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Discovery of a New Shade of Violet

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Anthracene and carbazole are two raw materials of organic chemistry and employed, among other things, for the manufacture of dyes. Together with Rütgers VFT AG (DE), Sulzer Chemtech has developed a process which enables these substances to be isolated from coal tar without the use of a solvent. Reduced environmental impact, energy savings and enhanced product quality are just a few advantages of the new process.

► The most important applications for carbazole are Violet 23 and Hydronblue R. The pigment Violet 23 is characterized by its high coloring strength and resistance to light degradation. As an original color shade, this pigment is found in the wrapper of a well-known chocolate bar (Fig. 1). Hydronblue R is an important, light-resistant dye and especially suitable for the dyeing of cotton fabrics, e.g. blue jeans.

Anthracene is the key chemical for the manufacture of anthraquinone and thus constitutes the starting product for anthraquinone dyes. Large quantities of anthraquinone are also required for the pulping of wood in the paper manufacture. The two raw materials are only found in a concentration of about 1% in coal tar and therefore have to be separated and highly purified. The feed materials, e.g. anthracene oil or raw anthracene, are

mixtures with more than 100 components. The knowledge of the separation behavior of these compounds is the key to a successful recovery of benzofurane-free, pure anthracene and colorless carbazole.

Material Separation Without Solvent

The traditional recovery process employs solvents. In view of the high production costs (especially for energy), the comparatively low yield and the environmental impact caused by the traditional process, industry has sought to replace this process for decades. However, due to the mixed crystal formation on the one hand, and the close boiling points of the substances accompanying the principal components on the other, isolation of the desired materials presents a great challenge for all thermal separation processes.

Using static, high-temperature melt crystallization (see box on page 6) together with a downstream distillation, Sulzer Chemtech and Rütgers VFT AG – the only company processing coal tar in Germany – have developed a new process which overcomes these difficulties. Both high-purity anthracene and carbazole are recovered from coal tar products without the use of solvents.

Patentable Process

In addition to sooty oil in a liquid state, the melt crystallization yields a high-quality fraction of anthracene, carbazole and phenanthrene in which the constituents form a solid solution of mixed crystals. Since these cannot be separated any further by means of crystallization, a high-tempera-

1 Thanks to the melt crystallization process from Sulzer Chemtech, the violet color of the chocolate bar “Lila Pause” from Milka can now be manufactured in an environment-friendly manner.



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ture, low-vacuum distillation column is provided downstream to segregate the light boiling anthracene from the higher boiling carbazole. The products anthracene and carbazole are discharged from the distillation column as side streams.

A claim has been made for the granting of a patent right for the new Anthracene-Carbazole Process. Innovative changes to the design and construction of the static crystallizer have made it possible to perform melt crystallization in the hitherto unattained temperature range near 300 °C.

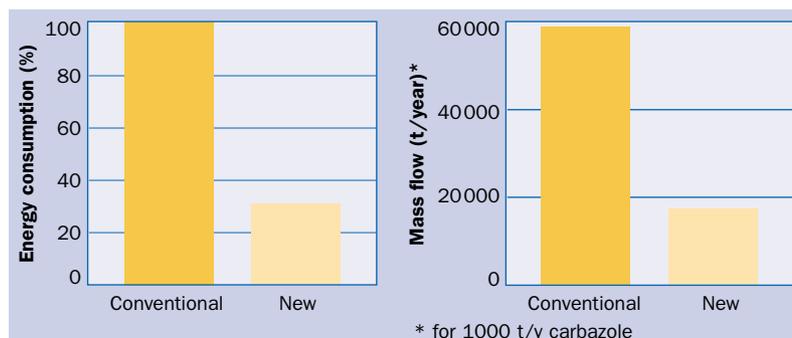
Energy Costs Reduced

As a result of the crystalline separation and subsequent distillation,

the specific costs for the recovery of anthracene and carbazole are reduced by 50%, and the yield of the complete process doubled (Fig. 2). Thanks to the improved removal of anthracene, the quality and value of other saleable products obtained from coal tar, e.g. technical oils or sooty oil, is increased. Furthermore, the new process has appreciable ecological advantages: The elimination of solvent results in a reduction of several thousand cubic meters of solvent-laden effluent a year.

During the development of the process, engineers from Sulzer Chemtech performed extensive tests in a high-temperature pilot plant built especially for this purpose (Fig. 3). The outstanding

2 The new process offers economical and ecological advantages. In comparison with the conventional process, the energy requirement is reduced appreciably. Thanks to the low mass flow, the size of the plant is smaller, but the yield remains the same.





3 The new process was tested exhaustively in this Sulzer Chemtech pilot plant. The research team of Rütgers VFT AG (one of the six divisions of the Rütgers Group) was awarded a gold medal for this process in the group-wide Innovation Competition 2001.

results of these tests have prompted Rütgers VFT AG to order the first plant using the new process.

Production Doubled

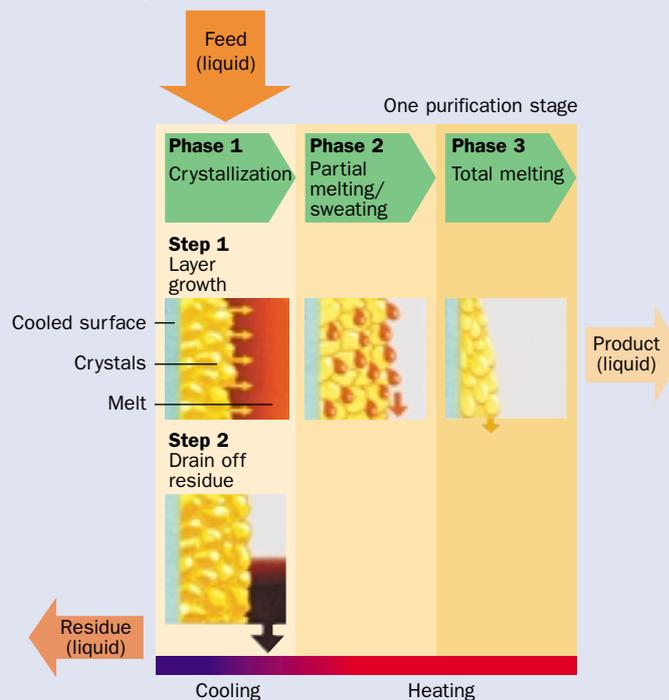
The new plant being built at the headquarters of Rütgers VFT AG in Castrop-Rauxel, north of Dortmund (DE), is to recover anthracene and carbazole using the new process. Thanks to the higher yield of this process, the total production capacity can be doubled compared with the previous production. For Rütgers VFT, the application of the new process signifies an important step towards the creation of new business structures: Once the new plant is put into operation, a number of old production facilities scattered around the Castrop-Rauxel works can be taken out of operation. ◀

Static Crystallization: Separation Without Solvent

The Sulzer Chemtech plant for static melt crystallization comprises vertical heat exchanger plates, which are immersed in the feed product to be crystallized. A heat transfer medium is circulated through the plates and ensures uniform conditions throughout the crystallizer during the crystallization process. During the first step, the plates are cooled to such an extent that crystals grow from the melt onto the plate surface. Following crystallization, the remaining liquid residue containing

impurities is drained off. The crystal layer is then heated slowly, allowing additional impurities to be exuded and further purifying the crystals. This partial melt fraction is also discharged. Finally, the purified crystall layer is melted by further heating, producing a liquid end product which is sent to the subsequent distillation. The previously discharged melt fractions are returned to the crystallizer in a later stage to increase the process yield.

Principle of the Static Crystallization



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