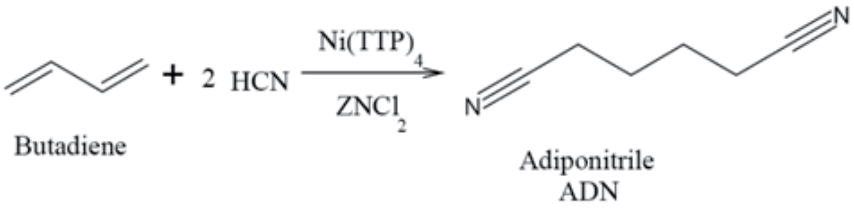
The effect of the increase in supplying flow on the separation in DSD

Adeline CAVALLI

Introduction

The adiponitrile (ADN) is an intermediate in the manufacturing of nylon. The synthesis of ADN consists in the addition of two molecules of hydrocyanic acid (HCN) on one molecule of butadiene. This reaction needs a catalyst (Ni(TTP)4), made up of nickel and phosphorus.



During this reaction, some intermediates are formed after the addition of the first molecule of HCN : pentenenitriles (PN). These react with the second molecule of HCN to form dinitriles (DN). The adiponitrile is the main dinitrile and the others are eliminated. In the second part of the synthesis, two settling tanks (DSD) separate the catalyst from the mixture pentenenitrilesdinitriles. The catalyst being sticky falls in the inferior phase and the mixture PN-DN composes the superior phase.
To develop the process, an increase of the supplying flow of the DSD is necessary. About DSD, increasing the flow will reduce the time of staying in the settling tank that could drag the catalyst into the superior phase.
Today the parameters for a good settling in DSD are fixed and the quantity of pentenenitriles, dinitriles and catalyst in each phases of DSD and in the supplying mixture are known.
Since the two DSD tanks operate similarly, only the parameters of one of both will be optimized.

Experimental conditions

An aliquot sample was removed in the superior and the inferior phases and also in the supplying mixture. By gas chromatography, PN and DN were separated using a 30 m x 0.32 mm SP 2380 column on an Agilent 6890 N-GC system in split mode equipped with a flame ionization detector (FID). Nickel and phosphorus were quantitated by X-ray fluorescence with a Panalitycal spectrometer in wavelength dispersive analysis. For phosphorus this quantification was operated at 2θ = 140.96° with a Ge 111-C crystal and a gas flow proportional counter ; for nickel, at 2θ = 48.65° with a LiF 200 crystal and a scintillation counter.
The relation between ligand (TTP) and phosphorus (P) is :
% TTP = (MTTP / MP) × % P

Results and discussion

The separation of PN and DN by gas chromatography was obtained in almost 60 minutes (Figure 1). PN were separated between 16 and 23 minutes although DN between 40 and 49 minutes.
Variations of PN and DN are controlled but the most significant parameter is the present of catalyst in the superior phase. The quantity of catalyst is calculated by the ratio TTP/Ni. If DSD do not bear the increase in supplying flow, the line graph which represents the ratio TTP/Ni as a function of the supplying flow should decline for the superior phase.

Figure 1 :

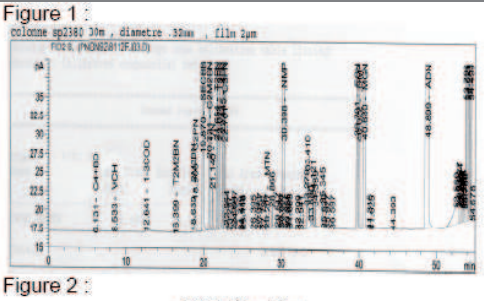


Figure 2 :

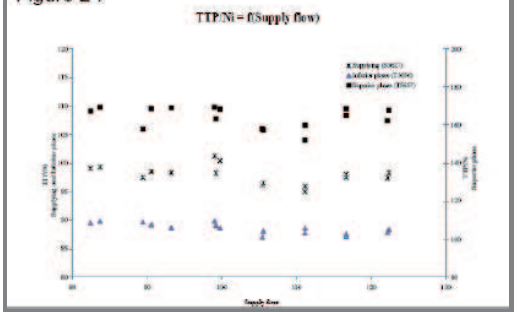


Figure 1 : GC analysis of the separation of PN and DN in superior phase. Gas vector : H2.
Figure 2 : Variation of TTP/Ni as a function of the flow.

Conclusion

Increasing the flow has no impact on good supplying in each samples, it offers the same quality in a reduced time. As a consequence, the project will be put in place without modifications pertain to DSD.

Rhodia Polyamide Intermediates
Butachimie
Usine de Chalampé
BP 267 – F 68055 MULHOUSE CEDEX