounds and suspended solids [3]. This liquid waste is produced in large volumes during ethanol manufacturing [4], with 10 to 15 liters of vinasse generated per liter of alcohol [5], causing significant negative effects on receiving water bodies and ecosystems by depleting dissolved oxygen and harming aquatic life [6] [7]. The need for effective vinasse treatment grows amid increasing environmental awareness [8] and stricter industrial effluent regulations [9]. Consequently, distilleries face the challenge of implementing sustainable solutions to mitigate their environmental impact [10] [11].

In Peru, national ethanol production increased from 43 million liters in 2013 to 72 million liters in 2019 [12], increasing vinasse generation accordingly. At an average of 15 liters per liter of alcohol, vinasse volumes can reach 800 million liters annually [5], representing a serious pollution problem when improperly disposed of, contaminating water and soil and affecting ecosystems [13] [14]. In the Lambayeque region, distilleries such as Destilería Zaña SAC [15], Grupo Comercial Bari SA [16], and Destilería Naylamp EIRL lack vinasse treatment systems [17], discharging effluents into Dren 4000, which empties into Santa Rosa beach [18], severely impacting the marine ecosystem [19].

Although some industries have attempted vinasse treatment [6], challenges remain regarding treatment efficacy [20], largely due to high implementation costs [21]. Clarification has emerged as a promising method to reduce turbidity and organic load [22]. However, the absence of precise methodologies for determining optimal coagulant and flocculant doses results in inconsistent outcomes and incomplete clarification [23] [24] [25], leading to effluents with elevated turbidity and BOD_5 [26], and increasing environmental risks [27]. Overuse of chemicals also causes unnecessary expenses and issues like sludge formation requiring proper disposal [28] [29].

Addressing vinasse treatment is crucial for environmental, economic, and regulatory reasons [30] [31] [32]. Inadequate treatment threatens biodiversity, human health, and company sustainability and reputation [33] [34]. Therefore, effective vinasse treatment is vital to demonstrate environmental responsibility and comply with regulations [35]. Economically, process optimization

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minimizes operating costs [23] and enhances distillation efficiency [36]. Determining optimal coagulant and flocculant dosages is essential to reduce chemical consumption and waste disposal costs [37] [38] [39]. This study aims to establish these optimal dosages to lower BOD_5 and turbidity in vinasse through clarification.

2. Materials and methods

2.1. Sampling and characterization of vinasse

Vinasse samples were collected from the discharge line of the bottom stream of the first distillation column in an alcohol distillery located in the Lambayeque region, Peru. The samples were stored in clean, amber-colored, airtight 3 L plastic bottles at 4°C for a period of six hours without any preservatives before laboratory analysis. The physicochemical characteristics of the collected sample are presented in Table 2, while the analytical methods used are detailed in Table 1.

Table 1. Analytical method for characterization.

Parameter	Analytical Method
Chemical Oxygen Demand (COD)	SMEWW-APHA-AWWA-WEF Part 5220 D, 24th Ed. 2023. Chemical Oxygen Demand (COD). Closed Reflux, Colorimetric Method.
Biochemical Oxygen Demand (BOD_5)	SMEWW-APHA-AWWA-WEF Part 5210 B, 24th Ed. 2023. Biochemical Oxygen Demand (BOD). 5-Day BOD Test.
Turbidity	SMEWW-APHA-AWWA-WEF. Part 2130 B. 24th Ed. 2023. Turbidity. Nephelometric Method.
Total Suspended Solids (TSS)	ASTM D5907-18. Standard Test Methods for Filterable Matter (Total Dissolved Solids) and Nonfilterable Matter (Total Suspended Solids) in Water.
Volatile Suspended Solids (VSS)	SMEWW-APHA-AWWA-WEF Part 2540 E, 24th Ed. 2023 Solids. Fixed and Volatile Solids Ignited at 550°C.
pH	SMEWW-APHA-AWWA-WEF Part 4500-H+ B. 24th Ed. 2023. pH Electrometric Method
Alkalinity	NTP 214.026:1999 (revised in 2019). 1st Edition. Determination of Alkalinity. Volumetric Method.
Electrical Conductivity	SMEWW-APHA-AWWA-WEF Part 2510 B. 24th Ed. 2023. Conductivity. Laboratory Method.

Source: Authors

Table 2. Physicochemical characterization of the vinasse samples.

Parameter	Unit	Value
Chemical Oxygen Demand (COD)	mg/L	54640
Biochemical Oxygen Demand (BOD_5)	mg/L	28260
Turbidity	NTU	11610
Total Suspended Solids (TSS)	mg/L	5200
Volatile Suspended Solids (VSS)	mg/L	3810
pH	-	4.45
Alkalinity	mg $CaCO_3$/L	410
Electrical Conductivity	µS/cm	12580

Source: Authors

2.2. Physicochemical properties of coagulants and flocculants

Coagulation was carried out using aluminum polychloride (Lipesa AC005) and a quaternary ammonium tannin-based solution (Lipesa 1700), applied separately. pH adjustment was unnecessary due to the broad effective pH ranges (5–10 for AC005 and 1–13 for Lipesa 1700). A flocculant (Lipesa 1587A), a highly cationic, high-molecular-weight polymer, was chosen to enhance floc settling. The physicochemical characteristics of the reagents are provided in Table 3, based on technical specifications.

Table 3. Physicochemical characteristics of coagulants and flocculants.

Product	Chemical composition	Concentration	pH[a]	Specific Gravity[a]
Lipesa 1700	Quaternary ammonium tannin solution	25%	2–3	1.01–1.02
Lipesa AC005	Aluminum polychloride solution	NE	3–4.5	1.26–1.33
Lipesa 1587A	Polydiallyl dimethyl ammonium chloride - PolyDADMAC	0.1%	NE	NE

Source: Product technical data sheets. Note: NE: Not specified; [a]: At 25°C.

2.3. Experimental design

Tests were conducted using a standardized coagulation–flocculation system at room temperature (25°C), with controlled conditions for rapid mixing, slow mixing, and sedimentation. A six-position programmable jar test apparatus was used according to ASTM D2035 standards; the glass jars had a capacity of 1 L. The mixing conditions were set to 150 rpm for 1 minute for rapid mixing and 30 rpm for 5 minutes for slow mixing. The treatment doses for the coagulants and flocculant are shown in Table 4; the coagulant was added at the start of agitation, while the flocculant was added 2 minutes into the slow mixing stage. Sedimentation was allowed for 5 minutes. Interactions between variables were evaluated, and the optimal dose combinations for contaminant removal were identified. In total, 18 tests were conducted with 3 replicates each, amounting to 54 evaluations. To assess the reduction of BOD_5 and turbidity in vinasse using the selected coagulants and flocculants, the response surface methodology (RSM) was employed to analyze the effects of the independent variables (different doses of inorganic coagulant – Lipesa AC005, organic coagulant – Lipesa 1700, and cationic flocculant – Lipesa 1587A) on clarification efficiency, as measured by BOD_5 and turbidity reduction.

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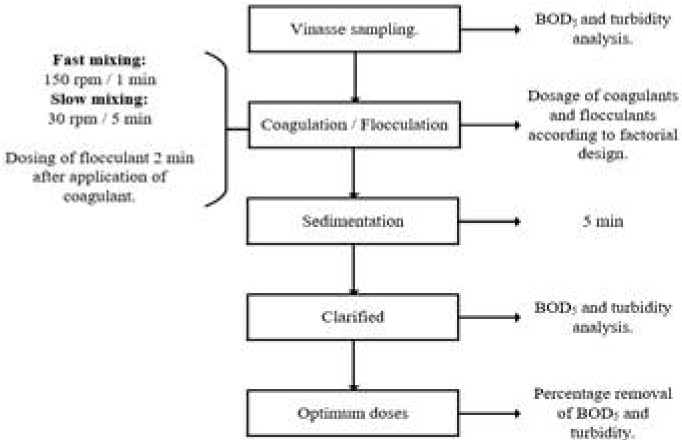
Table 4. Treatment is for experimental design.

A \ B		Lipesa AC005 (mg/L)			Lipesa 1700 (mg/L)		
		(A1) 1600	(A2) 2000	(A3) 2400	(A4) 2000	(A5) 2400	(A6) 2800
Lipesa 1587A (mg/L)	(B1) 10	A1 B1	A2 B1	A3 B1	A4 B1	A5 B1	A6 B1
	(B2) 15	A1 B2	A2 B2	A3 B2	A4 B2	A5 B2	A6 B2
	(B3) 20	A1 B3	A2 B3	A3 B3	A4 B3	A5 B3	A6 B3

Source: Authors.
Note: Treatment combinations under study; Factor A: organic or inorganic coagulant; Factor B: flocculant.

Figure 1. Process flow diagram.



Source: Authors

3. Results and discussion

3.1. Modeling BOD_5 reduction in vinasse using the inorganic coagulant Lipesa AC005

The response surface regression model applied to BOD_5 reduction via the inorganic coagulant Lipesa AC005 combined with the flocculant Lipesa 1587A shows moderate explanatory power but limited predictive capacity. As indicated in Table 5, the standard error of the estimate (S = 757.152) is high, reflecting considerable residual variability and suggesting the model does not fully capture the relationship between independent variables and BOD_5 reduction. This points to additional influencing factors not included in the analysis. The coefficient of determination (R^2 = 62.24%) indicates a moderate fit, yet the adjusted R^2 (55.95%) and predicted R^2 (46.77%) values reveal potential overfitting and low generalization for new operational conditions. The discrepancy between R^2 and adjusted R^2 further implies that some variables may not significantly contribute, highlighting the need to incorporate other relevant factors such as pH, temperature, or colloidal matter proportion [40], [41]. Compared to Rodrigues et al. [24], who reported R^2 values above 80% for organic contaminant reduction in vinasse via coagulation-flocculation, the model here performs less effectively, possibly due to the inherent variability of vinasse composition affecting treatment reproducibility. Moreover, studies such as those by Silva et al. [28] demonstrated that coagulant interactions with complex matrices can produce nonlinear removal efficiencies, justifying exploration of advanced models like nonlinear regression or machine learning. From an applied perspective, the moderate predictive capacity implies significant variability in BOD_5 reduction across vinasse batches or operational conditions, making pilot-scale trials essential before industrial application to optimize dosages based on effluent characteristics [42].

Table 5. Summary of the Response Surface Regression Model: BOD_5 vs. Lipesa AC005 and Lipesa 1587A.

S	R-squared	Adjusted R-squared	Predicted R-squared
757.152	62.24%	55.95%	46.77%

Source: Authors' elaboration using Minitab Statistical Software, Version 21.1.0

The ANOVA results in Table 6 confirm a statistically significant effect of both reagents on BOD_5 reduction ($p < 0.05$). The overall model ($F = 9.89$, $p = 0.000$) indicates that at least one variable significantly affects BOD_5 removal. Individually, Lipesa AC005 ($F = 10.89$, $p = 0.003$) and Lipesa 1587A ($F = 9.00$, $p = 0.005$) contribute significantly, while quadratic terms ($F = 12.01$, $p = 0.000$) suggest nonlinear process efficiency. Notably, the quadratic term for Lipesa AC005 ($F = 14.57$, $p = 0.001$) reveals a saturation point beyond which increasing dosage does not improve BOD_5 reduction and may cause adverse effects such as oversaturation and floc re-dispersion, as previously reported with inorganic coagulants at high doses [43]. The significant interaction between both reagents ($F = 5.54$, $p = 0.025$) indicates synergistic effects rather than additive ones, consistent with prior findings that combining inorganic coagulants and cationic polymers enhances organic matter removal at optimal doses [44]. This highlights the importance of precise dosage control to optimize treatment. However, the lack-of-fit test ($F = 75.01$, $p = 0.000$) suggests the model does not account for all factors influencing BOD_5 reduction, likely due to omission of variables such as pH, temperature, specific vinasse composition, or sedimentation time [45]. Future studies should include these variables and consider advanced methods like multi-objective optimization or AI-based techniques to improve predictive accuracy and treatment reproducibility [46].

Table 6. Summary of the Response Surface Regression Model: BOD_5 vs. Lipesa AC005 and Lipesa 1587A.

Source	DF	Adj SS	Adj MS	F-value	p-value
Modelo	5	28349201	5669840	9.89	0.000
Lineal	2	11403937	5701969	9.95	0.000
Lipesa AC005	1	6242400	6242400	10.89	0.003
Lipesa 1587A	1	5161537	5161537	9.00	0.005
Cuadrado	2	13767957	6883978	12.01	0.000
Lipesa AC005 * Lipesa AC005	1	8350422	8350422	14.57	0.001
Lipesa 1587A * Lipesa 1587A	1	5417535	5417535	9.45	0.004

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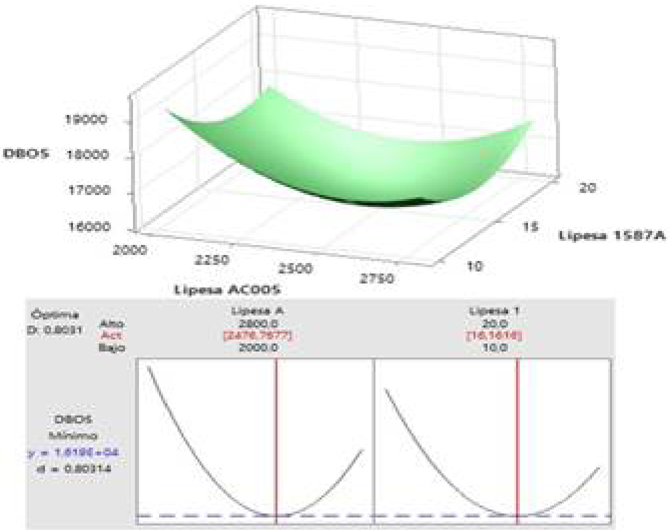
Interacción de 2 factores	1	3177306	3177306	5.54	0.025
Lipesa AC005 * Lipesa 1587A	1	3177306	3177306	5.54	0.025
Error	30	17198363	573279		
Falta de ajuste	3	15355988	5118663	75.01	0.000
Error puro	27	1842375	68236		
Total	35	45547564			

Source: Authors' elaboration using Minitab Statistical Software, Version 21.1.0

Figure 2 illustrates the effect of Lipesa AC005 and Lipesa 1587A dosages on BOD_5 reduction. The treatment efficiency shows a nonlinear trend, indicating a saturation point beyond which increasing the dosage no longer significantly improves organic load removal. This aligns with prior studies noting that excessive concentrations of inorganic coagulants may destabilize flocs and re-disperse colloidal particles, diminishing process efficiency [28], [47]. Additionally, the observed synergistic interaction between Lipesa AC005 and Lipesa 1587A confirms their combined effectiveness in reducing BOD_5. However, the quadratic term associated with Lipesa AC005 suggests a limit to its effectiveness, reinforcing the need to define an optimal dosage range to avoid diminishing returns or adverse effects. These findings emphasize the importance of optimizing operational conditions to enhance clarification efficiency while minimizing excessive chemical use, associated costs, and waste generation [48].

Figure 2. Surface plot of BOD_5 vs. Lipesa 1587A and Lipesa AC005.



Source: Authors' elaboration using Minitab Statistical Software, Version 21.1.0

The optimization process identified an optimal dosage of 2476.77 mg/L for Lipesa AC005 and 16.1616 mg/L for Lipesa 1587A, resulting in an adjusted BOD_5 of 16193.5 mg O_2/L and a composite desirability of 0.803136 (Table 7). This indicates high treatment effectiveness, though not the maximum possible (1.0), suggesting possible influence from unmodeled factors such as vinasse variability or operational changes. Compared to the combination of Lipesa 1700 and Lipesa 1587A—which achieved a higher desirability (0.897068)—the performance of the inorganic coagulant could be further optimized through refined dosage adjustments or by incorporating additional parameters into the model. Practically, this formulation represents a viable option for vinasse clarification. However, its industrial application should account for variability in performance, and further exploration of nonlinear interactions could enhance the treatment's robustness and predictive accuracy [40].

Table 7. Response Optimization Solution: BOD_5 vs. Lipesa AC005 and Lipesa 1587A

Solution	Lipesa AC005 (mg/L)	Lipesa 1587A (mg/L)	Adjusted BOD_5 (mg O_2/L)	Composite Desirability
1	2476.77	16.1616	16193.5	0.803136

Source: Authors' elaboration using Minitab Statistical Software, Version 21.1.0

3.2. Modeling BOD_5 reduction in vinasse using the organic coagulant Lipesa 1700

The response surface regression model for BOD_5 reduction using the organic coagulant Lipesa 1700 combined with the flocculant Lipesa 1587A (Table 8) shows a significantly better fit than the model based on Lipesa AC005 and Lipesa 1587A (Table 5). The standard error of the estimate (S = 361.233) is notably lower, indicating less residual variability and a better fit. The coefficient of determination (R^2 = 96.07%) reflects a very strong relationship between the independent variables and BOD_5 reduction. Adjusted R^2 (95.42%) and predicted R^2 (94.31%) are similarly high, demonstrating minimal overfitting and strong predictive power. This robustness confirms reliable predictions for new datasets.

Compared to similar studies reporting R^2 values between 85% and 90% [24], [23], this model outperforms previous results, likely due to the high efficiency of the tannin-based coagulant Lipesa 1700 in neutralizing organic charges, its synergy with the flocculant Lipesa 1587A promoting particle aggregation and sedimentation, and the precise dosage optimization enabled by response surface methodology (RSM). Despite the excellent fit, high R^2 values do not guarantee optimal cost-efficiency or operability. Organic coagulants may require higher reagent consumption, generate more sludge, and involve longer settling times than inorganic coagulants, as noted by Formann et al. [40] and Padilha et al. [43]. Additionally, the model's predictive accuracy under controlled conditions may vary at large scale due to vinasse heterogeneity.

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Table 8. Summary of the Response Surface Regression Model: BOD_5 vs. Lipesa 1700 and Lipesa 1587A.

S	R-squared	Adjusted R-squared	Predicted R-squared
361.233	96.07%	95.42%	94.31%

Source: Authors' elaboration using Minitab Statistical Software, Version 21.1.0

ANOVA results (Table 9) reveal high statistical significance ($p < 0.05$) for nearly all sources of variation, with an overall model F-value of 146.74 ($p = 0.000$), indicating strong explanation of BOD_5 variability by the studied variables. Compared to the Lipesa AC005 model ($F = 9.89$, Table 6), this confirms greater efficiency of Lipesa 1700. The linear effects of Lipesa 1700 ($F = 60.37$, $p < 0.001$) and Lipesa 1587A ($F = 212.22$, $p < 0.001$) significantly contribute to BOD_5 reduction, with the flocculant having a stronger influence, consistent with prior findings on cationic flocculants enhancing aggregation [44]. Quadratic effects ($F = 229.42$, $p = 0.000$) reveal nonlinear efficiency; notably, the quadratic term for Lipesa 1700 ($F = 456.78$, $p = 0.000$) suggests a saturation point beyond which dosage increases yield no further benefits and may cause colloidal dispersion. In contrast, the quadratic term for Lipesa 1587A ($F = 2.06$, $p = 0.161$) is not significant, indicating a mostly linear effect and a more flexible dosing range. Unlike the significant interaction between Lipesa AC005 and Lipesa 1587A ($p = 0.025$), the interaction between Lipesa 1700 and Lipesa 1587A is not significant ($F = 2.26$, $p = 0.144$), suggesting that the organic coagulant's performance is less dependent on flocculant dosage. This may be advantageous in industrial contexts with variable vinasse composition. The lack-of-fit test ($F = 9.23$, $p = 0.000$) indicates some model-data discrepancy, though substantially less than in the Lipesa AC005 model ($F = 75.01$). Overall, the high R^2 confirms the model explains almost all BOD_5 variability, surpassing prior coagulation-flocculation studies.

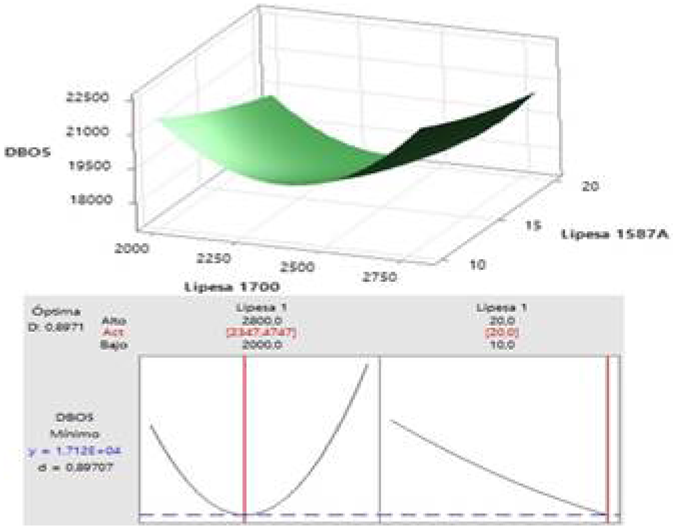
Table 9. Summary of the Response Surface Regression Model: BOD_5 vs. Lipesa 1700 and Lipesa 1587A.

Source	DF	Adj SS	Adj MS	F-value	p-value
Modelo	5	95737817	19147563	147	0.000
Lineal	2	35569621	17784810	136	0.000
Lipesa AC005	1	7877604	7877604	60.3	0.000
Lipesa 1587A	1	27692017	27692017	212	0.000
Cuadrado	2	59873890	29936945	229	0.000
Lipesa AC005 * Lipesa AC005	1	59605001	59605001	456	0.000
Lipesa 1587A * Lipesa 1587A	1	268889	268889	2.06	0.161
Interacción de 2 factores	1	294306	294306	2.26	0.144
Lipesa AC005 * Lipesa 1587A	1	294306	294306	2.26	0.144
Error	30	3914680	130489		
Falta de ajuste	3	1981855	660618	9.23	0.000
Error puro	27	1932825	71586		
Total	35	99652497			

Source: Authors' elaboration using Minitab Statistical Software, Version 21.1.0

Figure 3 shows the interaction between Lipesa 1700 (organic coagulant) and Lipesa 1587A (flocculant) dosages in BOD_5 reduction. A nonlinear efficiency trend suggests a saturation point beyond which increasing the coagulant dosage does not significantly improve organic load removal. This effect aligns with previous findings that excessive organic coagulants may stabilize colloidal particles and reduce sedimentation efficiency [28], [48]. Compared to the combination in Figure 2 (Lipesa AC005 and Lipesa 1587A), the Lipesa 1700 and Lipesa 1587A pairing exhibits a more predictable response and better model fit, indicating tannin-based coagulants as a promising alternative for vinasse clarification [25]. However, the interaction does not show as strong a synergistic effect as the inorganic coagulant combination, implying treatment efficiency depends more on individual reagent dosages.

Figure 3. Surface plot of BOD_5 vs. Lipesa 1587A and Lipesa 1700.



Source: Authors' elaboration using Minitab Statistical Software, Version 21.1.0

Optimization identified optimal dosages of 2347.47 mg/L for Lipesa 1700 and 20 mg/L for Lipesa 1587A, achieving an adjusted BOD_5 of 17121.0 mg O_2/L and a composite desirability of 0.897068 (Table 10), close to the model's optimal level. In contrast, the Lipesa AC005 and Lipesa 1587A combination yielded a lower desirability (0.803136), reflecting the superior efficiency of the organic coagulant. This is further supported by a higher coefficient of determination ($R^2 = 96.07\%$) compared to the Lipesa AC005 model ($R^2 = 62.24\%$). The enhanced performance is attributed to improved organic load removal via charge neutralization and formation of more stable flocs, which facilitate sedimentation and organic matter removal [25], [49].

Table 10. Response Optimization Solution: BOD_5 vs. Lipesa 1700 and Lipesa 1587A

Solution	Lipesa 1700 (mg/L)	Lipesa 1587A (mg/L)	Adjusted BOD_5 (mg O_2/L)	Composite Desirability
1	2347.47	20	17121.0	0.897068

Source: Authors' elaboration using Minitab Statistical Software, Version 21.1.0

3.3. Modeling turbidity reduction in vinasse using the inorganic coagulant Lipesa AC005

The response surface regression model for turbidity reduction using Lipesa AC005 and the high molecular weight cationic flocculant Lipesa 1587A (Table 11) shows moderate fitting capacity, like the BOD_5 model with the same reagents. The standard error (S = 313.249) suggests notable residual variability, indicating additional influencing factors beyond coagulant and flocculant dosages, such as pH, temperature, and settling time [45]. The coefficient of determination (R^2 = 62.25%) indicates that the model explains approximately 62% of turbidity variability, with 38% unexplained. Adjusted R^2 (55.96%) and predicted R^2 (46.79%) are lower, suggesting moderate predictive capacity and possible nonlinear interactions or omitted variables like vinasse composition [50]. These results are lower than those reported by Sacchi et al. [26], who achieved R^2 values above 80% using coagulation followed by microfiltration. This emphasizes the potential benefit of combining organic coagulants or additional treatments (e.g., filtration or advanced oxidation) for improved suspended solids removal. The moderate performance of the model may relate to the variable efficiency of Lipesa AC005 in complex matrices, as Kee et al. [27] noted that colloidal load and dissolved organic matter influence floc formation and settling. High suspended solids content may also increase coagulant demand, suggesting the dosages evaluated might not have been optimal [44]. Despite the limited fit, Lipesa 1587A appears critical for clarification, as high molecular weight flocculants enhance sedimentation efficiency based on dosage and contact time. Including detailed evaluations of mixing and settling times could enhance the model by better capturing floc stability and clarification efficiency [26].

Table 11. Summary of the Response Surface Regression Model: Turbidity vs. Lipesa AC005 and Lipesa 1587A.

S	R-squared	Adjusted R-squared	Predicted R-squared
313.249	62.25%	55.96%	46.79%

Source: Authors' elaboration using Minitab Statistical Software, Version 21.1.0

ANOVA results (Table 12) confirm the overall model is significant (F = 9.89, p = 0.000), indicating that the combination of coagulant and flocculant significantly affects turbidity reduction. However, the model's predictive capacity (46.79%) is lower than for BOD_5 reduction (Table 5), reflecting greater unexplained variability. Both Lipesa AC005 (F = 10.85, p = 0.003) and Lipesa 1587A (F = 9.00, p = 0.005) significantly influence turbidity, though with slightly lower effect sizes than in BOD_5 removal. Quadratic terms show a nonlinear relationship: Lipesa AC005's quadratic effect (F = 14.60, p = 0.001) indicates a saturation point beyond which increased dosage reduces effectiveness, potentially causing colloidal stabilization rather than aggregation—an effect reported with excess inorganic coagulants [26] [27]. Lipesa 1587A's quadratic term (F = 9.46, p = 0.004) also suggests an optimal dosage range, as excess flocculant may stabilize flocs excessively, hindering sedimentation [35]. The interaction between coagulant and flocculant is significant (F = 5.56, p = 0.025), indicating that their dosages influence each other's performance, consistent with previous findings that cationic polymers synergistically enhance floc formation initiated by inorganic coagulants [9], [28]. However, the moderate F-value (5.56) suggests room for improvement by refining dosages or including variables like pH or settling time [25]. A high lack-of-fit value (F = 75.39, p = 0.000) reveals significant discrepancies between the model and experimental data, likely due to unmodeled factors such as vinasse composition (dissolved solids, phenolic compounds, colloidal matter), operational parameters (pH, temperature, agitation), and settling time affecting floc stability and clarification efficiency [41] [43] [44]. This lack of fit is slightly greater than that observed in the BOD_5 model (F = 75.01), indicating turbidity is more challenging to model due to these complex influences.

Table 12. Summary of the Response Surface Regression Model: Turbidity vs. Lipesa AC005 and Lipesa 1587A.

Source	DF	Adj SS	Adj MS	F-value	p-value
Modelo	5	4854527	970905	9.89	0.000
Lineal	2	1948294	974147	9.93	0.000
Lipesa AC005	1	1064709	1064709	10.85	0.003
Lipesa 1587A	1	883584	883584	9.00	0.005
Cuadrado	2	2360481	1180241	12.03	0.000
Lipesa AC005 * Lipesa AC005	1	1432278	1432278	14.60	0.001
Lipesa 1587A * Lipesa 1587A	1	928203	928203	9.46	0.004
Interacción de 2 factores	1	545752	545752	5.56	0.025
Lipesa AC005 * Lipesa 1587A	1	545752	545752	5.56	0.025
Error	30	2943742	98125		
Falta de ajuste	3	2629811	876604	75.39	0.000
Error puro	27	313931	11627		
Total	35	7798269			

Source: Authors' elaboration using Minitab Statistical Software, Version 21.1.0

Figure 4 illustrates the relationship between Lipesa AC005 and Lipesa 1587A dosages in turbidity reduction of vinasse. The process exhibits nonlinear behavior, with a saturation point beyond which increasing dosages no longer significantly improve suspended solids removal. This aligns with previous findings showing that high concentrations of inorganic coagulants can cause

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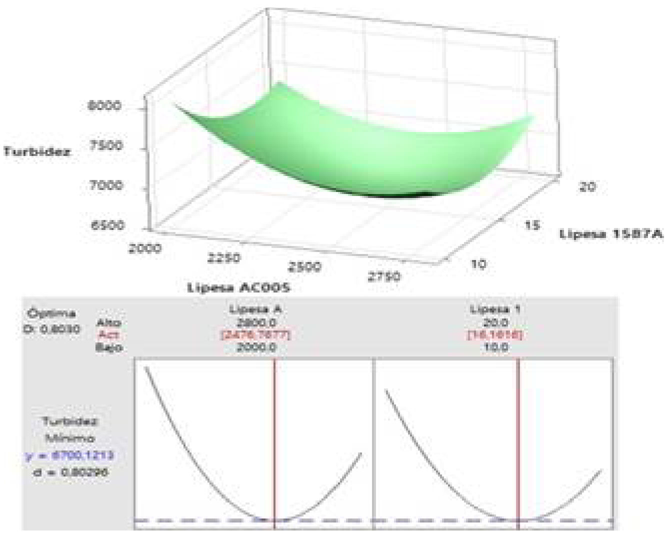
colloidal particle stabilization and floc re-dispersion, reducing treatment efficiency [39]. The interaction between the two reagents moderately influences turbidity reduction, emphasizing the need to optimize dosages for effective clarification. Additionally, adjusting operational conditions such as settling time or pH—known to impact coagulation–flocculation efficiency—could enhance performance [27]. Given that turbidity reduction does not reach optimal levels, complementary strategies like combining filtration or advanced oxidation treatments are recommended to improve suspended solids removal [45]. These results highlight the importance of balancing coagulant and flocculant dosing to maximize turbidity reduction without compromising treatment stability.

Figure 4. Surface plot of turbidity vs. Lipesa 1587A and Lipesa AC005.



Source: Authors' elaboration using Minitab Statistical Software, Version 21.1.0

Optimization yielded optimal dosages of 2476.77 mg/L (Lipesa AC005) and 16.1616 mg/L (Lipesa 1587A), achieving an adjusted turbidity of 6700.12 NTU and a composite desirability of 0.802957 (Table 13). This level of optimization is acceptable but indicates potential for improvement in the model or operational conditions. The turbidity reduction performance is like the BOD_5 optimization with the same reagents (desirability = 0.803136), reflecting limitations in removing organic matter and suspended solids. The moderate explanatory power of the model (R^2 = 62.25%) suggests treatment efficiency is affected by the complex matrix of vinasse, where factors like colloidal load and dissolved organics influence floc stability. Therefore, further methodological adjustments and hybrid treatment approaches are necessary to improve process efficiency and stability [26], [43].

Table 13. Response Optimization Solution: Turbidity vs. Lipesa AC005 and Lipesa 1587A

Solution	Lipesa AC005 (mg/L)	Lipesa 1587A (mg/L)	Adjusted Turbidity (NTU)	Composite Desirability
1	2476.77	16.1616	6700.12	0.802957

Source: Authors' elaboration using Minitab Statistical Software, Version 21.1.0

3.4. Modeling turbidity reduction in vinasse using the organic coagulant Lipesa 1700

The response surface regression model for turbidity reduction using the organic coagulant Lipesa 1700 combined with the flocculant Lipesa 1587A shows a markedly better fit than the model based on Lipesa AC005 and Lipesa 1587A (Table 14). The standard error of the estimate decreases substantially (S = 149.504 vs. 313.249, Table 11), indicating reduced residual variability and enhanced explanatory capacity. The coefficient of determination (R^2 = 96.07%) captures most turbidity variability, far surpassing the Lipesa AC005 model (R^2 = 62.25%). Minimal differences between adjusted R^2 (95.41%) and predicted R^2 (94.30%) confirm strong predictive ability without overfitting. These results outperform previous studies reporting R^2 between 85% and 90% [24], [28]. The improved efficiency is attributed to the tannin-based Lipesa 1700's superior charge neutralization in colloidal-rich effluents [49], its synergy with Lipesa 1587A optimizing floc formation, and the precise dosage optimization enabled by Response Surface Methodology (RSM). Economically, despite its higher efficiency, industrial use of Lipesa 1700 must consider increased reagent costs and sludge handling, as organic coagulants often require greater dosages than inorganic ones.

Table 14. Summary of the Response Surface Regression Model: Turbidity vs. Lipesa 1700 and Lipesa 1587A.

S	R-squared	Adjusted R-squared	Predicted R-squared
149.504	96.07%	95.41%	94.30%

Source: Authors' elaboration using Minitab Statistical Software, Version 21.1.0

ANOVA (Table 15) confirms high statistical significance ($p < 0.05$) for most terms. The overall model's F-value (146.62, p = 0.000) indicates Lipesa 1700 combined with Lipesa 1587A explains a large portion of turbidity variability, outperforming the Lipesa AC005 model (F = 9.89, Table 12). Both reagents significantly affect turbidity reduction, with Lipesa 1587A exerting a stronger influence (F = 212.03 vs. 60.25), consistent with its role in particle aggregation and sedimentation [23]. Quadratic terms reveal nonlinear effects: Lipesa 1700's quadratic term (F = 456.52, p = 0.000) suggests an optimal dosage beyond which efficiency declines due to charge oversaturation causing colloid stabilization [25], [27]. The quadratic term of Lipesa 1587A (F = 2.05, p = 0.163) is not significant, indicating its effect remains stable and linear over the dosage range, reflecting the

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predictable behavior of high molecular weight flocculants [24], [28]. Unlike the significant interaction in the Lipesa AC005 and Lipesa 1587A model ($F = 5.56$, $p = 0.025$), the interaction between Lipesa 1700 and Lipesa 1587A is not significant ($F = 2.26$, $p = 0.143$), suggesting independent actions. Operationally, this independence allows more flexible control without simultaneous dosage optimization, though turbidity improvements rely more on optimizing individual dosages [20], [29]. The lack-of-fit test ($F = 9.21$, $p = 0.000$) indicates minor unexplained variability, yet considerably lower than in the Lipesa AC005 model ($F = 75.39$), confirming greater stability and reliability.

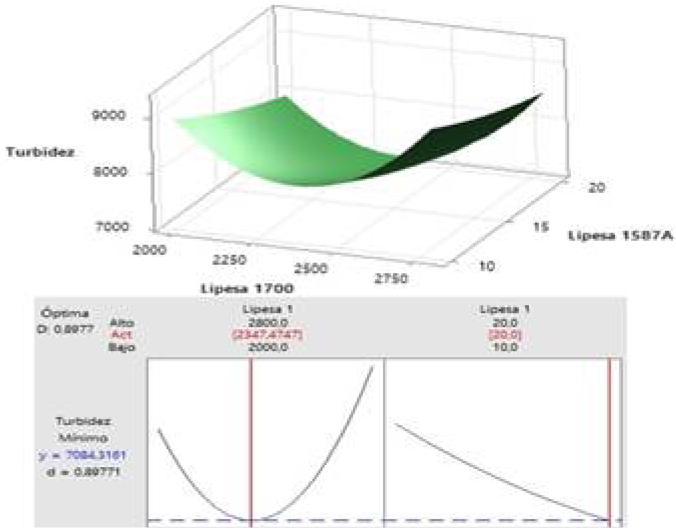
Table 15. Summary of the Response Surface Regression Model: Turbidity vs. Lipesa 1700 and Lipesa 1587A.

Source	DF	Adj SS	Adj MS	F-value	p-value
Modelo	5	16386175	3277235	147	0.000
Lineal	2	6085894	3042947	136	0.000
Lipesa AC005	1	1346634	1346634	60	0.000
Lipesa 1587A	1	4739259	4739259	212	0.000
Cuadrado	2	10249656	5124828	229	0.000
Lipesa AC005 * Lipesa AC005	1	10203903	10203903	456	0.000
Lipesa 1587A * Lipesa 1587A	1	45753	45753	2.05	0.163
Interacción de 2 factores	1	50625	50625	2.26	0.143
Lipesa AC005 * Lipesa 1587A	1	50625	50625	2.26	0.143
Error	30	670544	22351		
Falta de ajuste	3	339175	113058	9.21	0.000
Error puro	27	331369	12273		
Total	35	17056719			

Source: Authors' elaboration using Minitab Statistical Software, Version 21.1.0

Figure 5 shows the interaction between Lipesa 1700 and Lipesa 1587A dosages in vinasse turbidity reduction. The response model fits better than the combination of Lipesa AC005 and Lipesa 1587A (Figure 4), indicating higher stability and efficiency when using the organic coagulant. Nevertheless, the nonlinear trend reveals a saturation point beyond which increasing reagent dosages no longer significantly enhances turbidity removal. Unlike the inorganic coagulant, the interaction between Lipesa 1700 and Lipesa 1587A does not produce a strong synergistic effect, suggesting turbidity reduction mainly depends on the individual dosages of each reagent. Previous studies report that organic coagulants, such as tannins, are more effective in neutralizing colloidal charges and forming stable flocs, which explains the improved predictability of this model [24].

Figure 5. Surface plot of Turbidity vs. Lipesa 1587A and Lipesa 1700.



Source: Authors' elaboration using Minitab Statistical Software, Version 21.1.0

Optimization determined optimal dosages of 2347.47 mg/L (Lipesa 1700) and 20 mg/L (Lipesa 1587A), achieving an adjusted turbidity of 7084.32 NTU and a composite desirability of 0.897706, the highest among all combinations evaluated—demonstrating its superiority in turbidity reduction (Table 16). Compared to the Lipesa AC005 and Lipesa 1587A combination (desirability = 0.802957), this reflects a significant improvement, supported by a coefficient of determination ($R^2 = 96.07\%$) that surpasses the inorganic coagulant model ($R^2 = 62.25\%$). From an operational perspective, organic coagulants may reduce environmental impact and dependence on inorganic salts; however, they can increase sludge production and costs, warranting thorough evaluation before industrial application.

Table 16. Response Optimization Solution: BOD_5 vs. Lipesa 1700 and Lipesa 1587A

Solution	Lipesa 1700 (mg/L)	Lipesa 1587A (mg/L)	Adjusted Turbidity (NTU)	Composite Desirability
1	2347.47	20	7084.32	0.897706

Source: Authors' elaboration using Minitab Statistical Software, Version 21.1.0

4. Conclusion

The results of this study identified the optimal dosages of coagulants and flocculants for reducing BOD_5 and turbidity in vinasse through clarification. Response Surface Methodology (RSM) analysis demonstrated that the organic coagulant Lipesa 1700 combined with flocculant Lipesa 1587A was more efficient in reducing both parameters than the inorganic coagulant Lipesa AC005 with the same flocculant. The regression model for BOD_5 reduction showed a better fit with Lipesa 1700 (R^2 =

96.07%) compared to Lipesa AC005 (R^2 = 62.24%), indicating the tannin-based coagulant's greater capacity to interact with dissolved organic matter in vinasse. Process optimization established optimal dosages of 2347.47 mg/L for Lipesa 1700 and 20 mg/L for Lipesa 1587A, achieving an adjusted BOD_5 of 17121.0 mg O_2/L and a composite desirability of 0.897. Similarly, for turbidity reduction, the Lipesa 1700 and Lipesa 1587A combination showed superior predictive accuracy (R^2 = 96.07%) versus Lipesa AC005 with Lipesa 1587A (R^2 = 62.25%). Optimal dosages were identical to those for BOD_5 reduction, reaching an adjusted turbidity of 7084.32 NTU and a desirability of 0.898. These results suggest that organic coagulant promotes more stable floc formation and effective sedimentation. Despite these promising outcomes, limitations were noted in the modeling, particularly for the Lipesa AC005 and Lipesa 1587A combination, which had moderate predictive capacity. Future research should include additional variables such as pH, temperature, and settling time to enhance model accuracy. The lack-of-fit analysis also revealed unaccounted factors that may affect process efficiency. Overall, this study supports the use of organic coagulants as an efficient and environmentally sustainable alternative for vinasse treatment by clarification. However, scaling to industrial applications requires further evaluation of operational factors, costs, and sludge management.

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