



159159

Extraction of phenol-containing effluent streams

The present invention provides a process for extracting phenol-containing effluent streams.

5

In industrial processes such as, for example, the production of phenol by the cumene process or the production of bisphenol A by the condensation of phenol and acetone, phenol-containing aqueous solutions are produced
10 as effluent streams.

The separation of phenol from such effluent streams is not possible using a simple distillation process due to the solubility of phenol in water (the two substances are fully
15 miscible with each other at temperatures above 65.3°C, and almost 10 wt.% of phenol still dissolves in water at room temperature) and the formation of azeotropic mixtures during distillation of such solutions (J.M. Sørensen, W. Arlt, Liquid-Liquid Equilibrium Data Collection, Chemistry
20 Data Series V/1, p. 356 - 361, Dechema, Frankfurt 1979; Ullmann's Encyclopedia of Industrial Chemistry, vol. A 19, p. 299 - 312, 5th edition, VCH Weinheim 1991).

When preparing bisphenol A with the aid of acid ion
25 exchangers, the water formed in the reaction has to be removed because otherwise the activity of the catalyst is reduced. This is achieved in the first instance by isolating the water of reaction from the bisphenol A, its isomers and the phenol used in excess as solvent in the
30 bisphenol A process by distillation. The water obtained by this type of distillation contains a lot of phenol (1-15 wt.% phenol) and optionally also contains acetone (0-5 wt.%). In addition to these two main contaminants, other organic components such as methanol, acetone self-
35 condensation products, such as for example mesityl oxide, the sulfur-containing cocatalyst (a thiol) used in the



G00002

bisphenol A process as well as degradation and secondary products of these compounds may also be present in the separated water. However, other aqueous streams which are produced in such a process may also contain phenol, as well
5 as other organic components (e.g. water for floating ring seals or vacuum equipment).

Due to the toxicity of phenol and also for economic reasons (recovery of phenol), the phenol-containing effluent is
10 purified in an extraction process using an organic solvent. Benzene, cumene, diisopropyl ether or methyl isobutyl ketone (MIBK), for example, are used for this purpose. The extraction may be performed using any technique known to a person skilled in the art (tank extractors, mixer-settler
15 systems, counterstream extraction, etc.). In a subsequent step, the effluent treated in this way is generally processed on a steam stripping column in order to lower the residual concentration of extraction agent. This is important because residual concentrations of extraction
20 agents lead to heavy organic pollution of the effluent. By reducing the concentration of extraction agent in the effluent, the use of steam, and thus energy costs, can be saved. The effluent treated in this way is then subjected to biological effluent treatment in an effluent treatment
25 plant.

It has now been found that, when extracting phenol-containing aqueous solutions with MIBK as the extraction agent, the residual concentration of MIBK after extraction
30 can be reduced when the MIBK used for extraction also contains some anisole and optionally mesitylene.

The invention provides a process for extracting phenol-containing aqueous solutions in which a mixture containing
35 60 to 99 wt.% of methyl isobutyl ketone, 1 to 40 wt.% of

anisole and 0 to 20 wt.% of mesitylene, with respect to the mixture, is used as extraction agent.

A process for extracting phenol-containing aqueous solutions in which a mixture containing 75 to 95 wt.% of methyl isobutyl ketone, 5 to 25 wt.% of anisole and 0 to 10 wt.% of mesitylene, with respect to the mixture, is used as extraction agent, is preferred.

10 By extracting a phenol- and acetone-containing effluent with a mixture of MIBK and anisole instead of pure MIBK, the residual concentration of MIBK in the effluent is greatly reduced, given the same conditions of extraction. The degree of extraction for phenol and acetone remains
15 virtually the same.

Although, when using a mixture of MIBK and anisole, small amounts of anisole are found in the effluent, the decrease in MIBK concentration is much greater than the increase in
20 concentration of anisole so that overall an effluent with a lower concentration of organic constituents is obtained. By adding mesitylene to the extraction mixture, the concentration of anisole in the water after extraction can be further reduced while retaining the same extraction
25 effect and residual concentration of MIBK.

The phenol concentration in the aqueous solution (the effluent) is preferably 1 to 15 wt.%, with respect to the aqueous solution.

30

In addition, up to 5 wt.% of acetone, with respect to the aqueous solution, may also be present.

To improve the extraction process, the aqueous solution may
35 also contain sodium sulfate and/or the aqueous solution may

be acidified to a pH <7, e.g. using sulfuric acid or other acids.

The extraction may be performed using any technique known
5 to a person skilled in the art (multi-stage extraction, tank extractors, mixer-settler systems, counterstream extraction, etc.).

After extraction, residues of the extraction agent may also
10 be removed from the effluent by distillation in a column, optionally while supplying steam.

The aqueous phase obtained after distillation or extraction may also be taken for treatment in a biological effluent
15 treatment plant.

The phenol-containing aqueous solution may also be subjected to a distillation process before extraction, during which for example, any ketones or other organic
20 components which are present are entirely or partly removed.

The phenol and optionally other extracted organic components (in particular optionally extracted acetone)
25 extracted from the phenol-containing aqueous solution may be separated from the extraction agent by distillation and thus recovered. The extraction agent which is purified in this way may be used again in the extraction process.

30 The aqueous phase obtained after extraction or after distillation may also be purified with activated carbon prior to treatment in a biological effluent treatment plant.

The process according to the invention is preferably used for the purification of effluents from the production of bisphenol A. In the process for the production of bisphenol A, for example, phenol and acetone are reacted on sulfonic acid cation exchangers as catalyst, optionally in the presence of a thiol as cocatalyst, the bisphenol A obtained is entirely or partly crystallised out in the form of bisphenol A-phenol adduct crystals and filtered off and then water is removed from the filtrate, entirely or partly, by distillation. Likewise it may be used in a process for the production of bisphenol A in which separation of the water of reaction takes place, differently from the process described above, entirely or partly prior to crystallisation of the bisphenol A-phenol adduct crystals. In addition, it is suitable for the extraction of effluents from a process for the production of bisphenol A in which water (optionally mixed with phenol or with other organic solvents) is added during or before the crystallisation step.

Examples:Example 1 (comparison example)

5 170 ml of a phenol-containing aqueous solution (process
effluent from the production of bisphenol A) is shaken with
100 ml of MIBK for 15 seconds in a 500 ml separating
funnel. The separating funnel is then allowed to stand for
5 minutes, the aqueous phase can be drained off and this
10 phase is tested using gas chromatography. The results are
given in table 1.

Example 2 (according to the invention)

15 Example 2 is performed in the same way as example 1, with
the difference that a mixture of 80 ml of MIBK and 20 ml of
anisole is used instead of 100 ml of MIBK. The results are
given in table 1.

20 Example 3 (according to the invention)

Example 3 is performed in the same way as example 1, with
the difference that a mixture of 83 ml of MIBK, 12 ml of
anisole and 5 ml of mesitylene is used instead of 100 ml of
25 MIBK. The results are given in table 1.

Compound	Example 1	Example 2	Example 3
Phenol	0.21	0.23	0.25
MIBK	1.40	1.00	1.05
Acetone	0.017	0.015	0.017
Anisole	<0.005	0.044	0.025
Mesitylene	<0.005	<0.005	<0.005
Total	approx. 1.63	approx. 1.29	approx. 1.35

Table 1: Results of extraction trials (wt.% in the aqueous phase after extraction)

Claims

1. A process for extracting phenol-containing aqueous
5 solutions in which a mixture containing 60 to 99 wt.% of
methyl isobutyl ketone, 1 to 40 wt.% of anisole and 0 to
20 wt.% of mesitylene, with respect to the mixture, is
used as extraction agent.
- 10 2. A process according to Claim 1, in which the phenol
concentration in the phenol-containing aqueous solutions
to be extracted is 1-15 wt.%.
- 15 3. A process according to one of Claims 1 or 2, in which
the phenol-containing aqueous solutions arise entirely
or partly from the production of bisphenol A.
- 20 4. A process according to one of Claims 1 to 3, in which
the phenol-containing aqueous solutions contain sodium
sulfate.
- 25 5. A process according to one of Claims 1 to 4, in which in
addition up to 5 wt.% of acetone is also present in the
phenol-containing aqueous solutions.
- 30 6. A process according to Claim 5, in which the phenol-
containing aqueous solutions are subjected to a
distillation process prior to extraction, wherein at
least some of the acetone is separated.
- 35 7. A process according to one of Claims 1 to 6, in which
the concentration of methyl isobutyl ketone in the
aqueous solutions after extraction is reduced by
distillation.

8. A process according to one of Claims 1 to 7, in which phenol and optionally other organic components are recovered by distillation from the methyl isobutyl ketone-containing mixture obtained from the extraction
5 process.
9. A process according to one of Claims 1 to 8, in which the concentration of organic components in the aqueous solutions after extraction is also reduced by passage
10 over activated carbon.
10. A process according to one of Claims 1 to 9, in which the aqueous solutions are acidified to a pH <7 prior to extraction.