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APPLICATION OF THE ONE-STAGE DOUBLE EXTRACTION METHOD IN SAFE ELIMINATION OF POST-DISTILLATION RESIDUES FROM BENZYL CYANIDE PROCESS

Post-distillation residues make many both technical and environmental troubles in industrial practice. The purpose of this work was to elaborate an effective way of treatment of post-distillation residues from benzyl cyanide industrial process. Several extraction techniques were investigated. The one-stage double extraction method proved to be the most effective in this process. The one-stage double extraction treatment of post-distillation residues depends on a selective extraction and liquidization by means of both water, which dissolves hydrophylic compounds, and lyphophylic combustible solvent, which liquidizes mainly organic high boilers. Application of the one-stage double extraction method may increase the efficiency of the distillation process. This method also solves the difficulties connected with the evacuation of distillation residues from an industrial vessel and, in the case of benzyl cyanide process, due to this method no sewage is produced.

1. INTRODUCTION

Refining of organic chemicals by distillation is probably the most popular process in both laboratory and industrial scales. Every type of such a distillation produces more or less post-distillation residues (PDR). Generally, PDR contain organic high boilers and inorganic compounds. Organic high boilers consist mainly of reaction side products, products of thermal destruction, and polymers. Inorganic products (mainly salts) are present when such compounds are used in chemical process. As PDR are often viscous, easily solidifying and difficult in treatment products, their neutralization in industrial scale may cause many troubles. These problems may not be even seen in a small scale but they appear when the processes

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are applied in industrial scale in chemical plants. Most difficult problems, connected with PDR, are as follows:

clogging the bottom outlet of industrial distillation vessels by viscous, easily solidifying products,

necessity of leaving some amount of distilled product in distillation vessel in order to avoid evacuation difficulties,

clogging of canalization when PDR are directly poured out from vessel without any treatment,

carrying out and storaging in any place can produce serious environmental pollution.

Methods of elimination or utilization of PDR as direct combustion [1], [2], storaging [3], or pouring into canalization systems [3] are technically difficult or environmentally unacceptable. The presence of both organic and inorganic compounds make difficult the application of simple extraction methods.

The one-stage double extraction (OSDE) method [4] makes it possible to transform viscous, easily solidifying PDR into easy to handle liquids. The OSDE method depends on a two phase extraction of PDR by means of both water and combustible, lyphophylic solvent. Water dissolves hydrophylic compounds, whereas solvent liquidizes organic components of PDR. The obtained liquids can be easily evacuated from distillation vessels in room temperature. In this paper the possibilities of application of the OSDE and other extraction methods are described. The investigations concerned an original industrial process of benzyl cyanide production.

2. MATERIALS AND METHODS

The experiments had to solve problems concerned with PDR, appearing in benzyl cyanide industrial process. Synthesis of benzyl cyanide was based on a phase-transfer reaction:

$$PhCH_2Cl + NaCN \xrightarrow[N(C_2H_5)_3]{H_2O} PhCH_2CN + NaCl.$$

5% excess of NaCN in relation to benzyl chloride underwent the reaction. When the synthesis was finished, water phase containing sodium chloride, sodium cyanide, and sodium hydroxide was separated from the organic phase. Crude organic phase contained benzyl cyanide as main product (ca. 80%), benzyl chloride, and side products of the reaction. Organic phase contained also variable amounts of dispersed aqueous solution. Additional amounts of water, containing hydrophylic compounds, were present in organic layer due to emulsions, making precise the separation of the two phases, difficult in industrial practice. After separation, organic

phase was distilled under vacuum of ca. 10 Torr. When the industrial distillation process was finished (the residue was still liquid in high temperature but continuation of distillation could cause crystallization, solidifying, and clogging the bottom outlet of the vessel), the PDR were poured out to containers. Samples were taken in this moment and distillation processes were continued in a laboratory scale in order to obtain additional amounts of benzyl cyanide. Then the extraction–liquidization procedures were applied.

3. RESULTS AND DISCUSSION

The results of distillation–extraction/liquidization operations are presented in a table. Addition of lyphophylic solvent (experiment 1) resulted in dissolution of organic constituents of PDR. Addition of water with emulsifying agents (experiment 2) did not cause liquidization of PDR. Organic matter of high viscosity was still

Table

Application of the one-stage double extraction method in treatment of post-distillation residues from benzyl cyanide process

No. of experiment	PDR sample (g)	Toluene (cm^3)	Water (cm^3)	Other agent	Temperature (°C)	Results
1	35	35			70	Salts undissolved
2	45		100	5 g of sodium alkilosulphate (C_{10}–C_{14})	95	Organic compounds undissolved
3	100	50	300		70	Liquidization. One phase
4	100		300	50 cm^3 of ethyl acetate	70	As above
5	40		100	100 g of 40% NaOH 1 g of TWEEN 80	95	Organic compounds undissolved
6	80	40	200	30 g of 40% NaOH	70	Liquidization. One phase
7	50	40	10		70	As above
8	40	30	10	10 g of 20% NaOH	70	As above
9	100	50	10		70	As above

present in the bottom of the reactor. Application of a typical OSDE method (experiment 3) resulted in good liquidization of the PDR. As one phase homogeneous liquid was obtained, separation of organics from inorganics was impossible. This disadvantage was tried to be overcome in the next experiments. Replacing toluene by ethyl acetate in the OSDE procedure (experiment 4) resulted in obtaining a homogeneous liquid similar to that obtained in the previous experiment. Sodium hydroxide

solution (experiment 5) was added in order to hydrolize organic high boilers having cyanide groups. This operation was supported by the presence of non-ionic emulsifying agent. The results were similar to those of the experiment 2. The experiments 6–9 depended on the OSDE treatment. These experiments presented the final solution of the PDR problem. Liquidization of the PDR took place when the residues/toluene ratio exceeded 2:1, while water content could be less than 10% (6.4% in the experiment 9). The obtained, easy to handle, liquids did not solidify in the temperatures above 0°C. Evacuation of laboratory distillation vessel in room temperature did not cause any troubles. No solid, viscous residues have remained in the vessel.

The described operation of liquidization of PDR by means of water/solvent treatment, where very small amounts of water are used and no separation of phases takes place, constitutes a new alternative of the OSDE method. The whole liquid mass can be incinerated despite the presence of some amounts of inorganic compounds. This type of the OSDE treatment, leaving no sewages, increases the possibilities of effective treatment of difficult PDR in chemical processes.

4. CONCLUSIONS

PDR from benzyl cyanide industrial process can be treated by means of the OSDE method. Evacuation of the liquidized PDR from distillation vessel can be carried out in room temperature. No viscous, easily solidifying materials are obtained as a result of the OSDE application. Application of the OSDE procedure permits a full recovery of benzyl cyanide in distillation process. No valuable product remains in the PDR as a result of evacuation.

Contrary to the previous experiments with the OSDE application [4], the separation of organic phase from water was impossible in any of the experiments concerning PDR from benzyl cyanide process. As after the OSDE treatment the PDR can be directed to incineration in full, this type of the OSDE procedure produces no sewages.

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ZASTOSOWANIE METODY JEDNOSTOPNIOWEJ PODWÓJNEJ EKSTRAKCJI DO BEZPIECZNEGO USUWANIA ODPADÓW PODESTYLACYJNYCH POWSTAJĄCYCH PODCZAS WYTWARZANIA CYJANKU BENZYLU

Odpady podestylacyjne przysparzają wielu problemów w praktyce przemysłowej. Celem pracy było znalezienie skutecznej metody postępowania z odpadami podestylacyjnymi, powstającymi w procesie wytwarzania cyjanku benzylu. Metoda jednostopniowej podwójnej ekstrakcji, polegająca na jednoczesnym użyciu wody i czynnika lipofilowego okazała się skuteczna, powodując upłynnienie powstałych odpadów, umożliwiając opróżnienie aparatu destylacyjnego w temperaturze pokojowej oraz dalszą obróbkę niekrzepnących, płynnych materiałów.

ПРИМЕНЕНИЕ ОДНОСТУПЕНЧАТОГО МЕТОДА ДВОЙНОГО ЭКСТРАГИРОВАНИЯ ДЛЯ БЕЗОПАСНОГО УДАЛЕНИЯ ПОСЛЕДИСТИЛЛЯЦИОННЫХ ОТХОДОВ, ВОЗНИКАЮЩИХ ВО ВРЕМЯ ПРОИЗВОДСТВА ЦИАНИСТОГО БЕНЗОЛА

Последистилляционные отходы доставляют многие проблемы в промышленной практике. Целью настоящей работы было найти эффективный метод удаления последистилляционных отходов, возникающих в процессе производства цианистого бензола. Метод одноступенчатого двойного экстрагирования, состоящий в одновременном употреблении воды и липофильного реагента оказался эффективным, вызывая сжижение возникших отходов, способствуя опорожнению дистилляционного аппарата в комнатной температуре, а также дальнейшей переработке незатвердевающих жидких материалов.