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Inovações e Soluções Sustentáveis em Engenharia Ambiental

Nutrient removal in anaerobic-aerobic reactors followed by a trickling filter

Remoção de nutrientes em reatores anaeróbio-aeróbio seguido por um filtro percolador

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ABSTRACT

This study aimed to evaluate the performance of a combined system used in nitrogen and phosphorus removal, with a variation of the hydraulic retention time (HRT) and the recirculation rate (R). The system consisted of three sequential reactors operated in series, composed of an anaerobic reactor (20 L), an aerobic fixed bed reactor (19 L), and a trickling filter (16 L). The recirculation rates of 150, 100, and 50% and HRT of 9, 7, and 5 h have been, with a constant aeration flow of 10 L min-1. For system evaluation, liquid temperature, pH, total alkalinity, bicarbonate alkalinity, volatile acids, dissolved oxygen, chemical oxygen demand, Kjeldahl total nitrogen, nitrogen ammonia, nitrite, nitrate, and total phosphorus have been analyzed. The combined system has reached achieved organic matter removal efficiencies of 99%, 98%, and 98% for the recirculation rates of 50, 100, and 150%, respectively. Under the same R rates, TKN removal efficiencies resulted in 96, 89, and 87%, and TP removal efficiencies in 78, 82, and 77%, respectively. When operated with HRT of 9, 7, and 5 h, the combined system achieved removal efficiencies of 86, 96, and 98% for COD, 90, 93, and 94% for TKN, and 74, 89, and 95% for TP, respectively. The best operational condition was experimentally established having with a recirculation rate of 150% and HRT of 5 h. However, after experimental validation by CCRD (through desirability), the optimal operational condition resulted in a recirculation rate of 123% and HRT of 5 h for TKN and PT removal efficiencies.

Keywords: Combined processes; Nitrogen removal; Phosphorus adsorption

RESUMO

O objetivo deste trabalho foi avaliar o desempenho de um sistema combinado utilizado na remoção de nitrogênio e fósforo, com variação do tempo de detenção hidráulica (TRH) e da taxa de recirculação (R). O sistema foi composto por três reatores sequenciais operados em série, composto por um reator anaeróbio (20 L), um reator aeróbio de leito fixo (19 L) e um filtro percolador (16 L). Foram testadas as razões de recirculação de 150, 100 e 50% e TDH de 9, 7 e 5 h, com vazão de aeração constante de 10 L min-1. Para avaliação do sistema foram analisados temperatura do líquido, pH, alcalinidade total, alcalinidade do bicarbonato, ácidos voláteis, oxigênio dissolvido, demanda química de oxigênio, nitrogênio total Kjeldahl, nitrogênio amoniacal, nitrito, nitrato e fósforo total. O sistema combinado atingiu eficiências de remoção de matéria orgânica de 99%, 98% e 98% para as taxas de recirculação de 50, 100 e 150%, respectivamente. Sob as mesmas taxas de R, as eficiências de remoção de NTK resultaram em 96, 89 e 87%, e as eficiências de remoção de PT em 78, 82 e 77%, respectivamente. Quando operado com TDH de 9, 7 e 5 h, o sistema combinado atingiu eficiências de remoção de 86, 96 e 98% para DQO, 90, 93 e 94% para NTK e 74, 89 e 95% para PT, respectivamente. A melhor condição operacional foi estabelecida experimentalmente com a taxa de recirculação de 150% e TDH de 5 h. Entretanto, após a validação experimental por DCCR (por desejabilidade), a condição operacional ótima resultou na taxa de recirculação de 123% e TDH de 5 h para eficiências de remoção de NTK e TP.

Palavras-chave: Processos combinados; Remoção de nitrogênio; Adsorção de fósforo

1 INTRODUCTION

Sanitary sewage is composed of carbohydrates (glucose, fructose), organic waste (food and human waste), and nutrients (nitrogen and phosphorus) (JORDÃO; Pessoa, 2011). Its composition may change mainly due to water use influenced by climate, economic situation, and population habits, among other factors.

Nitrogen and phosphorus are widely used in agriculture considering they are essential for developing cultures. However, these nutrients lead to eutrophication discarded into water bodies at high concentrations. In water bodies, the found nitrogen forms are molecular nitrogen (N2), organic nitrogen (dissolved and suspended), ammonia, nitrite (NO2-), and nitrate (NO3-); and phosphorus are organic phosphates, orthophosphates, and polyphosphates that are hydrolyzed into orthophosphates (CHERNICHARO, 2006; SPERLING, 2006).

Studies with combined processes have been conducted to evaluate the removal of these nutrients to meet the standards established in the current environmental legislation, aiming for operation simplicity and a better cost/benefit ratio. These processes include aerobic and anaerobic reactors that have advantages such as removal of organic matter in terms of COD up to 70%, low energy cost on conventional aerobic reactors, lower sludge production, lower requirements by area of implantation, reduced construction and operating costs, and improvement in overall process efficiency (LIER *et al.*, 2015; KAVOUSI; BORGHEI, 2023).

In addition to the advantages mentioned above, the combination of anaerobic-aerobic systems makes it possible for effluent discharge with quality to achieve the standards established in Resolutions 357 (BRASIL, 2005) and 430 (BRASIL, 2011) of the Conselho Nacional do Meio Ambiente (CONAMA), improvement of the nitrification and denitrification processes, among others (ABREU; ZAIAT, 2008, FOCO; NOUR, 2014, OLIVEIRA NETTO, 2007). However, these systems present an achievement of a satisfactory nutrient removal performance, so advanced process control strategies and supplemental treatment technologies may be necessary, such as adsorption (UGWUANYI *et al.*, 2024).

Adsorption provides high removal efficiency, flexibility in selecting adsorbents, availability and environmentally friendly character, and simplicity in operation. This process can also be employed as a post-treatment to polish effluent from biological treatment or as a single method for targeted nutrient removal (WANG *et al.*, 2015; RATHI; KUMAR, 2021; UGWUANYI *et al.*, 2024).

Thus, incorporating additional filter units containing adsorbents has emerged to increase nutrients removal efficiency, especially phosphorus, in aquatic matrices. Several adsorbent materials, such as synthesized metal oxides/hydroxides, layered double hydroxides, carbonate minerals, clay minerals, zeolites, porous silica, activated carbon and biochar, polymers, and bio-derived materials, and industrial wastes, have been evaluated for their ability to remove phosphorus, involving the ion exchange with surface hydroxyl groups (BACELO *et al.*, 2020).

Therefore, advances in developing nutrient removal methods and the search for efficient phosphorus adsorption materials are still necessary. For that, the performance of a system composed of sequential anaerobic-aerobic reactors followed by a trickling filter was evaluated in removing nitrogen and phosphorus in a liquid phase recirculation and subjected to hydraulic retention time (HRT) and recirculation rate (R) variations.

2 MATERIAL AND METHODS

2.1 Treatment System and Substrate

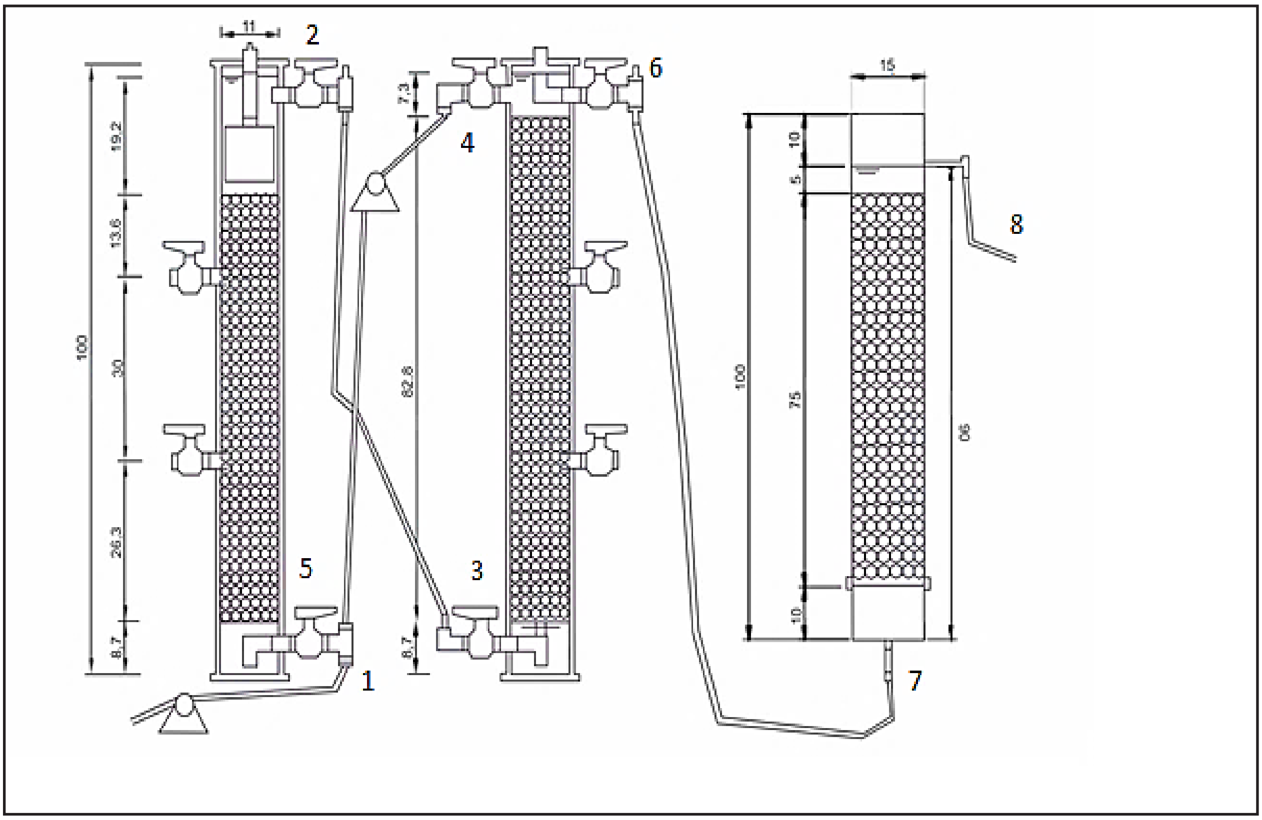
The treatment system consisted of three sequential reactors, starting with an anaerobic reactor (20 L and 110 mm diameter), followed by an aerobic reactor (19 L and 110 mm diameter), both fixed beds, and an adsorption column (16 L and 150 mm diameter) (Figure 1).

For biomass immobilization, corrugated polyvinyl chloride (PVC) pipes with a diameter of 0.01 m, length of 0.025 m, and surface area of 0.0012 m^2 were used in the reactors. The effluent of the aerobic reactor was recirculated to the anaerobic

reactor to promote nitrogen removal. The aerobic reactor has presented an aeration flow rate of 10 L min^{-1}, in which porous stones were used to promote better air injection and diffusion into the liquid phase.

A trickling filter (PVC column) was set up and installed as a post-treatment of the combined system. The upflow filter was operated on a filtration rate ranging from 371 to 668 m^3 m^{-2} d^{-1}, and the hydraulic retention time (HRT) from 5 to 9 h. The filter was filled with activated red ceramic fragments prepared by calcining at 200 °C for 2 hours, dolomite lime solution washing (10 g L^{-1}), immersion in water for 24 h, and drying at 100 °C for 24 h. The washing was done every 30 days to reactivate the adsorption on the material.

Figure 1 – Schematic layout of the treatment system



Source: Authors (2024). Legend: 1. Anaerobic reactor input; 2. Anaerobic reactor output; 3. Aerobic reactor input; 4. Effluent recirculation outlet; 5. Effluent recirculation input; 6. Aerobic reactor output; 7. Adsorption column inlet; 8. Adsorption column output

Synthetic effluent was prepared according to Torres (1992) for the system feeding on an average COD value of 500 mg L^{-1}.

2.2 Steps and System Monitoring

The experiment was divided into three steps, as follows: a) initial step – HRT of 8 h and R of 100% for 26 days, i.e., without variations in the operating conditions; b) step 1 – HRT variation 9, 7 and 5 h, and R maintained at 100% for 63 days; c) step 2 - HRT of 7 h and R variation at 150, 100, and 50% for 63 days. A summary of the system operation steps is presented in Table 1.

Table 1 – Operating steps of the system

Step	Operation period (d)	HRT (h)	Flow rate (L h^{-1})	Recirculation rate (%)	Number of sampling profiles
Initial	0 a 26	8	2.5	100	2
1	27 a 90	9 7 5	2.2 2.9 4.0	100	6
2	91 a 154	7	2.9	150 100 50	6

Source: Authors (2024)

The physical-chemical parameters were determined for samples of the influent (B), effluent from the anaerobic reactor (1), effluent from the aerobic reactor (2), and effluent from the trickling filter (3). Their respective methods of analysis, method numbers, and references are presented in Table 2. The parameters were determined in duplicate during the operation.

Table 2 – Physical-chemical parameters determined in the influent and effluent samples characterization

Parameters	Analysis method	Method Number	Reference
LT (°C)	Potentiometric	-	-
pH	Potentiometric	4500_H^+	APHA (2017)
DO (mg L^{-1})	Polarographic	4500_O	APHA (2017)
BA (mg$CaCO_3$ L^{-1})	Titrimetric	-	Ripley, Boyle e Converse (1986)
VA (mgHAc L^{-1})	Titrimetric	-	Dillalo e Albertson (1961)
COD (mg L^{-1})	Spectrophotometric	5220_D	APHA (2017)
BOD (mg L^{-1})	Respirometric	5210 D	APHA (2017)
TKN (mgN-NTK L^{-1})	Titrimetric	4500_NTK	APHA (2017)
TAN (mgN-NH_4^+ L^{-1})	Titrimetric	4500_NH_4^+	APHA (2017)
Nitrite (mgN-NO_2^- L^{-1})	Spectrophotometric	4500_NO_2	APHA (2017)
Nitrate (mgN-NO_3^- L^{-1})	Spectrophotometric	4500_NO_3	APHA (2017)
TP (mgPO_4^{3-} L^{-1})	Spectrophotometric	4500_P	APHA (2017)
TS (mg L^{-1})	Gravimetric	2540_B	APHA (2017)
TSS (mg L^{-1})	Gravimetric	2540_G	APHA (2017)

Source: Authors (2024). Legend: LT - Liquid Temperature; DO - Dissolved Oxygen; BA - Bicarbonate alkalinity; VA - Volatile Acids; COD - Chemical Oxygen Demand; BOD - Biochemical Oxygen Demand; TKN - Total Kjeldahl Nitrogen; TAN – Total Ammonia nitrogen; TP - Total phosphorus; TS - Total solids; TSS - Total Suspended Solids

2.3 Statistical Planning

The selected parameters (hydraulic retention time HRT and recirculation rate R) were investigated using central composite rotational design method (CCRD, star type, α = 45°) due to its suitability for quadratic surface fitting, effective parameters optimization with a minimum number of experiments, and interaction analysis between these parameters (Kaçan and Kütahyalı, 2012). This design was composed of factorial planning (2^2) with levels (-1 and +1) and axial points (-1.414 and +1.414), as well as repetition at the central point (0), resulting in 10 trials. The range and levels of the independent numerical variables in terms of actual and coded values are shown in Table 3. Commercial software *Statistica*® was used to carry out the modeling and planning.

Table 3 – Levels of experimental factors used in the design

Experimental / Unit Factors	Coded Variables				
	-α	-1	0	+1	+α
Factor 1 (HRT) (h)	5	5.6	7	8.4	9
Factor 2 (R) (%)	50	64.6	100	135.4	150

Source: Authors (2024). Legend: $α= (2^N)^{1/4}$ = 1,414; N= number of independent variables

Table 4 shows the DCCR statistical planning matrix containing the two investigated variables, their respective levels of the factors that were analyzed, and the coded and decoded variables.

The resulting data were regressed in order to derive a suitable equation for each response. All variable parameters and their interactions were considered in a model to obtain the greatest efficiency on nitrification and denitrification efficiencies and total nitrogen and total phosphorus removal. The physical-chemical analyses were performed on the system being operated from the highest HRT value to the lowest value as not to cause “stress” to the microorganisms, compromising the system performance.

Table 4 – Experimental design matrix

Test	Coded Variables		Decoded Variables	
	HRT (h)	R	HRT (h)	R (%)
1	-1	-1	5.6	64.6
2	+1	-1	8.4	64.6
3	-1	+1	5.6	135.4
4	+1	+1	8.4	135.4
5	-1.414	0	5	100
6	+1.414	0	9	100
7	0	-1.414	7	50
8	0	+1.414	7	150
9	0	0	7	100
10	0	0	7	100

Source: Authors (2024)

The experimental validation of the proposed models was performed in triplicate in the optimum condition for nitrogen and phosphorus removal, as determined by the desirability test. Then, the means, standard deviations, and coefficients of variation of the data obtained in the model validation were calculated.

3 RESULTS AND DISCUSSION

The summary of the overall removals for COD, TKN, TAN, TN, TP, nitrification, and denitrification is shown in Table 5.

Despite no significant differences in denitrification results (p-value > 0.05), the system has shown better performance on nitrification and TKN and TN removal efficiencies when operated at R of 150% and HRT of 7 h.

When observing the variation of the recirculation rate concerning the COD removal, the system has presented damping capacity despite the gradual increase of the volumetric hydraulic loading rate and the applied organic volumetric loading rate. The removal efficiency of COD varied from 95-99% for variations of the recirculation rate and 81 to 98% for variations of HRT during the operation.

Table 5 – Summary of removals of COD, nitrogen forms, TP, and nitrification, and denitrification in the Steps I and II

Parameter (%)	Recirculation ratio (at HRT = 7 h)			HRT (at R = 100 %)		
	50%	**100%**	**150%**	**5 h**	**7 h**	**9 h**
COD raw samples	99	98	98	98	96	86
COD filtered samples	97	93	95	98	93	81
TKN	87	89	96	94	93	90
TAN	94	95	93	93	95	80
Nitrification	80	86	92	88	87	92
Denitrification	84	82	85	84	82	79
TN	74	72	82	79	76	72
TP	77	82	78	95	89	74

Source: Authors (2024). Legend: COD: Chemical Oxygen Demand; TKN: Total Kjeldah Nitrogen; TAN: Total ammonia nitrogen: TN: Total nitrogen.; TP: Total Phosphorus

Lower COD removal efficiencies were observed in the treatment of different wastewater by single UASB-type reactors, as reported by Yaya-Beas *et al.* (2016), who verified COD removal efficiencies varying from 37 to 62% in a UASB-type reactor (29 L) operated with HRT between 3 and 5 h, treating sanitary sewage; and Niwa *et al.* (2016), who observed removal efficiency of 91% in a UASB type reactor (4550 m^3 d-1) treating industrial effluent. By comparing our results to those of other configurations of reactors, our results were greater than those obtained by Tawfik *et al.* (2012), who achieved 72% COD removal when treating sanitary sewage in an up-flow anaerobic sponge reactor (UASR, 1.3 L) followed by a moving bed biofilm reactor (MBBR, 1.65 L); and Wosiack *et al.* (2015), who verified 80% of COD removal efficiency in a continuous flow structured-bed reactor (9 L) treating animal feed industry effluent.

Regarding the nitrogen forms (Table 4), the operational condition with R 150% presented better nitrification (92%), denitrification (85%), TKN (96%), and TN (82%) removal efficiencies, indicating that the higher recirculation rate improved the system performance. Oliveira Netto and Zaiat (2012) verified TAN removal efficiency of 95% in an anaerobic-aerobic fixed bed reactor (6.8 L) operated with recirculation rates of 50% and 150% and HRT of 6, 8, and 10 h in the treatment of sanitary sewage. Araújo and Freitas (2014) observed 25% of removal in a UASB-type reactor followed by an aerated biofilter submerse (BFAS, 14 L) treating synthetic sewage, and Jiang *et al.* (2016) verified 83% TAN removal efficiency in sequential anaerobic/aerobic/anoxic reactors (total volume 9 L) treating sanitary sewage.

Higher TKN (94%) and TN removal (79%) efficiencies were achieved when the combined system was operated with an HRT of 5 h and TAN removal (95%) efficiency with an HRT of 7 h. The higher HRT was more efficient for nitrification, while denitrification was better when the HRT was lower, even though it was complete.

Removal efficiencies above 74% can be observed during the operational conditions regarding the total phosphorus, probably promoted by the presence of calcium silicate in the support media of the trickling filter that promoted the adsorption of this pollutant.

The values of the experimental design and the response variables regarding nitrogen and phosphorus removal efficiencies obtained in the experimental steps are shown in Table 6.

The results indicate efficiency variations among the conditions evaluated (Table 6). Tests 7, 1, and 2 presented lower overall removal efficiencies for nitrification, denitrification, total nitrogen, and total phosphorus, respectively. Tests 9, 8, and 10 presented higher overall removal efficiencies for nitrification, denitrification, total nitrogen, and phosphorus. Considering the values obtained by ANOVA and the estimated effects, second-order coded models were elaborated for the response variables, considering only the significant parameters (Table 6).

Table 6 – Experimental design and response variables

Test	Real Factor Levels		Variable responses (%)			
	HRT (h)	R (%)	Nitrification	Denitrification	TN	TP
1	9	100	86 (1)	75 (4)	62 (5)	73 (17)
2	8.4	135.4	84 (9)	80 (9)	69 (12)	10 (4)
3	8.4	64.6	92 (3)	79 (2)	73 (4)	69 (9)
4	7	150	92 (4)	85 (2)	82 (2)	78 (8)
5	7	100	86 (1)	82 (2)	72 (2)	82 (10)
6	7	100	87 (1)	82 (1)	76 (3)	92 (4)
7	7	50	80 (5)	84 (3)	74 (3)	77 (10)
8	5.6	135.4	82 (2)	92 (3)	88 (3)	95 (4)
9	5.6	64.6	94 (1)	84 (1)	81 (1)	83 (4)
10	5	100	88 (0,3)	84 (1)	79 (1)	95 (1)

Source: Authors (2024). Legend: Variable responses: average efficiency of profiles performed; (standard deviation)

The R^2 values of 0.93, 0.70, 0.78, and 0.75 for the statistical models 1, 2, 3, and 4, respectively, indicated that these models explain variations in the removal efficiencies (Table 7) even in biological continuous systems.

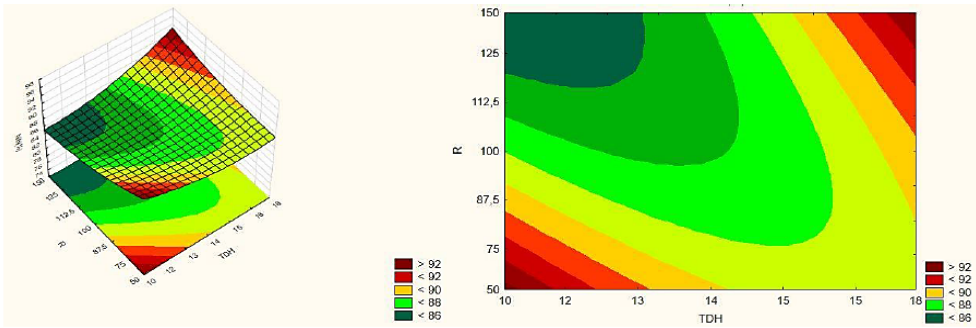
Table 7 – Mathematical models and determination coefficients (R^2) of the adjusted models

Variable responses (%)	Models	R^2 (%)
Nitrification (model 1)	$88.07+1.62(HRTxR)$	93
Denitrification (model 2)	$81.33-2.78(HRT)+2.90(HRT)^2+1.43(R)+1.96(R)^2$	70
Total Nitrogen (model 3)	$73.71-2.84(HRT)+4.72(HRT)^2+2.99(R)+2.27(R)^2$	78
Total Phosphorus (model 4)	$89.13-17.73(HRT)-8.16(HRT)^2-7.24(R)-8.24(R)^2-20.58(HRTxR)$	75

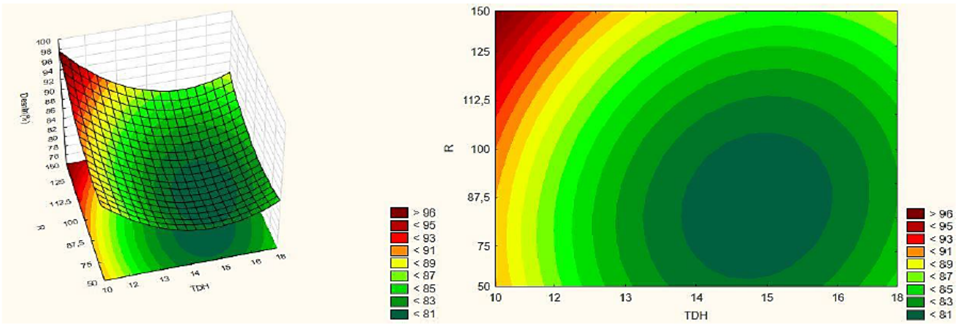
Source: Authors (2024)

The response surfaces and the level curves for nitrogen and phosphorus removal can be observed in Figure 2.

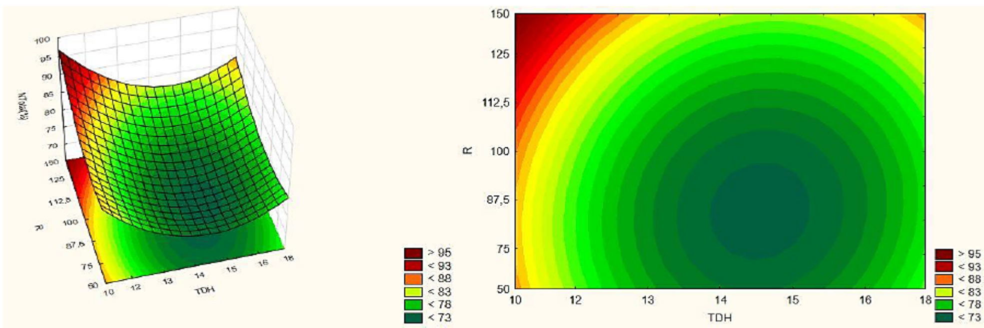
Figure 2 – Response surfaces and level curves for (a) nitrification; (b) denitrification; (c) total nitrogen; (d) phosphorus



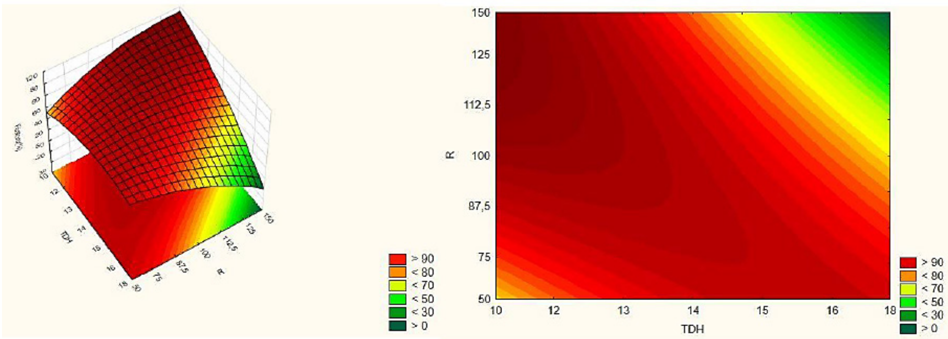
(a)



(b)



(c)



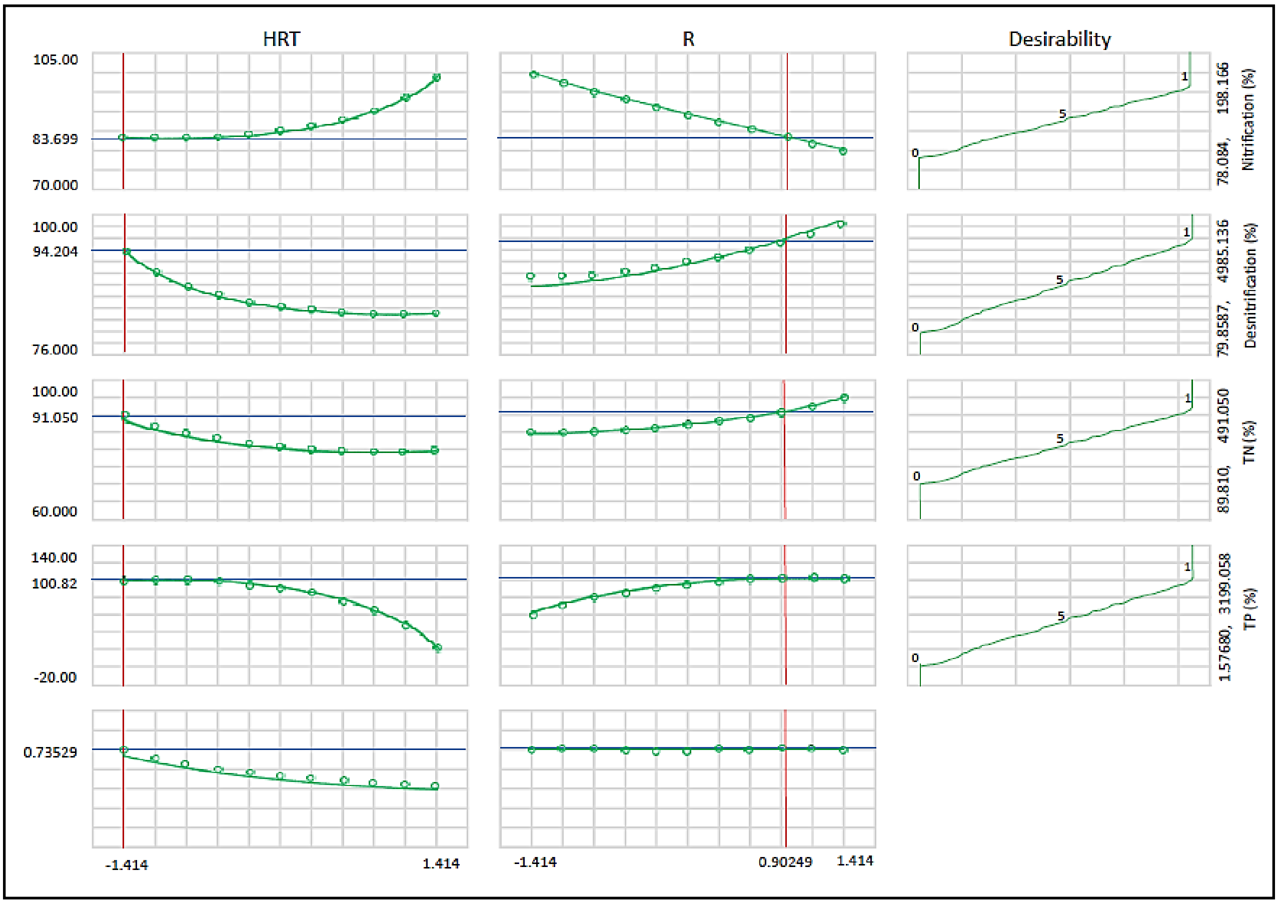
(d)

Source: Authors (2024)

The results showed that nitrification resulted in greater efficiencies of the nitrification process with decreasing HRT and recirculation rate. For the denitrification process, the highest removal efficiencies occur when there is a decrease in HRT and an increase in the recirculation rate. Regarding the removal of phosphorus, the greatest efficiencies are obtained when the HRT decreases and recirculation increases.

The validation of the models was performed for the optimal condition found in the desirability, in which all the values of the response variables were crossed for the selection of the optimal point. In order to achieve desirability (Figure 3), HRT and recirculation rate (R) parameters were used.

Figure 3 – Analysis of the desirability to find the optimal condition



Source: Authors (2024)

After this definition, the optimal condition was analyzed experimentally to compare the experimental values to the predicted ones from the statistical model calculations.

The optimum condition defined the coded value of -1.414 for HRT and 0.90249 for R, corresponding to HRT of 5 h and a recirculation rate of 123% (Table 8).

Table 8 – Predicted values calculated in the found model

Test	Coded Variables		Decoded Variables		(Answers calculated in model %) [Experimental value found%]			
	HRT (h)	R	HRT (h)	R (%)	Nitrification (R^2 0.93)	Denitrification (R^2 0.70)	TN (R^2 0.78)	TP (R^2 0.75)
1	-1	-1	5.6	64.6	(90) [94]	(88) [84]	(80) [81]	(77) [83]
2	+1	-1	8.4	64.6	(86) [92]	(82) [79]	(75) [73]	(83) [69]
3	-1	+1	5.6	135.4	(86) [82]	(90) [92]	(86) [88]	(94) [95]
4	+1	+1	8.4	135.4	(90) [84]	(85) [80]	(81) [69]	(27) [10]
5	-1.414	0	5	100	(88) [88]	(91) [84]	(87) [79]	(98) [95]
6	+1.414	0	9	100	(88) [86]	(83) [75]	(80) [62]	(48) [73]
7	0	-1.414	7	50	(88) [80]	(83) [84]	(74) [74]	(83) [77]
8	0	+1.414	7	150	(88) [92]	(87) [85]	(82) [82]	(62) [78]
9	0	0	7	100	(88) [86]	(81) [82]	(74) [72]	(89) [82]
10	0	0	7	100	(88) [87]	(81) [82]	(74) [76]	(89) [92]
11*	-1.414	0.902	5	123	(86) [82]	(94) [88]	(91) [75]	(91) [76]

Source: Authors (2024)

The statistical found model can be applied to any value in the studied range: R of 50, 100, and 150% and HRT of 5, 7, and 9 h, i.e., in these intervals, any value can be calculated in the model, not only the tested ones. Assay 11 is the optimum condition found by the desirability. For this test, the predicted values calculated by the model were 86, 94, 91, and 91% for nitrification, denitrification, total nitrogen, and total phosphorus, respectively. The experimental values obtained in the laboratory were 82, 88, 75, and 76% for nitrification, denitrification, total nitrogen, and total phosphorus, respectively.

4 CONCLUSIONS

The combined system has achieved the highest global removal efficiencies when operated at HRT of 7 h with 96% for TKN for R 150%, 95% for TAN for R 100%, and 82% for TP for R 100%. COD removal efficiencies resulted in 99% for raw and 97% for filtered samples, with a recirculation rate of 50%.

When the system was operated with a recirculation rate of 100%, the maximum overall removal efficiencies were 94% for TKN and 95% for TP with HRT 5 h, and 95% for TAN with HRT 7. The best results for COD removal efficiencies were 98% for raw and filtered samples with HRT 5 h.

Through the desirability test, the optimal condition of the operation was on an HRT of 5 h and a recirculation rate of 123%. When testing this condition in the laboratory, it was possible to validate the predicted statistical models. The R^2 values obtained with the statistical models for nitrification, denitrification, and removal of total nitrogen and total phosphorus indicated that the models explained 93, 70, 78, and 75% of the variations in removal efficiencies, respectively.

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