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Optimization of NMP extraction in 1, 3-butadiene production line
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acetate, n-Heptane, and petroleum ether used were from Merk, and the HPLC was from Shimadzo. A 25 cm. Shimadzo ODS column was used with the packing size of 5qm. Water was deionized by milli-pore milli-Q water purifier. The BD unit purpose is to separate 1, 3-Butadiene from the compounds with close boiling points with a 99% purity yields. This process is done with combination of distillation and extractions using NMP having 8.3% water content. Once the separation process is over, the un-evaporated residue from the solvent poses a great threat to the environment. Our first priority was to determine the amount of NMP in the residue then to devise a proper regeneration process and eventually a suitable discarding method.

RESULTS AND DISCUSSION

Extractions

The residue had some dissolution in various solvents such as water, Methanol, Benzene, Toluene, Chloroform, Carbon tetrachloride, Acetone, Xylene, Ethyl acetate, n-Heptane, and Petroleum ether which prompted further analyses of

each solvent. First, the residue was dissolved in water, then liquid- liquid extractions proceeded using non-polar solvents. Extractions with Xylene produced three phases, thus rendering an unsuccessful extraction. The same procedures were used with Toluene, Ethyl acetate Methanol, and chloroform which all proved to be unacceptable where in each extraction, the amount of NMP entering the organic phase was too little or in a trace amount thus unsuccessful. The extraction with n-heptane, however, produced better results. Though NMP is sparingly soluble in n-heptane, the yield was exceptionally pure, therefore, we decided to further improve the extraction using different temperatures. Three different temperatures were determined to be used, at room, near the boiling point, and post boiling point.

Extraction at room temperature

10.0 grams of residue was mixed with 20.0 grams of n- Heptane at 25°C. Only 1.05 grams residue was dissolved in n-Heptane. The dissolution formed two phases. The upper phase identified as E_{25°C} contained 23ml. (9.47gr.), and the lower phase S_{25°C} contained 3.5ml. (0.52gr.) n-heptane respectively.

Table1. N-heptane extraction of NMP at 25

Sample	Sample weight gr	%NMP weight in sample	% Impurities in sample	% n-Heptane in sample
S _{25°C}	0.5185	91.80	0.66	7.54
E _{25°C}	9.4713	6.01	0.04	93.95

shows the HPLC analyses of both phases, as it can be seen, the impurities present in both phases have been reduced in such a way that in the S_{25°C} phase, 0.66 and the E_{25°C} phase, 0.04% weight is present while the amount of NMP in S_{25°C} is 91.80 and in E_{25°C} is 6.01% weight. Therefore, n-heptane has selectively separated NMP from the impurities.

Extraction at near the boiling point

10.0 gram residue was refluxed in 20.0 gram n-heptane at 70°C for three hours. As expected, more residue material about 4.08 grams was dissolved in n-heptane at higher temperature. Again two different phases were identified, the rich organic phase E_{70°C} containing 19.32 gram n-

heptane and NMP rich S_{70°C} containing 1.11gr. The E_{70°C} phase was allowed to form two phases itself as the temperature lowered to ambient. The newly formed two phases of E'_{70°C} (18.26gr.) and S'_{70°C} (1.06gr.) were also analyzed using HPLC

Table 4. Summary of the NMP extraction in n-heptane in different temperatures.

Extraction temperature °C	NMP efficiency	Total efficiency	%sample extraction	%NMP by weight
25	5.53	10.49	0.5185	9.80
70	21.64	40.79	2.1723	85.68
100	53.26	64.23	5.0589	90.69

Shows the summary of the different temperatures used. As it can be seen with the increase in temperature more of the residue is dissolved in n-heptane also more NMP is extracted which results in the increase of the efficiency of the process.

Optimization of the extraction by increasing solvent residue ratio

Once the optimum temperature was determined, it was necessary to determine the effect of solvent residue ratio on the efficiency of the extraction. Four sets of weight percent ratios 1:1, 2:1, 4:1, 6:1 were prepared in such a way that 10 gr. residue and equal or increasing amount of n-heptane were mixed at 100°C. There was no separate phase formation as previous sections, however, as the mixture cooled phases began to form. The results are summarized in table 5.

Table 5. n-heptane extraction of NMP in different ratios.

Solvent residue ratio	NMP efficiency	Total efficiency	Weight of extracted sample	%NMP by weight	%Impurity by weight
2:1	53.26	64.28	5.0589	90.69	1.55
4:1	47.66	77.73	4.4521	92.23	0.59
6:1	30.43	83.38	3.2001	82.28	0.37
8:1	25.27	85.29	3.0101	81.15	0.31
9:1	20.18	86.97	2.9862	80.27	0.28

As it evident from the table, with the increase in solvent, total efficiency is also increased, however, because the NMP solubility in n-heptane is constant at set temperature, by the reduction in temperature, the NMP rich phase is reduced, therefore, the more solvent is used the more NMP remains in solvent thus reduces NMP weight percent which is our goal. However, at higher solvent-residue ratio, this process renders inefficient with an intangible reduction in total efficiency and NMP weight percent. Therefore, a 6:1 solvent ratio seems to be optimum, and since this ratio tends to hold the highest acceptable total efficiency, and since the separation of NMP and n-heptane both having two very different boiling points, this method could save large amount of very valuable solvent.