EXTRACTION OF CAPROLACTAM IN TWO STAGES IN A MULTIPLE-STAGE BARBOTATION EXTRACTOR

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Annotation
This article presents the results of caprolactam extraction experiments carried out in a multi-stage bubbling extractor.

Keywords: Caprolactam, organic synthesis, extraction, organic solvent, lactam oil, benzene, inert gas, rotary disk extractor, stage, compressor, mixer, precipitation zone, nitrogen gas, heavy phase, light phase, equilibrium curve, concentration, coefficient distribution, efficiency.

Introduction

At present, the plant produces about 3.5 million tons of caprolactam per year. Uzbekistan produces 80,000 tons of caprolactam a year. Demand for this product is huge. Therefore, special attention is paid to the improvement of technological processes of caprolactam production, reconstruction of production lines and modernization of equipment. Caprolactam

production is an important branch of the modern organic synthesis industry. Improving caprolactam production as well as improving its quality is a requirement of the period [1-4].
The production of a high-quality caprolactam product depends on how the basic technological processes, including extraction processes, are organized [5-9].
The extraction process, which is carried out in two stages, allows for obtaining high-quality caprolactam. In the first stage, caprolactam is extracted from lactam oil using an organic solvent. In the second stage, caprolactam is separated from the organic solvent by water [10-17].

Methodology

As an organic solvent can be used an azeotropic mixture of benzene, ethylene trichloride, benzene and cyclohexane, carbon tetrachloride, chloroform and others. Benzene was used as an organic solvent in the laboratory of "Technological machines and equipment" of Fergana Polytechnic Institute.
In industry, rotor-disc extractors are often used for caprolactam extraction. We used a bubble extractor for this purpose [18-24]. This is because, even though rotor-disc extractors are widely used in the production of caprolactam, these devices imported from foreign companies have several shortcomings. The overall dimensions of such devices are large (up to 24 m in height), and high metal consumption, and changes in fluid consumption interfere with the accurate operation of the device. Most importantly, the construction is very complex, and operational reliability is low [25-37].

Fig.1. General view of the extractor

From the same point of view of operational reliability - we decided to use a bubble extractor. In such an extractor, the liquid is broken down into fine particles using inert gases that form a bubble. Another important advantage of bubble extractors is that they do not have moving parts or devices inside. The very compact bubble extractors [38-44] were made in the form of a vertical column (Fig. 1) using a simple standard compressor to deliver the inert gas to it. Unlike the multi-stage extractors known so far, a relatively new, improved design of the extractor was tested and experimented with in the laboratory. Each step of the vertical column, bordered by horizontal bars at the top and bottom, forms sedimentation zones in which bubble-tube mixers are run in parallel [45-50].

Results and discussion

Such a design allows experiments on one element of the mixer and the generalization of conclusions. We researched a 3-stage extractor model (internal diameter 104 mm, total height 1.6 m, made of glass tubes and compressed with metal flanges). The diameter of the extractor mixing elements is 33 mm; the height of each stage is 400 mm. The experiments were also carried out on a 6-stage experimental production unit with a diameter of 200 mm, a height of 4.5 m, a diameter of the mixing elements of 70 mm and a step height of 450 mm. Nitrogen gas was used as the mixing agent. The consumption of gases and liquids was determined using a rotameter. Samples were taken from each step of the extractor to determine the amount of caprolactam in the heavy and light phases. Concentrations of caprolactam in benzene and aqueous solutions were determined using Tsvet-102 chromatography. In lactam oil, there are two ways:

1) up to 40 mg / l - by spectrophotometric method;
2) When more than 40 mg / l, RDU was detected using a refractometer.

The “lactam oil-caprolactam-benzene” and “water-caprolactam-benzene” systems were studied at 40 ± 1 °C. The data obtained for the first system are given in Table 1.

Table 1. Equilibrium values of the system "Lactam oil-caprolactam-benzene" at 40 ± 10 C

The amount of caprolactam in lactam oil, by weight %	2,7	5,6	12	23,2	45,5
The amount of caprolactam in benzene, by weight %	0,68	1,41	3,95	9,31	20,67
Scattering coefficient	3,97	3,97	3,06	2,49	2,2

Table 2. Stable equilibrium values for the system "water - caprolactam - benzene" at a temperature of 40 according to the generally accepted method [5,6].

Aqueous layer composition, by weight %			Benzene layer composition, by weight %			Scattering coefficient
Caprolactam	Water	gasoline	Caprolactam	Water	gasoline	
0,80	99,2	0,1	0,12	0,11	99,67	6,7
2,50	97,5	0,1	0,47	0,13	99,40	5,3
4,82	95,1	0,1	1,11	0,16	98,73	4,4
8,9	91,1	0,1	2,3	0,22	97,48	3,9
13,1	86,9	0,1	3,7	0,29	96,01	3,5
19,2	80,7	0,1	5,8	0,46	93,74	3,3
29,5	69,7	0,8	10,8	0,83	88,7	2,8
35,8	62,7	1,5	13,2	1,17	85,63	2,7
41,5	55,8	2,7	16,7	1,70	81,60	2,5
47,8	53,3	5,5	22,1	2,50	75,40	2,2

It can be seen that the water-caprolactam-benzene system is consistent with stable equilibrium values with a difference of almost ± 5%.

Based on the data in Tables 1 and 2, we construct stable equilibrium lines in the right-angled coordinate system. Through them, it will be possible to graphically determine the required number of steps of extraction. [3].

In order to measure the efficiency of the steps of the extraction process, the steps were determined according to the method described in C.O.U. [4].

The C.O.U.obtained in the benzene and aqueous extraction processes of the extractor stages are given in Tables 3 and 4.

Table 3. Results of benzene extraction experiments on a three-stage extractor model of caprolactam

S/N №	consumption, 1/hour			W, m^3/m^2, Hour	Caprolactam concentration by steps, g/l												Steps C.O.U.		
					1- leap				2- leap				3- leap				1- leap	2- leap	3- leap
					in the beginning		in the end		in the beginning		in the end		in the beginning		in the end				
	V_{ear}	V_{or}	V_a		X_1^6	y_1^6	X_1^0	y_1^0	X_2^6	y_2^6	X_2^0	y_2^0	X_3^6	y_3^6	X_3^0	y_3^0			
1	85	30	77	13,6	2010	87	1040	224	1040	21,1	320	87	320	0	91	21,1	86	100	100
2	120	40	77	18,9	2090	87	1020	256	1020	21,1	344	87	344	0	116	21,1	93,5	97	91
3	180	65	77	29,0	2090	69,6	986	224	986	20,2	358	69,6	358	0	139	20,2	91	91	88
4	85	30	154	13,6	2010	88	985	232	885	21,1	232	88	232	0	91	21,1	90	100	100
5	120	44	154	19,3	2090	69,6	874	249	874	22	240	69,6	240	0	84	22	100	100	100
6	180	70	154	29,5	2090	69,6	918	256	918	22	259	69,5	258	0	83,8	22	95	99	100

Table 4. Results of experiments on aqueous extraction of caprolactam in a 3-stage extractor laboratory model.

S/N N	consumption, 1/hour			W, m^3/m^2, Hour	Caprolactam concentration by steps, g/l												Steps C.O.U.		
					1- leap				2- leap				3- leap				1- leap	2- leap	3- leap
					in the beginning		in the end		in the beginning		in the end		in the beginning		in the end				
	V_{ear}	V_{or}	V_a		X_1^6	y_1^6	X_1^0	y_1^0	X_2^6	y_2^6	X_2^0	y_2^0	X_3^6	y_3^6	X_3^0	y_3^0			
1	63	21	220	11	0	46,6	82,5	18,7	82,5	95,0	202	46,6	202	166	400	95,2	100	100	100
2	60	22	450	10,6	0	43,6	68,5	13,7	68,5	90,2	202	43,6	202	166	400	90,2	100	100	100
3	45	15	220	7,1	0	42,1	68,4	13,7	68,4	89,0	202	41,1	202	166	389	89	100	100	100
4	246	80	304	38,5	0	37,0	63,0	11,2	63	81,7	190	38,9	190	155	372	81,7	97	98	100
5	184	60	304	28,8	0	36	57	9,5	57	76,2	169	34,5	169	152	352	76,2	100	100	100
6	123	40	304	19,2	0	41	60	10,3	60	88	193	41	193	170	394	88	100	100	100

It can be seen from the tables that even at very high specific flow rates of liquids (38.5 m3 / m2 h) the C.O.U. of the step is close to 100%.

Based on the results obtained graphically, the number of extraction steps required to separate the required amount of caprolactam from lactam oil in the first stage and from the benzene lactam liquid in the second stage was determined. You will also need a 6-step extractor at the benzene extraction stage (caprolactam concentration in the aqueous residue is 1% and 17% in

the benzene lactam) and a 5-step extractor at the aqueous extraction stage (caprolactam concentration in water is 28%).
The results of experiments conducted on a 6-digit experimental production device are given in Table 5.

Table 5. Experiment - the results of test experiments performed on an industrial device.

S/N	consumption, l/hour			Caprolactam concentration, weight %			
				Access to the column		Exit the column	
	Lactam oil	Benzene	Nitrogen	Lactam oil	Benzene	Aqueous residue	Benzene lactam
1	250	680	650	71	≤0,1	1,6	24
2	250	750	650	71	≤0,1	1,5	23
3	250	875	680	71	≤0,1	2,1	22,7
4	250	770	800	71	≤0,1	2,0	23
5	200	700	680	69	≤0,1	1,7	20
6	200	800	680	69	≤0,1	1,66	19
7	150	680	680	69	≤0,1	0,93	16,2
8	150	500	450	69	≤0,1	0,90	16,8
9	292	1260	1150	69,8	<0,1	1,7	19
10	292	1300	910	69,8	<0,1	1,2	17,3
11	292	1200	520	69,8	<0,1	1,1	18,4
12	200	680	680	69,8	<0,1	0,43	19,2

As can be seen from Table 5, relatively low specific nitrogen is used to carry out the extraction process. (0.4-0.6 m3 for 1 m3 of liquid at a pressure of 0.25-0.3 atm). The total specific flow rate for the liquid is 40-45 m3 / m2. hours.
The optical density of benzene lactam and the amount of benzene in the aqueous residue were recorded within the technological regulation. The removal of lactam oil by benzene lactam was 2 times less than that of the benzene extraction industrial column. [8].
In the future, the use of an 8-step extractor in the industry may be recommended to further reduce the amount of caprolactam in the aqueous residue during benzene extraction. It is recommended to use a 6-step bubble extractor during the aqueous extraction phase of caprolactam.

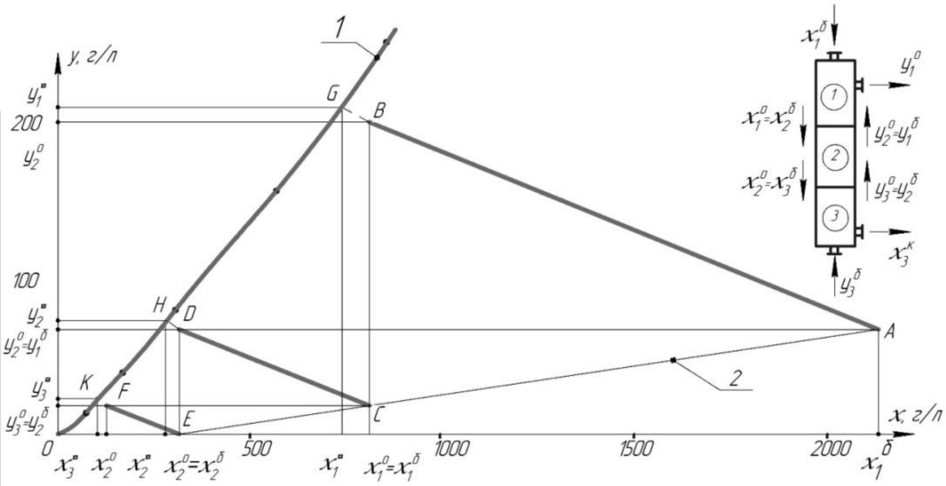

Figure 2. Caprolactal extraction by three-stage benzene extraction in a bubble extractor
1 - Stable equilibrium curve, AV is the working line of the first step, SD is the working line of the second step, EF is the working line of the third step, EA is the working line of the process, x *, u * is the concentration of caprolactam corresponding to one theoretical step.

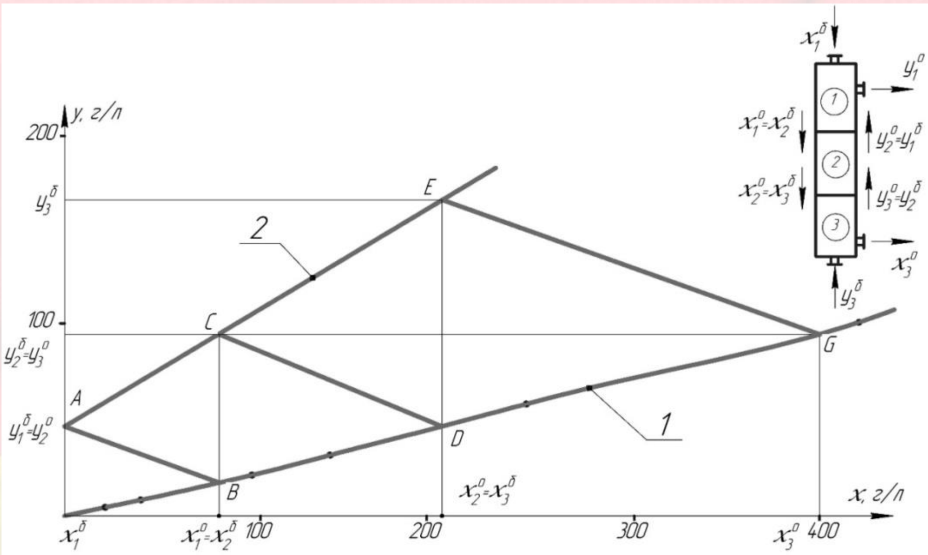

Figure 3. . Caprolactal extraction by three-stage aqueous extraction in a bubble extractor.

1 - Stable equilibrium curve, 2 - working line of the process, AV - working line of the first step, SD - working line of the second step, EG - working line of the third step, x - concentration of caprolactam in aqueous solution, o - concentration of caprolactam in benzene solution

In conclusion, the presence of a "nitrogen cushion" (R = 0.05 atm.). The liquid storage tanks of the extraction device also solve the problem of inert gas-nitrogen supply to the columns. In this case, the following cyclic scheme of gas supply to the column can be proposed: gas cushion - gas hood - receiver - column - gas cushion. A standard compressor can be used as a gas burner.

Symbols:

Ve - light phase consumption, l / h;

V0- heavy phase consumption, l / h;

Va- nitrogen consumption, l / h;

W - total specific fluid consumption, m3 / m2. Hour;

X - is the concentration of caprolactam in lactam oil solution, g / l

Y - concentration of caprolactam in benzene solution, g / l.

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