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Experimental Investigation of Mass-Transfer Processes in The Treatment of Phenolic Wastewater Using A Pilot-Scale Bubble Column Extractor

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Abstract: This study investigates the extraction-based treatment of phenol-containing wastewater in a pilot-scale bubble column extractor. Experiments were conducted to determine the stage efficiencies of the apparatus as functions of the wastewater-to-extractant flow-rate ratio, gas velocity, and phase-mixing time. The corresponding theoretical efficiencies were also calculated and compared with the measured values. The experimental findings were in close agreement with the theoretical predictions. On this basis, operating parameters suitable for the design of a bubble column extractor are recommended.

Keywords: Bubble column extractor, gas flow rate, phenol, wastewater, liquid flow rate, gas orifices, orifice spacing, phase mixing, orifice diameter, internal mixing zone, external mixing zone, stage efficiency.

INTRODUCTION:

Extraction-based wastewater treatment separates harmful dissolved chemicals from water by transferring them into a suitable solvent (extractant). The method is commonly applied to remove phenols, oils, and heavy metals.

The extraction treatment of industrial wastewater is based on the distribution of a contaminant between two mutually immiscible liquid phases and on its relative solubility in those phases [1].

This approach is widely used to remove organic contaminants, particularly phenols from wastewater generated during the processing of solid fuels such as coal and peat.

Liquid-liquid extraction is economically attractive when the concentration of organic contaminants is high or when the recovered compound has substantial value. For example, extraction provides effective treatment when the phenol content of wastewater exceeds 0.2 g/L [2].

Experimental Setup and Research Object

The research object was a pilot unit of the bubble column extractor developed by the authors (Figures 1 and 2). The gas-cushion relationship given in Equation (1), which had been proposed in the preceding theoretical analysis, was also used [1-3].

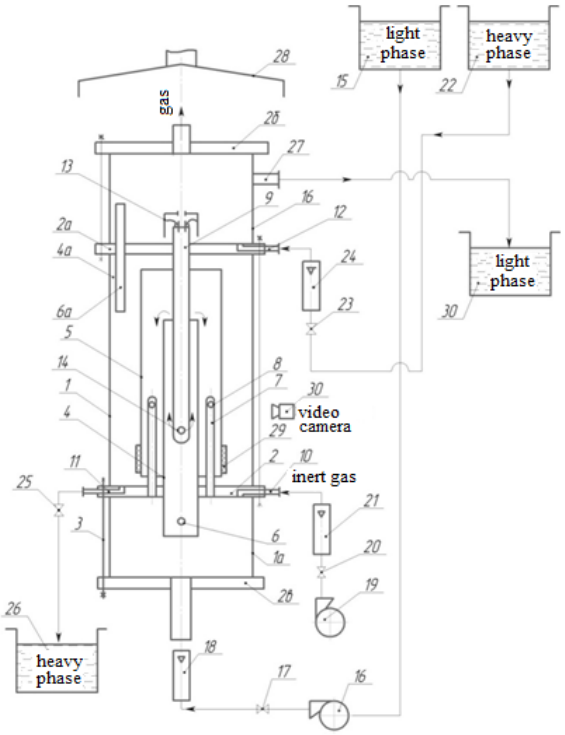

Figure 1. Schematic diagram of the pilot unit.

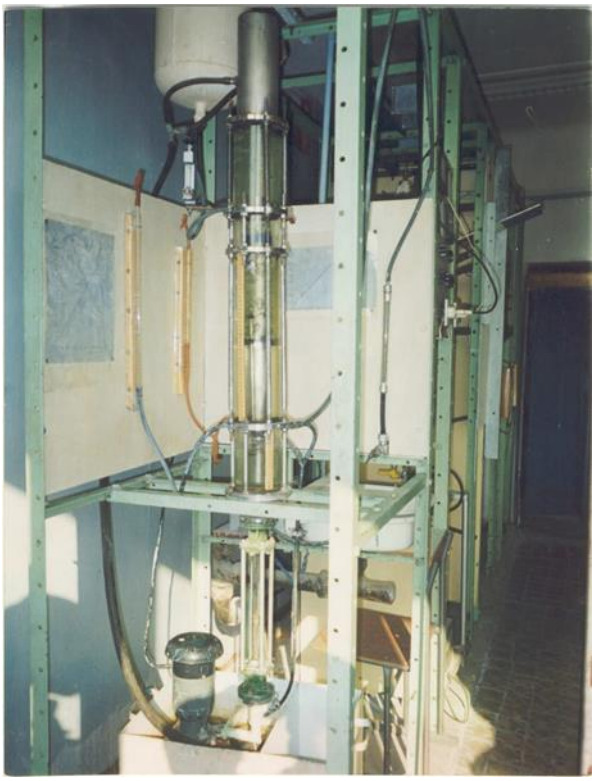

Figure 2. General view of the pilot unit.

1 - glass column; 2, 2a, 2b, 2c - flanges; 3 - tie rod; 4 - internal sparging pipe; 5 - external sparging pipe; 6 - gas orifice; 7 - gas supply line; 8 - gas orifice; 9 - heavy-liquid line; 10 - gas inlet channel; 11 - heavy-liquid outlet channel; 12 - heavy-liquid inlet channel; 13 - cap; 14 - heavy-liquid overflow opening; 15 - light-liquid tank; 16 - pump; 17 - valve; 18 - rotameter; 19 - compressor; 20 - gas valve; 21 - rotameter; 22 - heavy-liquid tank; 23 - tap; 24 - rotameter; 25 - tap; 26 - heavy-liquid tank; 27 - light-liquid outlet channel; 28 - hood; 29 - filter; 30 - video camera.

The study addressed the treatment of phenolic wastewater generated in the phenol unit of Fergana Oil Refinery LLC. A pilot apparatus based on the newly developed multistage bubble column extractor (Uzbekistan Patent No. IAP 06714, January 31, 2022) was used. A readily available, low-cost solvent containing 25% toluene and 75% kerosene served as the extractant. The tests were carried out in the laboratory of Workshop No. 10 at the refinery.

RESULTS AND DISCUSSION

Earlier hydrodynamic tests on the pilot extractor established the gas-cushion height [4], gas holdup in the mixing zones [5], and the distribution of gas flow between the internal and external mixing zones [6]. The present work focuses on identifying operating conditions that provide effective mass transfer among the heavy liquid phase, light liquid phase, and gas.

Phenolic wastewater samples from the phenol unit were delivered to the central laboratory of Workshop No. 10. Phenol concentration was measured by the photometric method specified in O'zO'U 07.0778:2018. The initial phenol concentration was X_b*= 0,3 mg/l*.

The photometric method for determining the mass concentration of volatile phenols (PND F 14.1:2.105-97) involves distilling phenols from an acidified water sample, reacting them with 4-aminoantipyrine in the presence of potassium hexacyanoferrate(III), and extracting the resulting colored compound with chloroform. The optical density of the extract was measured with a spectrophotometer at λ = 470 nm or with a photometer fitted with a filter whose transmission maximum lies between 460 and 490 nm. The stated measurement range is 0.002-0.3 mg/m³.

Under Sanitary Rules and Standards No. 0294-11 of the Republic of Uzbekistan, the maximum permissible

concentration (MPC) of phenol in discharged wastewater is $X_o = 0.001$ *mg/L*.

During the experiments, phenolic wastewater was supplied to the mixing zones at $Q_s = 0.23\text{-}0.39$ *m³/h* in increments of 0.08 m³/h.

At each fixed liquid flow rate, the gas flow rate was varied from $Q_g = 0.55$ to 0.75 m³/h in increments of 0.10 m³/h. The experiments were conducted at 22 °C. The wastewater-to-extractant flow-rate ratio was maintained at 4:1; accordingly, the extractant flow rate ranged from $Q_e = 0.06$ to 0.10 m³/h. Gas entered the internal mixing zone through four orifices of diameter d0 = 1.5 mm and the external zone through four orifices of diameter d1 = 1.0 mm. The orifice spacing was H_1 = 165 mm. The mixing time was 22 s at Q_s = 0.23 m³/h, 14 s at Q_s = 0.31 m³/h, and 6 s at Q_s = 0.39 m³/h. Surface tension was measured by the capillary-tube method, while viscosity was determined by rotational viscometry in the refinery's central laboratory. To determine the required number of stages, wastewater and extractant were passed repeatedly through the pilot unit until the phenol concentration fell from X_b = 0.3 mg/L to the MPC of 0.001 mg/L. The target was reached after four successive passes. A sample from each stage was analyzed in the central laboratory. The experiments were run in the following sequence. 1. At a constant wastewater flow rate of Q_s = 0.23 m³/h and a constant extractant flow rate of Q_e = 0.06 m³/h, the gas flow rate was varied from Q_g = 0.55 to 0.75 m³/h. The liquid phases were mixed for 22 s. The results are presented in Table 1.

Table 1. Results of phenolic wastewater treatment in the pilot-scale bubble column extractor.

Phases	Concentration	Stage I	Stage II	Stage III	Stage IV	Density, kg/m³ ρ	Viscosity, μ, Pa·s μ·	Surface tension σ, N/mσ	Interfacial tension σi, N/mσ
Gas flow Q_g = 0.55 m³/h; Q_s = 0.23 m³/h; Q_e = 0.06 m³/h; t = 22 s									
Aqueous	X_b	0.3	0.21	0.14	0.06	1000	1.28 × 10⁻³	71.2 × 10⁻³	33.4 × 10⁻³
	X_o	0.21	0.14	0.06	0.012				
Organic	Y_b	0	0.09	0.16	0.23	851	1.37 × 10⁻³	37.8 × 10⁻³	
	Y_o	0.09	0.16	0.23	0.288				
η	%	30	53	80	96	MPC not met			
Gas flow Q_g = 0.65 m³/h; Q_s = 0.23 m³/h; Q_e = 0.06 m³/h; t = 22 s									
Aqueous	X_b	0.3	0.16	0.1	0.05	1000	1.28 × 10⁻³	71.2 × 10⁻³	33.4 × 10⁻³
	X_o	0.16	0.1	0.05	0.007				
Organic	Y_b	0	0.14	0.2	0.25	851	1.37 × 10⁻³	37.8 × 10⁻³	
	Y_o	0.14	0.2	0.25	0.293				
η	%	46.6	66.6	83.3	97.6	MPC not met			
Gas flow Q_g = 0.75 m³/h; Q_s = 0.23 m³/h; Q_e = 0.06 m³/h; t = 22 s									

Aqueous	X_b	0.3	0.11	0.04	0.007	1000	1.28 × 10^{-3}	71.2 × 10^{-3}	33.4 × 10^{-3}
	X_o	0.11	0.04	0.007	0.0005				
Organic	Y_b	0	0.19	0.26	0.293	851	1.37 × 10^{-3}	37.8 × 10^{-3}	
	Y_o	0.19	0.26	0.293	0.2995				
η	%	67.3	86.6	97.6	99.83	MPC met			

The stage efficiencies were calculated from the data in Table 1. With a fixed mixing time of 22 s and Q_g = 0.55 m³/h, the overall efficiency of the four-stage pilot unit was:

$$\eta = \frac{X_b - X_o}{X_b} \cdot 100 = \frac{0,3 - 0,012}{0.3} = 96\% \quad (1)$$

At Q_g = 0.65 m³/h, the corresponding four-stage efficiency was:

$$\eta = \frac{Xb - X_o}{X_b} \cdot 100 = \frac{0,3 - 0,007}{0.3} \cdot 100 = 97,7\% \quad (2)$$

At Q_g = 0.75 m³/h, the four-stage efficiency increased to:

$$\eta = \frac{X_b - X_o}{X_b} \cdot 100 = \frac{0,3 - 0,0005}{0.3} \cdot 100 = 99,83\% \quad (3)$$

2. In the second operating mode, the wastewater flow rate was held at Q_s = 0.31 m³/h and the extractant flow rate at Q_e = 0.0775 m³/h, while Q_g was varied from 0.55 to 0.75 m³/h. The liquid-phase mixing time was 14 s. The experimental results are given in Table 2.

Table 2. Results of phenolic wastewater treatment in the pilot-scale bubble column extractor.

Phases	Concentration	Stage I	Stage II	Stage III	Stage IV	Density, kg/m³ ρ	Viscosity, μ, Pa·s μ·	Surface tension σ, N/mσ	Interfacial tension σi, N/mσ
Gas flow Q_g = 0.55 m³/h; Q_s = 0.31 m³/h; Q_e = 0.0775 m³/h; t = 14 s									
Aqueous	X_b	0.3	0.23	0.16	0.09	1000	1.28 × 10^{-3}	71.2 × 10^{-3}	33.4 × 10^{-3}
	X_o	0.23	0.16	0.09	0.02				
Organic	Y_b	0	0.07	0.14	0.21	851	1.37 × 10^{-3}	37.8 × 10^{-3}	
	Y_o	0.07	0.14	0.21	0.28				

η	%	23.3	46.6	70	93.3	MPC not met			
Gas flow Q_g = 0.65 m³/h; Q_s = 0.31 m³/h; Q_e = 0.0775 m³/h; t = 14 s									
Aqueous	X_b	0.3	0.18	0.12	0.07	1000	1.28×10^{-3}	71.2×10^{-3}	33.4×10^{-3}
	X_o	0.18	0.12	0.07	0.012				
Organic	Y_b	0	0.12	0.18	0.23	851	1.37×10^{-3}	37.8×10^{-3}	
	Y_o	0.12	0.18	0.23	0.288				
η	%	40	60	76.6	96	MPC not met			
Gas flow Q_g = 0.75 m³/h; Q_s = 0.31 m³/h; Q_e = 0.0775 m³/h; t = 14 s									
Aqueous	X_b	0.3	0.09	0.045	0.009	1000	1.28×10^{-3}	71.2×10^{-3}	33.4×10^{-3}
	X_o	0.09	0.045	0.009	0.0008				
Organic	Y_b	0	0.21	0.255	0.291	851	1.37×10^{-3}	37.8×10^{-3}	
	Y_o	0.21	0.255	0.291	0.2992				
η	%	70	85	97	99.73	MPC met			

The four-stage treatment efficiencies were then calculated from Table 2. At a constant mixing time of 14 s and Q_g = 0.55 m³/h, the efficiency was:

$$\eta = \frac{X_b - X_o}{X_b} \cdot 100 = \frac{0,3 - 0,02}{0.3} = 93,3\% \quad (4)$$

At Qg = 0.65 m³/h, the efficiency was:

$$\eta = \frac{X_b - X_o}{X_b} \cdot 100 = \frac{0,3 - 0,012}{0.3} = 96\% \quad (5)$$

At Q_g = 0.75 m³/h, it reached:

$$\eta = \frac{X_b - X_o}{X_b} \cdot 100 = \frac{0,3 - 0,0008}{0.3} = 99,73\% \quad (6)$$

3. In the third operating mode, the wastewater and extractant flow rates were held at Q_s = 0.39 m³/h and Q_e = 0.0975 m³/h, respectively, while Q_g was varied from 0.55 to 0.75 m³/h. The mixing time was 6 s. Even at the maximum gas flow rate of Q_g = 0.75 m³/h, the four-stage treatment efficiency was only 91.7%. Because this result did not satisfy the MPC requirement, the detailed data for this mode were not included in the dissertation.

Tables 1 and 2 show that the MPC was achieved in two operating modes. At a mixing time of 22 s and Q_g = 0.75 m³/h, the four-stage efficiency was 99.83%, leaving a residual phenol concentration of X_o = 0.0005 mg/L. At a mixing time of 14 s and the same gas flow rate, the efficiency was 99.7% and X_o = 0.0008 mg/L. Both residual concentrations were below the MPC of 0.001 mg/L.

For the industrial design, the higher-throughput mode was therefore selected: a mixing time of 14 s, a phenolic wastewater flow rate of Q_s = 0.31 m³/h, and

an extractant flow rate of Q_e = 0.0775 m³/h.

Relative to the existing benzene-based extraction process, the proposed bubble column extractor reduced extractant consumption by a factor of 1.4 and enabled the use of a locally available solvent.

Using the experimental results in Tables 1 and 2, the distribution coefficients for phenol extraction with the toluene-kerosene solvent were determined from Equation (1). The phenolic wastewater and extractant must be mixed until phase equilibrium is established before the distribution coefficient is evaluated.

In the tested bubble column extractor, the approach to equilibrium depends on the contact time in the liquid-phase mixing zones. The data in Table 1 were used to calculate the distribution coefficient as a function of gas velocity and mixing time [7].

For the first mode, the wastewater flow rate was V_{ya} = 0.23 m³/h and the extractant flow rate was V_d = 0.06 m³/h.

At Q_g = 0.75 m³/h, the gas velocities in the internal and external mixing zones were w_{gi} = 0.09 m/s and wgt = 0.08 m/s, respectively, and the average mixing time was t_{av} = 22 s.

$$m = \frac{C_e}{C_r} \cdot = \frac{(Y_b - Y_1) \cdot V_{er}}{Y_1 \cdot V_d} = \frac{(0,3 - 0,11) \cdot 0,23}{0,11 \cdot 0,06} = \frac{0,0437}{0,0066} = 6,6 \qquad (7)$$

For the second mode, V_{ya} = 0.31 m³/h, V_d = 0.0775 m³/h, Q_g = 0.75 m³/h, w_{gi} = 0.09 m/s, w_{gt} = 0.08 m/s, and t_{av} = 14 s.

$$m = \frac{C_e}{C_r} \cdot = \frac{(Y_b - Y_1) \cdot V_{er}}{Y_1 \cdot V_d} = \frac{(0,3 - 0,09) \cdot 0,31}{0,09 \cdot 0,0775} = \frac{0,0651}{0,007} = 9,3 \qquad (8)$$

The next stage of the analysis concerned the theoretical efficiency of each extractor stage. Stage efficiency was calculated from Equation (3), while the parameter A was determined from Equation (4). The volumetric mass-transfer coefficient K_{vy} was evaluated with Equation (5). First, the liquid-phase residence time in the mixing zones was calculated from the phase flow rates using Equation (6).

The combined volume of the internal and external mixing zones was calculated as follows [7]:

$$V_p = V_i + V_t = 0,0004 + 0,000485 = 0,000885\ m^3, \qquad (9)$$

The phenolic wastewater flow rate supplied to the unit was:

V_{ya} = 0.23/3600 = 0.000064 m³/s, while the extractant flow rate was V_d = 0.06/3600 = 0.000016 m³/s.

Substitution of these values into the following expression gives the liquid-phase residence time in the mixing zones [7]:

$$\tau = \frac{V_r}{V_{ya} + V_d} = \frac{0,000885}{0,000064 + 0,000016} = \frac{0,000885}{0,000080} = 11s \qquad (10)$$

The volume fraction of extractant in the phenolic wastewater was calculated from [7]:

$$\varphi = \frac{V_d}{V_{ya} + V_d} = \frac{0,000016}{0,000064 + 0,000016} = \frac{0,000016}{0,000080} = 0,2 \qquad (11)$$

The theoretical volumetric mass-transfer coefficient was obtained from [7]:

$$K_{vy} = 2,11 \cdot 10^{-6} \frac{\left(\omega_g^i \cdot \omega_g^T\right)^{0,25} \cdot \phi}{\tau \cdot \sigma^{0,8} \cdot \mu_{ya}^{0,8} \cdot \mu_d^{0,8}} s^{-1} \qquad (12)$$

The interfacial tension σ and the viscosities of the wastewater and extractant, μya and μd, used in this equation are listed in Tables 1 and 2.

At Q_g = 0.75 m³/h, the gas velocity was 0.09 m/s in the internal mixing zone and 0.08 m/s in the external zone. The liquid-phase residence time was 11 s. The wastewater and extractant flow rates were V_{ya} = 0.23 m³/h and V_d = 0.06 m³/h, respectively, and the mixing time was 22 s.

The distribution coefficient calculated above was m = 6.6. Substitution into Equation (13) gives:

$$K_{vy} = 2{,}11\cdot 10^{-6}\frac{(0{,}09\cdot 0{,}08)^{0{,}25}\cdot 0{,}2}{11\cdot 0{,}00334^{0{,}8}\cdot 0{,}00128^{0{,}8}} = \frac{0{,}00000015}{0{,}00053} = 0{,}0003c^{-1} \quad (13)$$

The phenol extraction term was calculated from the following relationship [7]:

$$A = \frac{(mV_я + V_d)\cdot V_p \cdot K_{vy}}{V_я \cdot V_d}$$

$$A = \frac{(6{,}6\cdot 0{,}000064 + 0{,}000016)\cdot 0{,}000885\cdot 0{,}0003}{0{,}000064\cdot 0{,}000016} = \frac{0{,}00000000135}{0{,}000000001} = 1{,}35$$

The efficiency of one extractor stage was then calculated from [7]:

$$\eta = 1 - e^{-A} = 1 - e^{-1{,}35} = 74\% \quad (14)$$

The overall efficiency of the four-stage unit was therefore:

$$\eta = 1 - e^{-A} = 1 - e^{-5{,}4} = 99{,}5\% \quad (15)$$

The experimental four-stage efficiency reported in Table 1 was η = 99.83%, which closely agrees with the theoretical prediction. The difference between the theoretical and experimental values was Δ=0.3%.

The volumetric mass-transfer coefficient was also calculated for the second operating mode.

Using the experimental data in Table 2, the theoretical stage efficiency was determined for this mode as well.

At Q_g = 0.75 m³/h, the gas velocities in the internal and external mixing zones were w_{gi} = 0.09 m/s and w_{gt} = 0.08 m/s, respectively. The distribution coefficient was m = 9.3, the wastewater flow rate was V_{ya} = 0.31 m³/h, the extractant flow rate was V_d = 0.0775 m³/h, and the mixing time was 14 s.

The liquid-phase residence time in the mixing zones was calculated as follows:

V_{ya} = 0.31/3600 = 0.000086 m³/s, while V_d = 0.0775/3600 = 0.0000215 m³/s.

$$\tau = \frac{V_r}{V_{ya} + V_d} = \frac{0{,}000885}{0{,}000086 + 0{,}0000215} = \frac{0{,}000885}{0{,}0001075} = 8{,}3s \quad (16)$$

The volume fraction of extractant in the phenolic wastewater was:

$$\varphi = \frac{V_d}{V_{ya} + V_d} = \frac{0{,}0000215}{0{,}000086 + 0{,}0000215} = \frac{0{,}0000215}{0{,}0001075} = 0{,}2 \quad (17)$$

The theoretical volumetric mass-transfer coefficient was:

$$K_{vy} = 2{,}11\cdot 10^{-6}\frac{(0{,}09\cdot 0{,}08)^{0{,}25}\cdot 0{,}2}{8{,}3\cdot 0{,}00334^{0{,}8}\cdot 0{,}00128^{0{,}8}} = \frac{0{,}00000015}{0{,}0004} = 0{,}000375c^{-1}$$

The phenol extraction term was:

$$A = \frac{(9{,}3\cdot 0{,}000086 + 0{,}0000215)\cdot 0{,}000885\cdot 0{,}000375}{0{,}000086\cdot 0{,}0000215} = \frac{0{,}00000000273}{0{,}0000000019} = 1{,}44$$

The efficiency of one extractor stage was:

$$\eta = 1 - e^{-A} = 1 - e^{-1,4} = 75\%$$

The overall efficiency of the four-stage unit was therefore:

$$\eta = 1 - e^{-A} = 1 - e^{-5,6} = 99,6\%$$

The experimental four-stage efficiency in Table 2 was η = 99.73%, again confirming the theoretical result. The difference between the theoretical and experimental values was Δ=0.13%.

Tables 1 and 2 report the experimental stage efficiencies, while the corresponding theoretical values were obtained from Equation (4.3). To make the agreement between the two data sets easier to assess, both are summarized in Table 3.

Table 3. Comparison of experimental and theoretical stage efficiencies (E = experimental, T = theoretical, Δ = difference).

#	Q_g = 0.75 m³/h, w_{gi} = 0.09 m/s, w_{gt} = 0.08 m/s, V_{ya} = 0.23 m³/h, V_d = 0.06 m³/h, t = 22 s											
1	Stage 1			Stage 2			Stage 3			Stage 4		
	E, %	T, %	Δ, %	E, %	T, %	Δ, %	E, %	T, %	Δ, %	E, %	T, %	Δ, %
2	67.3	74	9.9	86.6	93	7.3	97.6	98	4	99.83	99.5	0.3
	Q_g = 0.75 m³/h, w_{gi} = 0.09 m/s, w_{gt} = 0.08 m/s, V_{ya} = 0.31 m³/h, V_d = 0.0775 m³/h, t = 14 s											
3	70	75	7	85	93	8	97	98	1	99.73	99.6	0.13

The relationships derived from the experimental results are plotted in Figures 3 and 4.

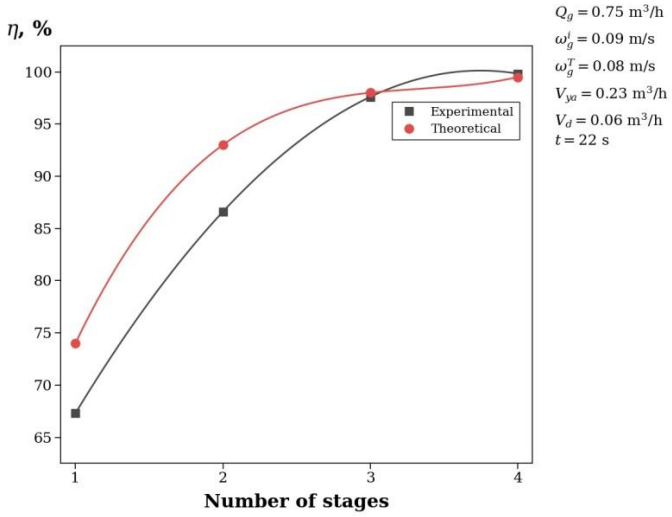

Figure 3. Stage efficiency as a function of the number of stages.

Regression equations: y = 10.85x + 60.70, R^2 = 0.8895; y = 8.15x + 70.75, R^2 = 0.8018

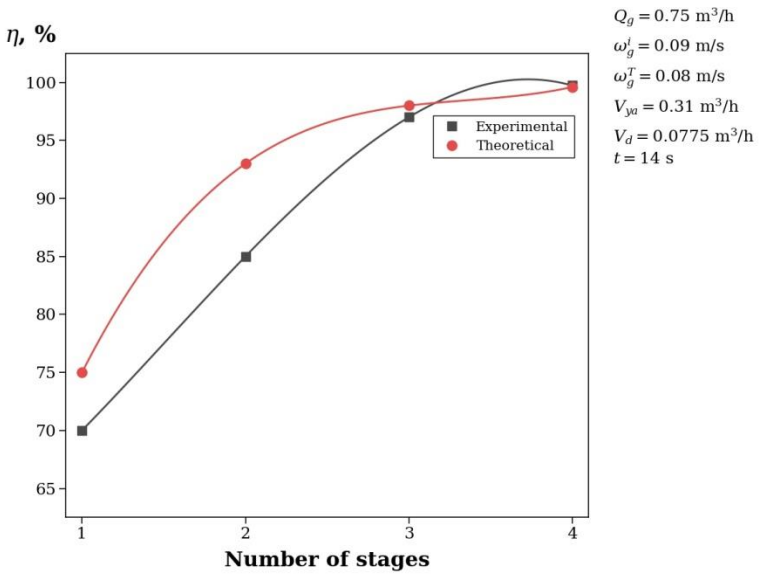

Figure 4. Stage efficiency as a function of the number of stages.

Regression equations: y = 10.119x + 62.635, R^2 = 0.9282; y = 7.88x + 71.70, R^2 = 0.8121

CONCLUSIONS

The extraction treatment of phenol-containing wastewater was investigated in a newly developed pilot-scale bubble column extractor. Experimental stage efficiencies were determined for different wastewater-to-extractant flow-rate ratios, gas velocities, and mixing times. Theoretical efficiencies were calculated independently and showed close agreement with the measurements. The results identify an operating mode that combines high treatment efficiency with greater throughput: Q_s = 0.31 m³/h, Q_e = 0.0775 m³/h, Q_g = 0.75 m³/h, and a mixing time of 14 s. These findings provide a practical basis for designing an industrial bubble column extractor for phenolic wastewater treatment.

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