A Technology for Reduction of Phenol Containing Waste Water and Power Consumption in Phenol and Acetone Plants

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Abstract

The aim of this paper is to present a new concept for separation of a reaction mixture (RM) that is result of degradation of cumenehydroperoxide in industrial plants. Except the main components - phenol and acetone, this mixture contains water and a great number of different compounds, which form a lot of azeotropic mixtures. The separation of RM is difficult, power consuming and is impossible to occur without chemisorption.

The proposed method has been implemented in the Phenol plant - "LUKoil Neftochim" in Bourgas, Bulgaria. The difference between the new and the existing technologies is that the main quantity of phenol, presenting in so-called acetone stream, is removed from the bottom of the first column for acetone purification and then it goes back for dilution of the reaction mixture.

The application of this method reduces at least by half the quantity of phenol containing waste water and the expenses for the water regeneration: NaOH, H_2SO_4 , steam, cooling water, electric power, etc. It lowers the consumption of steam and cooling water as a result of usage of partial condensers at first two columns. The quality of the finished acetone is improved too.

The most popular method for producing of phenol and acetone is the oxygenation of cumene to make the intermediate cumene hydroperoxide (CHP). With the aim of sulphuric acid CHP is cleaved into phenol and acetone. Except this main component, the reaction mixture (RM) contains water and great number of different compounds, which form a lot of azeotropic mixtures: cumene, alpha-methylstyrene (AMS), acetophenone, mesityl oxide, diacetone alcohol etc. In Table 1 are given some of known azeotropes [1,2].

The separation of such mixture is difficult, power consuming and is impossible to occur without chemisorption. At the beginning of the process two streams are separated from a splitter column. The first one (acetone stream) includes all admixtures of light hydrocarbons, the major amount of acetone, water, AMS, cumene and some phenol. The second one (phenol stream) includes all heavy hydrocarbons, the major amount of phenol and admixtures such as water, acetone, AMS and cumene. Figure1 illustrates the most used technological scheme for acetone purification [3].

The stream for the top of the first column, in liquid

state, enters two consecutively connected columns in order to be separated. The admixtures of the light hydrocarbons are separated from the top of the second column, and the product from the bottom of this column enters the next column, where 5 - 10% caustic is added to neutralize the acid admixtures and phenol. The finished acetone is separated from the top of the third column. Two liquid phases are separated from the bottom of this column. The first one is an organic layer of AMS and cumene. The second phase is an aqueous layer made up of dissolved sodium phenate, sodium acetate and admixtures. The aqueous layer from the bottom of this column is processed with diluted sulphuric acid is fed to convert the sodium phenate into phenol and in the second stage the phenol is separated from the waste waters with the help of ionexchange resins or extraction and rectification of the phenol from the extracting agent. This technological scheme provides high quality of the finished acetone, but it is accompanied by irreversible loss of caustic and sulphuric acid as well as accumulation of large amount of waste waters.

The existence of azeotropes between water-cumene and water-AMS with boiling points lower than the

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Systems	% 1 st Component	Pressure, MPa	Boiling point °C	References
water-phenol	98 mol	0.1013	99.6	1
water-cumene	83.7 mol	0.1013	95	1
water-styrene	40.9 mass	0.1013	93.9	2
water-mesityl oxide	29 vol.	0.1013	91.3	2
water-diacetone alcohol	84.3 mass	0.1013	95.5	2
water-acetophenone	97.7 mol	0.0155	54.2	1
cumene- phenol	92.5 mol	0.1013	150.8	1
AMS-phenol	93 mass	0.1013	162	2
mesityl oxide-phenol	96.1 mol	0.0155	104	1
phenol-acetophenone	7.8 mass	0.1013	202	2
water-cumene-phenol	79.5/20.33/0.17 mol	0.1013	94	1

Table 1 Azeotropes



Fig. 1. Classical scheme for acetone purification.

boiling point of the azeotrope in the system waterphenol permits the separation of the phenol containing in the acetone stream, to make by means of azetropic rectification instead of chemisorption. In Figure 2 is presented the technological scheme for azeotropic purification of the acetone stream from the phenol, containing in it [4].

The proposed method has been implemented in the Phenol plant - "Neftochim" in Bourgas, Bulgaria. The difference between the new and the existing technologies is that the phenol, presenting in so-called acetone stream, is removed from the bottom of the first column for acetone purification and then it goes back for dilution of the reaction mixture (Fig. 2). The fraction, which is separated from the bottom of the last column, forms two layers. The first one is an aqueous solution of sodium salts of the admixtures. The second layer consists of cumene and AMS with phenol less than 0.01% mass.

The application of this method reduces at least by half the quantity of phenol containing waste water and the expenses for the water regeneration: NaOH, H_2SO_4 , steam, cooling water, electric power, etc. It additionally lowers the consumption of steam and cooling water as a result of usage of partial condensers at first two columns. For the term of the above

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Fig. 2. Technological scheme for azeotropic purification of the acetone stream.

mentioned plant the economy only at the steam is about 0.8 t steam for ton acetone compared to the previous scheme for acetone purification (Fig. 1). The quality

of the finished acetone is improved too (Table 2). The new concept allows production of acetone with purirty corresponding to "pure for analysis".

Property		Value	Test Method	
Purity, % mass		not less than 99.5		
Water, % mass		not more than 0.1		
Acidity as CH ₃ COOH		0.002		
Alcohols (CH ₃ OH)		0.02	GC	
Aldehydes (H ₂ CO)		0.001		
Alkalinity		Alkalinity absence		
Non-volatile residue		0.0005		
Metals	Al, Cd, Ca	5.10-5 (for everyone singly)		
	Ba, Fe, Pb, Mg, Sn, Zn	1.10-5	AAC	
	B, Cr, Co, Cu, Mn, Ni 2.10-6			
Permanganate time, h		not less than 24	_	

Table 2Acetone quality

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