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Comparative Simulation of the Purification of Wet Phosphoric Acid by Tbp, Mibk and a Mixture (MIBK+TBP)

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Abstract

Liquid-liquid solvent extraction technology is used in several industrial processes such as petrochemical processing, pharmaceutical production, food and hydrometallurgy. It is successfully applied for the purification of Wet Phosphoric Acid (WPA) with fairly good purification performances. The purification process is basically carried out in three steps: extraction, washing and recovery, each of these steps is a liquid-liquid extraction operation. The aim was to develop a computer code that helps predicting the overall performance of the purification process for a given set of operating conditions. The adopted approach is based on determining the necessary theoretical stages for each step in the purification process using a numerical technique. Some simulation results for the WPA purification process with Tri-Butyl Phosphate (TBP) at 45°C, Methyl Isobutyl Ketone (MIBK) at 25°C and a mixture (MIBK+TBP) at 30°C are presented.

Keywords: Wet process phosphoric acid; Purification; Solvent extraction; Simulation; NTS

Introduction

The most commonly used process for the production of phosphoric acid is: thermal and wet-process. The thermal process produces a pure acid with huge energy consumption. The wet-process involves reaction of phosphate rock with an acid (mainly sulfuric acid). This process is economic and practiced everywhere in the world [1,2]. Phosphoric acid produced by the latter process contains a variety of impurities, which vary according to the origin of the minerals. These impurities are removed from the acid by several techniques such as precipitation, adsorption, ion exchange and solvent extraction [1-12].

Liquid-liquid extraction is at the heart of many WPA purification processes. Several solvents can be used for the purification of WPA. The majority of solvents are able to extract selectively phosphoric acid (H_3PO_4), indeed alcohols, the etheroxydes, the ketones like Methyl Isobutyl Ketone (MIBK) and the phosphoric esters as Tributyle phosphates (TBP). The purification process is basically carried out in three steps: extraction, washing and recovery. All of these three steps are liquid-liquid extraction operations. Because of the complexity of such operations, it is very difficult to predict the overall performance of the purification process for a given set of operating conditions, making it very hard to find the optimal conditions for conducting the purification process [8-12].

On the basis of experiment undertaken to a scale laboratory, this study proposes to exploit the equilibriums isotherms to develop a computer code for the simulation of the operation of purification of WPA. The considered solvent is Methyl Isobutyl Ketone (MIBK).

The objective of this work is then to unfold a method for predicting the overall performance of the purification process by solvent extraction. This technique is founded on the simulation of the WPA purification process. The adopted approach is based on the determination of the necessary theoretical stages for each step in the purification process using a numerical technique. Some simulation results of a WPA purification process will be shown.

Modelling of the WPA purification

The modeling of the WPA purification was limited to three consecutive operations: extraction, washing and stripping, in order to simulate the purification of WPA by solvents with TPB, MIBK and mixtures MIBK+TBP. In this study, each step of the purification process is assumed to be conduct in a cascade of counter current theoretical stages. The phase diagram of the ternary system H_3PO_4 – water – considered solvents and the portion ratios for some trace impurities for the three systems have to be approximated. For the three solvents TBP, MIBK and mixtures MIBK+TBP, a representative H_3PO_4 distribution between the organic and the aqueous phases is shown respectively in Figure 1, 2 and 3. Also, the partition ratios for some trace impurities as function of the acid concentration in the organic phase are shown in Figure 4, 5 and 6.

For a given feed quality and an acid extraction rate in the extraction phase, the number of theoretical stages is determined as follows (Figure 7):

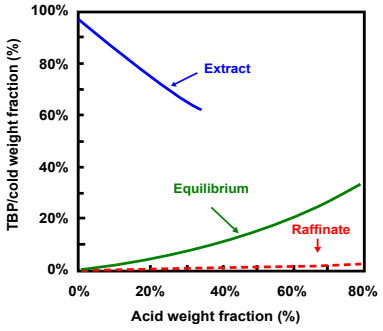

Figure 1: Equilibrium isotherms (H3PO4-H2O-TBP at 45°C).

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i. Assuming the raffinate quality and performing an overall balance yields the acid composition in the extract;

ii. The acid composition in the aqueous phase leaving the first stage is determined from the equilibrium curve;

iii. An overall balance between the second and the last stage yields the acid composition in the extra phase leaving the second stage and from the acid distribution isotherm the acid composition in the raffinate phase exiting the same stage;

iv. This calculation procedure is repeated until the last stage for which inlet extract phase and the outlet raffinate compositions should be consistent with the assumed extraction rate and the solvent composition. If these conditions are not satisfied, the solvent rate has to be modified and the calculation procedure has to be repeated until satisfaction of the convergence criteria.

This calculation procedure gives also the acid contents in all fluxes. A similar iterative numerical technique can be also applied to obtain impurities' contents in every flux. These iterative computation techniques have been implemented in a computer code that is capable of finding the number of theoretical stages as well as the composition of all fluxes in each step of the purification process.

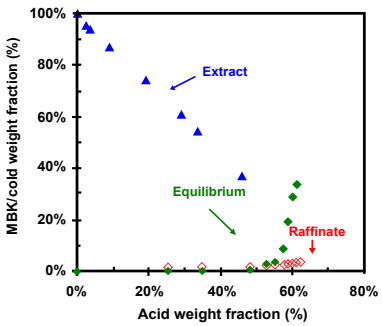

Figure 2: Equilibrium isotherms (H3PO4-H2O-MIBK at 25°C).

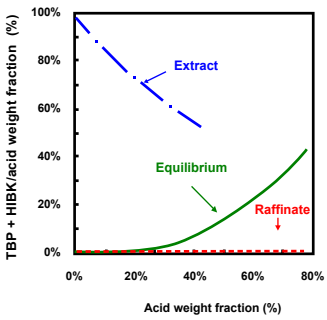

Figure 3: Equilibrium isotherms (H3PO4-H2O- TBP+MIBK at 30°C).

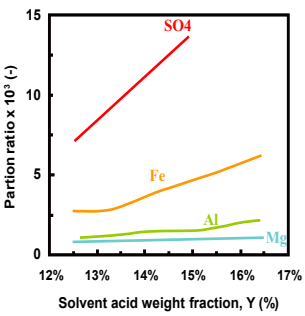

Figure 4: Some impurities' partition ratios (TBP at 45°C).

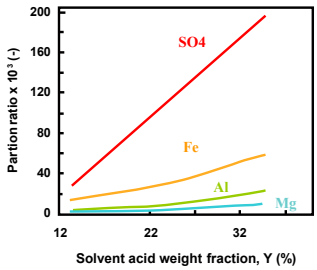

Figure 5: Some impurities' partition ratios (MIBK+TBP at 30°C).

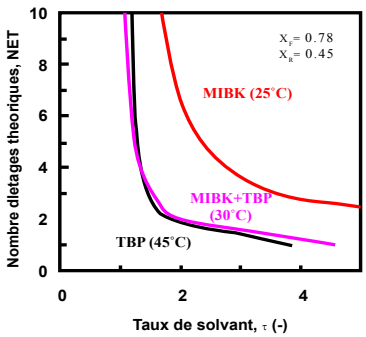

Figure 6: Number of theoretical stages for extracting H3PO4 from the WPA versus the solvent rate.

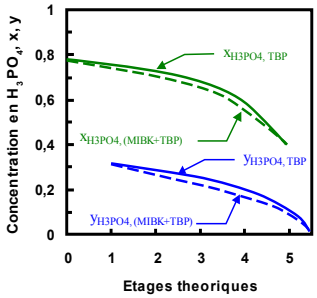

Figure 7: Raffinate and extract acid contents along the ideal extractors.

Results and Discussion

The Number of Theoretical Stages' (NTS) is an important parameter for the dimensioning of the equipments used in the purification process. A large NTS is equivalent of a difficult operation that needs huge equipments to insure adequate mass transfer between phases. For the three considered solvents, the NTS required for extracting a given amount of the acid in feed at 78% H_3PO_4 weight fraction for a residual content equal to 45% in H_3PO_4. For the same solvent rate, the operation of extraction with TPB at 45°C and mixtures MIBK+TBP require the same NTS, however MIBK the operation of extraction requires a NTS more important than foe the other solvent.

For a given amount of acid extracted by the solvent, the computer code gives the acid as well as impurities' weight fractions in all streams

flowing in out of all theoretical stages in the extraction operation. As an example, for an operation that requires five stages, Figures 8, 9 and 10 give details of the composition of the acid and impurities in both phases at each stage, for the three solvents, for a WPA at 78% H_3PO_4 with a fixed H_3PO_4 extraction rate at 82%. These details provide valuable information on the intensity of solute transfer taking place in each stage. It is clear from these figures that much of the transfer is happening in the stages close to the fresh solvent feed.

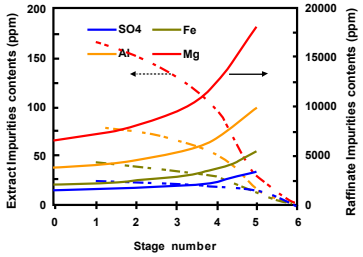

Figure 8: Raffinate and extract impurities' contents along the ideal extractors for TBP.

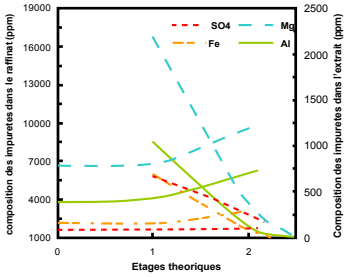

Figure 9: Raffinate and extract impurities' contents along the ideal extractors for MIBK.

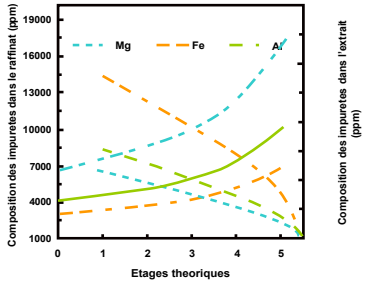

Figure 10: Raffinate and extract impurities' contents along the ideal extractors for MIBK+TBP.

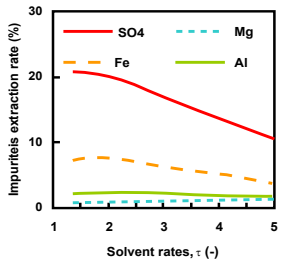

Figure 11: Purified acid impurities' contents versus acid content of the stripped solvent (TBP).

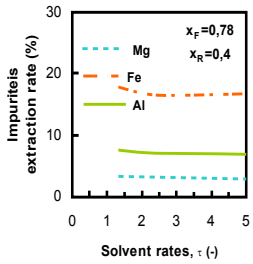

Figure 12: Purified acid impurities' contents versus acid content of the stripped solvent (MIBK+TBP).

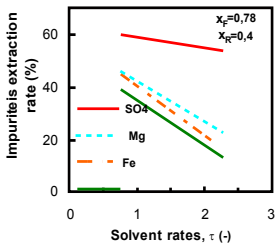

Figure 13: Purified acid impurities' contents versus acid content of the stripped solvent.

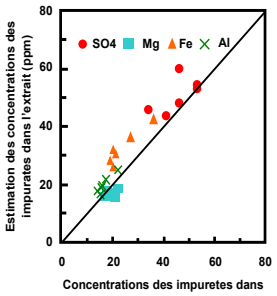

Figure 14: Comparison between the experimental results and theoretical results.

For the same operating conditions and for various solvent rates, the computed impurities' extraction rates for the TPB at 45°C, MIBK at 25°C and mixtures MIBK+TBP at 30°C are given respectively in Figure 11,12 and 13. As expected because of the increase of the required NTS, the extraction rate for the undesired matter intensifies when the solvent rate decreases. Comparison to other solvents, TBP at 45°C presents the best performances as for the reduction of the metallic impurities' content. The classification by descending order of the extraction rates of the impurities for the TBP is as follows: Mg, Al, Fe and SO_4.

The comparison between the experimental results obtained with various solvent rates for the operations of extraction with those provided by the developed code is necessary to valid our approach. Figure 14 and 15 give the extract impurities content obtained by the computer code, according to the experimental results of analysis of the impurities in the extract. It's noticed that the point are close to the first bisector, with those experimental with a light over-estimate for the TBP. For sulphates and iron, the error is higher because of the equilibrium isotherms Figure 16.

The extract exiting the first step in the H_3PO_4 purification process is then washed with pure water. This treatment is indented to eliminate the undesired impurities in the extract. The same numerical procedure as for the extraction, the result of the washing operation can also be

predicted. For an extract feeding the washing operation at 31% H_3PO_4 and a fixed acid weight fraction of the washed extract at 25%, Figure 17 gives the amount of the considered impurities discharged with the washing water and their concentration levels in the cleaned extract. As expected, the impurities extraction rate in decreasing order is for Mg, Al, Fe and SO_4. The sulphates removal rates for the washing operation is important because the extract in feed is rich in sulphates. By comparing the numerical result with those experimental in Figure 18 witches shows a good agreement.

The washed extract is then feeding the stripping operation in order to re-extract the acid to obtain the final purified phosphoric acid. The last one will undergo a post treatment process. A similar approach that applied for the extraction and washing can be used for the stripping operation. For a washed extract 25% H_3PO_4 supplying the stripping operation and for three acid concentration levels of the purified acid, the concentration of some trace impurities for the TPB at 45°C, MIBK at 25°C and mixtures MIBK+TBP at 30°C. For three acid concentrations, the ratio of the impurities' content in the purified acid, rapporting at 78% H_3PO_4, with the acid feeding the first step of extraction shows that for the TBP, as an example, Aluminum content in the purified acid are lower than 0.1% of those in the feeding acid. It is clear that one has to find the optimum stripping rate that leads to high acid concentration with minimum impurities contents in the purified acid.

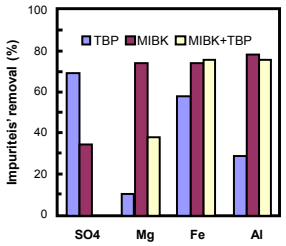

Figure 15: Impurities' removal rates for the washing operation.

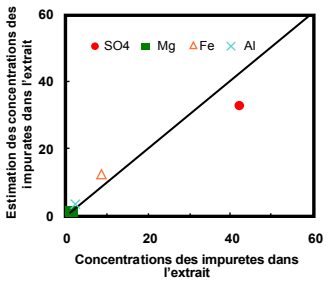

Figure 16: Comparison between the experimental results and theoretical results.

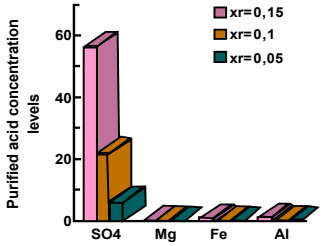

Figure 17: Purified acid impurities' contents versus acid content of the stripped solvent.

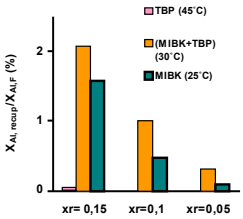

Figure 18: Ratio between Purified acid impurities' contents and feeding acid versus acid content of the stripped solvent.

Conclusion

In this study, a simulation of the purification of Wet Phosphoric Acid (WPA) was developed. The purification is based on solvent extraction. The computer code is applied for the extraction, washing and stripping the WPA. These operations are carried out in a cascade of counter current theoretical stages with three solvents witch knowing: Tri-Butyl Phosphate (TBP), Methyl Isobutyl Ketone (MIBK) and mixture (MIBK+TBP). The adopted approach is based on determining the necessary theoretical stages and the content of acid and impurities in the two phases for each step in the purification process. This approach could be used as a tool for predicting the WPA purification results in order to look for the optimal operating condition to conduct this operation.

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