

# Research on purification of refined carbazole and anthracene by Solvent crystallization

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**ABSTRACT:** Solvent selection is the key of solvent crystallization method. In this paper, the optimal crystallization conditions for xylene and DMF extraction of refined carbazole is studied that the crude carbazole as a raw material, dissolved temperature is 85°C, crystal-keeping time is 90min, the solvent ratio is 1.8g/ml, the cooling crystallization time is 50min, the cooling crystallization temperature is 25°C. The results showed that the process has hardly environmental pollution and the lower cost, the solvent was circularly utilized, the Purity of refined anthracene and carbazole could reach to more than 96%, the total yield of refined anthracene could reach over 67%.

## Introduction

Anthracene and carbazole<sup>[1]</sup> is an important chemical resources in coal tar, more than 90% anthracene and 100% of carbazole of the world's is produced from coking byproduct crude anthracene. Anthracene and carbazole are widely used in dye, medicine, pesticide and guide optical electric specialty materials and other fields, with the rapid development of modern fine chemicals and organic synthesis technology, the demand of market of anthracene and carbazole is increase gradually, at the same time, many new applications are being developed. So it is great significance that to explore the technology refining anthracene and carbazole separation from crude anthracene.

The method that has been widely adopted refining anthracene and carbazole from crude anthracene include physical separation process, chemical separation process and composite separation process. The physical separation process is divided into solvent crystallization、distillation、zone melting method、emulsion membrane method、emulsion crystallization and zeolite adsorption separation method<sup>[4], [5]</sup>. The chemical separation process is mainly divided into sulfuric acid method and potassium fusion method<sup>[8]</sup>. it is mainly adopted the solvent - distillation coupled process to separate the crude anthracene at foreign and mostly already realize industrialized production, the technology is relatively mature, but higher construction investment and complicated process. Such as the solvent crystallization and distillation process that separate-refined the anthracene and carbazole from anthracene oil- I at France BEFS company, is simple and automated production, large capacity, but larger the equipment investment, the crystallization technical control complex. the reduced pressure distillation-acetophenone washing crystallization process for the company USES Germany<sup>[7]</sup>, can be obtained with purity of 95% refined anthracene, but has higher dosage of acetophenone as solvent, could be used in the moderate price situation. At present, The solvent method that is generally adopted to refine the crude anthracene at domestic<sup>[3]</sup>, is only suitable to small-scale production, the disadvantage has the longer process flow and consume lots of solvent, higher cost and large pollution. the key to reduce cost, improve product quality and production rate, increase benefits for domestic crude anthracene refined is that choose low toxicity, easy to operate, easy to recycle, good selective solvent<sup>[6]</sup>.

In this experiment, it is studied that DMF is based the main solvent in the dissolution and crystallization process that be used for the separation and purification of the crude anthracene.

## Experiment

### Reagent and Instrument

In this experiment , the crude anthracene (anthracene (23.18%), carbazole (23.18%) as the raw material is from the zhonglian chemical factory. the main solvent that has been selected ,such as ,DMF and dimethyl benzene, chlorobenzene and NMP, DMA etc, are analytical reagent.Main instruments are DF-101s collector heating and constant temperature magnetic agitator, Electrothermal constant temperature water-bath, Water-ring vacuum pump, HDM500tempering temperature electric heating jacket, WRD-1B digital instrument of melting point.

### Experimental

#### First solvent crystallization

First, the crude anthracene is joined in the three-necked flask(250ml). at the same time, the mixed solvent is joined in according to the solid-liquid ratio 1:1.8. Second, it is heated to 90°C in the thermostatic oil bath pot, stired fully it till be soluble and kepted warm 50min, and then slowly cooling groove to 25°C in the constant temperature water bath, crystal growing time 50min. Next step, filter cake by suction filtration is semi-refined anthracene that is dark yellow green and the filtrate of blackish green color. Next step, the solvent will be recycled by vacuum distillation under the temperature of 130°C to 140°C and the main components of the solid black residue is carbazole and phenanthrene, that is the raw materials for the extraction of carbazole.

#### Second solvent crystallization

First, the semi-refined anthracene Will be joined in the three-necked flask that was obtained at the first solvent crystallization, at the same time, the mixed solvent is joined in according to the certain solid-liquid ratio. Second, it is heated to 90°C in the thermostatic oil bath pot, stired fully it till be soluble and kepted warm 1hour, and then slowly cooling groove to 25°C in the constant temperature water bath, crystal growing time 50min. Next step, filter cake by suction filtration is refined anthracene that is yellowish-green and is dried in the drying oven. Next step, then the refined anthracene is determinated the purity and calculated the yield according to the YB/T5086-2005 standard method. The technological process is shown in figure 1.

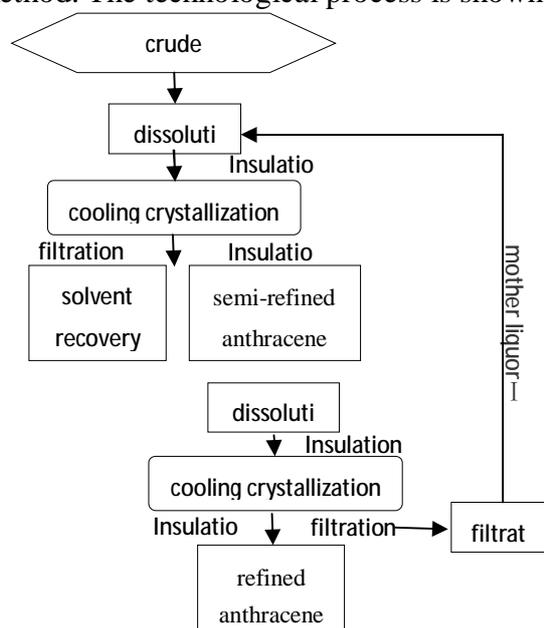


Figure1 Solvent crystallization process flow chart

## Results and discussion

### solvent selection

In this experiment, the DMF is the main solvent of the mixed solvent preparation to be refined anthracene in which suitable amount of NMP are added. It is studied to the influence of the mixed solvent on the yield and purity of refined anthracene at the dissolution 90°C and insulation 50min, and then slowly cooled to 25°C and crystal growing time 50min. By figure2 and figure3, the purity and yield of product changes are flat, there is no special values. when the amount of NMP joined in DMF is less than 10%, the purity of refined-anthracene is below 93%, the yield is 67% -69%; When the addition of NMP amount is more than 15%, the purity of refined-anthracene is more than 95%, the yield is 65%-67%; When the addition of NMP amount is more than 25%, the purity of refined-anthracene can reach more than 96%, the maximum of yield can reach 68.13% .Considering the purity of industrial refined anthracene with, the amount of NMP is 25%, namely the DMF and NMP compound ratio 3:1.

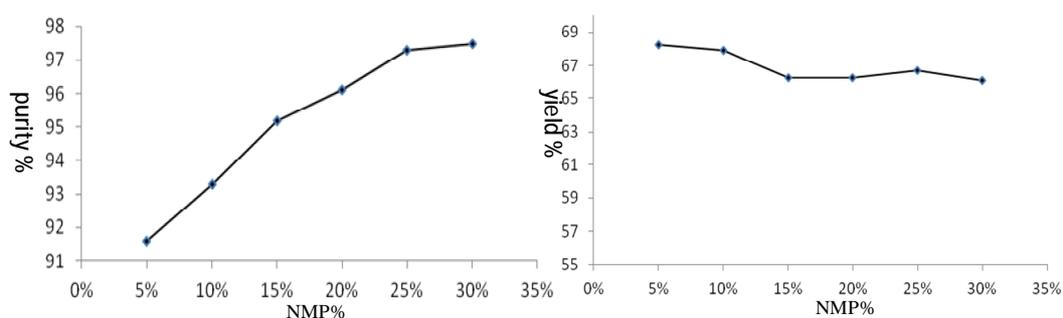


Figure2 the influence of the mixed solvent on the purity Figure3 the influence of the mixed solvent on the purity

### Solvent dosage

It can be concluded that when the liquid-solid ratio is less than 1.8 by figures 4 and 5, the purity of product increased gradually; when liquid-solid ratio is greater than 1.8, increasing the purity of the product is slightly and but the basic stable, its purity is above 95%; Product yield figure into a parabola, the highest yield when liquid-solid ratio is 1.8. when liquid-solid ratio is greater than 1.8, the yield of product drops rapidly, therefore, the mixed solvent of DMF and NMP the best liquid-solid ratio is 1.8.

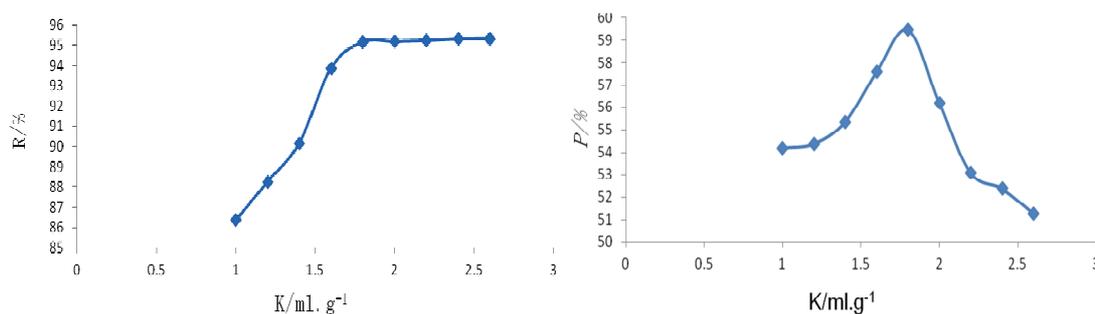


Figure4 the influence of solvent dosage on the purity Figure5 the influence of solvent dosage on the purity

### crystallizing point

With liquid-solid ratio of the mixed solvent is 1.8 (ml/g), crystal growing time is 50min, melting temperature is 90°C, it is studied the influence of different crystallization temperature to the purity of the product. with the increasing temperature, the purity of the anthracene product shows a trend of rise, and the purity of product anthracene with temperature increases with the increase of amplitude is more uniform, but the yield has a downward trend, at the same time ,the content of anthracene in the crude carbazole is larger, it is difficult to the processing of crude carbazole and the refined carbazole. Comprehensive, the cooling temperature is 25°C.

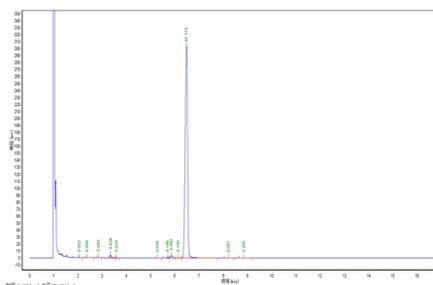


Figure6 GC of refining products by DMF

## Conclusion

The solvent crystallization process is used to be refined anthracene for the crude anthracene as raw materials and DMF as the mixed solvent. the best process conditions: with DMF as solvent, NMP as cooperate with solvent, best compound ratio 3:1, best liquid-solid ratio 1.8ml/g and the dissolution 90°C and holding time 50min, the cooling crystallization temperature 25°C and holding time 50 min. This method is simple in process and easy to recycle solvent, the low cost.the purity of refined anthracene was up to 96% and the yield is 67%.

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