



Catalysts BY EVONIK

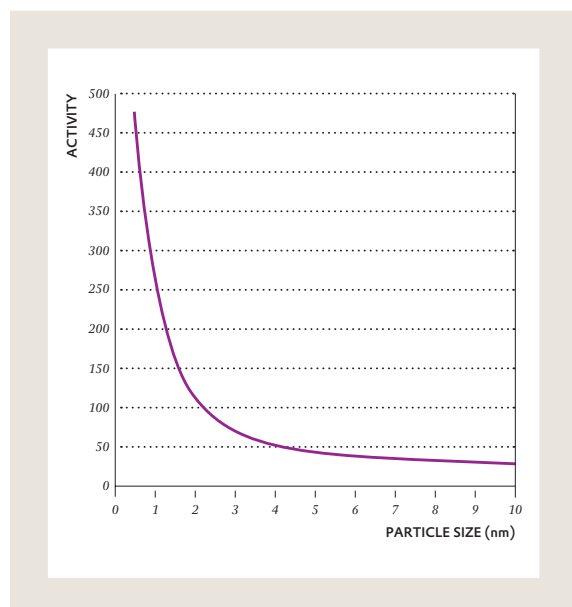
Handling Procedures for Activated Metal Catalysts (AMC)

Separation of AMC from Reaction Solutions

General Comments

After performing the desired reaction, it is necessary to separate the catalyst from the reaction solution to purify the product, recycle the catalyst for additional reactions, and/or to remove the deactivated catalyst. An ineffective separation method could result in lost time, lower product yields, less catalyst recovery, and -

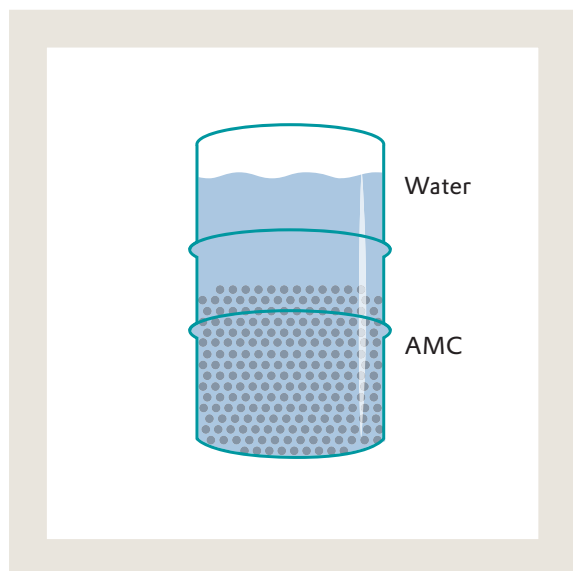
in the worst-case - process safety could be jeopardized. The choice of the proper catalyst for the desired reaction and the equipment used to produce the product can help to improve catalyst separation. Larger AMC particle sizes are easier to separate, but they are also less active. However, by adjusting the particle size and settling characteristics, an optimum catalyst can be found for each use. There are many commercial methods for catalyst separation and a few of these are listed in the sections below.



a) Sedimentation and centrifugation

Due to the settling behavior of AMC, most users separate them from their reaction solution by sedimentation. Once stirring in the reaction vessel stops, the particles of standard Ni AMC types agglomerate and rapidly sink to form a loosely packed catalyst layer with a bulk density lower than 1.5 kg/L. In the purest sense, sedimentation uses the gravitation pull exerted on the catalyst to separate it from the supernatant liquid. Hence, the rate of sedimentation can be improved by mechanically increasing the g-forces on the catalyst via centrifugation.

Typically, the more expensive centrifuge methods that increase the g-forces are also the most effective in collecting smaller catalyst particles. In the order of increasing efficiency and expense, hydroclones, decanter-type centrifuges, and nozzle centrifuges are commonly used for AMC separation.



A possible concern about the use of either sedimentation or centrifugation is that a significant amount of filtrate can still remain in the catalyst after separation. Techniques such as repeated solvent addition and removal could be used to improve product removal from the catalyst.

b) Magnetic separation

Augmenting *g*-forces with properly placed magnets can also enhance the sedimentation rates of AMC made out of magnetically susceptible metals (e.g. nickel and iron). The design of these filters can allow for either batch or continuous catalyst separations. Electromagnets are more flexible than permanent ones when it comes to catalyst re-slurrying and discharge.

c) Filtration through a medium

Filtration through a medium is also a common method for the separation of AMC. Many times filtration is used in coordination with other methods such as sedimentation. For example, a majority of the catalyst can be separated via sedimentation or centrifugation. This filtrate could be further filtered through a medium to remove the finer particles. As long as these finer particles have not been deactivated or diluted, they could also be recycled with the rest of the catalyst into the next reaction. Since smaller particles are more active than larger ones, the loss of the finer particles will have a detrimental effect on the recycled catalyst's activity. The selection of the medium itself plays an important role when considering filtration. A non-binding weave of wire cloth with openings of about 40 μm on a horizontal leaf filter can be used for this purpose. In this case, the reaction solution containing the catalyst is recycled through the filter to build up the filter cake. This recirculation continues until the filtrate runs clear. After that the clear filtrate is collected.

A simple wire cloth on either a plate and frame filter press, a vertical leaf filter, or a horizontal leaf filter may also be used, but in this case a precoat of filter aid (e.g. diatomaceous earth or cellulose) on the cloth is recommended.

The filter aid precoat is accomplished by recycling a filter aid suspension across the filter until the filtrate runs clear. After the precoat is established, the filter is kept under constant pressure and the reaction mixture is introduced to the coated filter. It is the filter aid itself that acts as the filter medium in this situation and its presence in the filtered catalyst makes it difficult, if not impossible, to recycle the catalyst back into the reaction. Moreover, spent catalysts diluted with filter aid are more difficult to refine. Filter mediums with openings smaller than the smallest catalyst particles may also be used. These mediums are usually made out of an expanded polymer or sintered metal powder and they are commonly in the form of cylindrical cartridges inside a candle filter system. After filtration, the catalyst can be easily discharged from these cartridges by reversing the pressure differential, which causes the filter medium to flex slightly.

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