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Process for purification of 3-hydroxy-2-naphthoic acid by selective extraction of 2-naphthol impurity with tributyl phosphate using supported liquid membrane

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ABSTRACT

3-Hydroxy-2-naphthoic acid (BON acid) is an important intermediate in the dyestuff and pigments industry. The BON acid produced in conventional way from the Kolbe–Schmitt reaction usually contains 2–5% 2-naphthol impurity. The objective of the work was to selectively remove 2-naphthol from BON acid using tributyl phosphate (TBP). The solubility of 2-naphthol and BON acid in water was modeled using the non random two liquid (NRTL) equation. The hollow fiber supported liquid membrane (HFSLM) was used for the purification. A mathematical model was developed to explain the results. The distribution coefficient of 2-naphthol was significantly higher than BON acid. The distribution coefficient decreased with increase in pH. In HFSLM experiments optimal separation was obtained in the pH range of 7–9. The stability of HFSLM module was investigated. During HFSLM runs separation factors in excess of 200 were obtained. HFSLM studies showed that it was possible to selectively remove 2-naphthol from the mixture, resulting in a significantly higher purity BON acid. Experimental data was adequately correlated using a developed mathematical model. Based on the experiments, a modified purification scheme is proposed.

1. Introduction

3-Hydroxy-2-naphthoic acid also known as BON acid or β-oxynaphthoic acid is an important intermediate in the dyestuff industry. BON acid is mainly used as a starting material in the synthesis of various dyes and pigments for preparing coupling components. Amides derived from BON acid are one of the most important coupling components for pigments. These coupling components are used in preparing benzimidazolone and disazocondensation pigments (Faulkner and Schwartz, 2009). BON acid and its derivatives are used as couplers for azoic dyes and metal lake pigments such as Pigment Red 57:1 (Hunger, 2003; Bekö et al., 2012).

The conventional process for producing BON acid is Kolbe–Schmitt reaction of 2-naphthol (Marković et al., 2015). The reaction scheme and a typical block flow diagram for the synthesis of BON acid is shown in Figs. 1 and 2 [3]. The moisture free sodium salt of 2-naphthol is subjected to step-wise carboxylation under high pressure and temperature. At the end of the reaction sodium naphthoxide is converted to disodium salt of BON acid and 2-naphthol. Thus according to the reaction scheme, at the end of the reaction half of the 2-naphthol charged initially is obtained along with the product. Majority of the 2-naphthol formed is distilled off during the course of the reaction. But this distillation does not make the final product completely free of 2-naphthol. The ratio of 2-naphthol to BON acid, after distilling 2-naphthol is around

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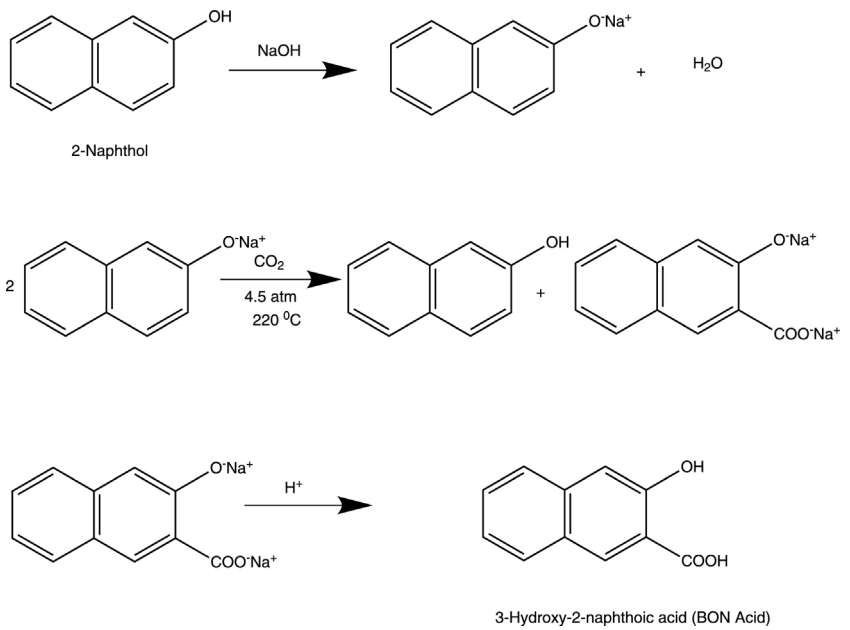

Fig. 1 – BON acid manufacturing reaction scheme.

20:80. 2-Naphthol is separated from the BON acid by adjusting pH of reaction mass to around 6–6.5. Precipitated 2-naphthol is then filtered off and the mother liquor is again acidified to pH 2–3, at which BON acid precipitates out. The precipitated BON acid in this manner usually contains 2–5% of 2-naphthol impurity. 2-Naphthol is also a coupling component; hence its impurity in the BON acid can cause the coupling to take place on 2-naphthol moiety as well. But dyes and pigments resulting from coupling on 2-naphthol have inferior fastness properties. Hence it is essential to have pure BON acid which is free of 2-naphthol which otherwise could result in poor tinctorial value of dyes and pigments.

Quadbeck-Seeger and Hoch (1977) have used different organic solvents to extract 2-naphthol. The organic solvents used were chlorobenzene, xylene, toluene, n-propylbenzene. In liquid–liquid extraction, 2-naphthol and alkali insoluble resins formed during the reaction were extracted in organic phase. BON acid in aqueous phase was acidified to give the pure product. Precipitated BON acid was reported to have around 0.4–0.5% of 2-naphthol as impurity.

Although conventional process utilizes 2-naphthol in molten form, carboxylation in water immiscible media such as kerosene has been reported (Phadtare and Doraiswamy, 1969). The solvent based processes help in easier dehydration of sodium salt of 2-naphthol. It can also be utilized to separate the impurities from the BON acid. Maegawa et al. (1981) have used di-isoproyltoluene to carry out carboxylation to obtain BON acid and the product was reported to have less than 0.1 wt% 2-naphthol. In one of the inventions Ueno et al. (2004) have extracted 2-naphthol from aqueous phase using xylene at 90 °C. But the plausible solvent losses due to extraction at higher temperature are not addressed. Sukhanov et al. (2004) have used TBP for extracting phenols and naphthols from aqueous phase. In their study, extraction of phenol by TBP adsorbed on polyurethane foam was investigated. The effect of pH and extractant loading was studied and it was observed that higher extraction was observed in acidic pH and on higher loading of TBP in the foam. Zidi et al. (2010) studied extraction of phenol by using TBP in various solvents using flat sheet membrane. Sodium hydroxide solution was used as a stripping reagent for feed phase concentration of phenol 200 mg L^{-1}. Effect of pH on phenol transport indicated that with increase in pH, amount of phenol transported in given time reduced. The distribution coefficient of phenol between aqueous and organic phase decreased with increase in pH values. Concentration of strip phase did not affect the phenol recovery significantly. Shen et al. (2009) have studied phenol recovery by using hollow fiber membrane contactor operated in non-dispersive manner by using TBP extractant. The effect of feed and strip size flow rates was investigated and a model was developed to study the phenol transfer across the membrane. Recently Shao et al. (2016) have demonstrated that TBP can give high values of distribution coefficients for the removal of 2-naphthol from aqueous phase.

Membrane based separations have been used in various fields such as pharmaceutical, hydro metallurgical, nuclear fuel processing (Sunsandee et al., 2012; Zhang et al., 2010; Vernekar et al., 2014). Supported liquid membranes (SLM) are being investigated as promising alternatives to conventional separation technologies. SLMs offer advantages such as high surface area to volume ratios, lower energy requirements, no need for phase disengagement, low solvent and extractant volumes, etc. But stability of the supported liquid membrane needs to be considered as well (Pabby and Sastre, 2013).

Established processes for BON acid manufacturing rely on separation of 2-naphthol from BON acid using precipitation technique. Often precipitation is carried out at temperature in the range of 50–70 °C followed by time required for cooling and decantation. This process of precipitation and filtration requires very large solution handling and likely to result in final product quality fluctuations depending on the variation in the precipitation conditions. Purification of BON acid from the mixture of 2-naphthol and BON acid by using TBP based solvent extraction process has not been explored in open literature to the best of our knowledge. Solvent extraction can overcome the shortcomings of conventional purification techniques and offer a novel approach towards BON acid purification by offering a possibility of eliminating intermediate precipitation and filtration. In this work we report the use of TBP extractant for selectively removing 2-naphthol using