### Webinar Q&A

### **SPONGE METAL catalyst uses in industry processes**

#### **Q 01.** What's the relationship between activity and particle size?

**A 01.** Activity is generally inversely related to the particle size of the catalyst. The smaller the particle size, the higher the apparent rate constant. There is often a trade-off between activity and settling characteristics. Smaller particle size gives higher apparent activity, but also gives a slower settling rate. Conversely, a larger particle size gives lower apparent activity but improved settling rate.

#### **Q 02.** What's the typical lifetime of fresh Sponge Catalyst?

**A 02.** Typical lifetime is 1-year from the manufacture date. Drums should be kept in a cool, dry place under reasonable conditions (not exposed to the elements of weather and extremes of temperatures – ideally kept between 5 - 30°C).

#### Q 03. What's the best event sequence for starting a batch reaction using sponge nickel catalyst?

**A 03.** The sequence of heat up, pressurization, feedstock and catalyst addition are critical to prolong catalyst life. Starting with a cold reactor at low pressure, add solvent & catalyst feedstock then purge and pressurize with hydrogen and heat up last. The presence of hydrogen during heat up replenishes the catalyst surface, preventing hydrogen depletion and subsequent oxidation of the catalyst surface.

#### Q 04. How many times can SPONGE METAL<sup>™</sup> catalyst be recycled?

A 04. The catalyst lifetime first depends on the reaction type, as well as, maintaining optimal reaction conditions is vital. Any interruptions that could lead to hydrogen starvation environment, should be avoided. Organics are attracted to active Ni surface and lead to deactivation. Since the catalyst has a gradual decline in activity after each batch, some small amount of fresh catalyst is usually added to every batch or every 2 – 3 batch recycles. The amount of such "make-up" catalyst is usually on the order of 0.5-1.5 percent of the original full charge amount.

#### **Q 05.** What can happen if catalyst loading is too high?

**A 05.** The first concern is full agitation mixing of the catalyst loading. High loading can lead to a hydrogen deficient environment, that can lead to deactivation.

# **Q 06.** After loading the catalyst to your medium, how long can you keep the catalyst "fresh" in nitrogen atmosphere?

**A 06.** The fresh catalyst will maintain it's activity if left within excess solvent, such as water or methanol, for a limited time. Organic feedstocks are attracted to the active Ni surface, and shouldn't be added to the catalyst/solvent mixture, if there's an extended delay.

# **Q 07.** I know that it's possible to improve sulfur resistance of Ni with additives Mo that selectively adsorb sulfur. Do you suggest this to prevent poisoning? And do you provide that kinds of elements?

**A 07.** We do offer Mo promoted catalyst, our A-7063 series, that are available for testing. Sulfur is particularly bad poison for any base metal catalyst and should be avoided or reduced. High hydrogen pressure may stabilize the catalyst during periods of sulfur contamination, preventing the adsorption of sulfur compound.



# **Q 08.** Can you characterize the typical porosity of the sponge metal catalyst? Is the catalytic process taking place mostly on the outer surface of the catalyst or does significant part of the reaction also happens in the pores?

**A 08.** Yes, the porosity (BET surface area) of Sponge metal catalyst can be determined by physisorption. The catalytic process occurs mostly in the catalyst pores, due to the high Ni BET surface area.

### **Q 09.** What do you suggest us to do with used catalyst? I mean, do you have a procedure for dead catalysts?

**A 09.** We have a handling guide that can be supplied to you on this. Please feel free to reach out to any of us and we can supply this to you.

# **Q 10.** Does the moly promotion, such as A-7063 series, still allow for diene hydrogenation (impurities in the feedstock)?

**A 10.** Our A-7063 series, Mo-promoted catalyst, is typical for carbonyl and some nitrile reductions. The first choice for a diene reaction would be a catalyst from our A-5000 series that are non-promoted. It's recommended to internally test the catalysts for the specific reaction.

# **Q 11.** Can the Ni-sponge catalyst be supplied in granular form (3-5 mm) instead of powder so it could be used in a fixed bed reactor?

**A 11.** We currently cannot supply large 3-5 mm granular form catalysts but we would be willing to look at your process to see what catalyst we can offer.

#### **Q 12.** Any safety issues with the formation of Ni Carbonyl?

**A 12.** The formation of Ni carbonyl, through carbon monixide poisoning is more of a catalyst deactivation issue, rather than a safety issue.

#### **Q 13.** Can you explain more about the desorption of CO? What helps drive the reversibility?

**A 13.** CO adsorption are more weakly bonded compared to the strongly chemisorbed nickel sulfied bonding. The desorption of CO from the catalyst surface is caused by soaking the catalyst in excess solvent.

#### **Q 14.** Can residual DMSO poison the catalyst?

**A 14.** Dimethyl Sulfoxide (DMSO), as well as other sulfur compounds, are irreversible poisons when they are absorbed onto Sponge Nickel catalysts. The DMSO concentration within the feedstock will dictate how much overall reaction activity is affected.

# **Q 15.** Does Sponge Nickel catalyst get deactivated in the presence of high concentration of K, Na, Zn ions?

**A 15.** High K and Na could act as an inhibitor slowing down the reaction, but not necessarily deactivating the catalyst. In some nitrile reactions, NaOH is used as an additive to prevent secondary amine formation.

# **Q 16.** Do you only produce Ni-based sponge catalysts, or can the Sevierville site also make Co-based sponge catalysts?



**A 16.** We can produce a Co catalyst and is part of our portfolio.

# **Q 17.** Can the catalyst activity be maintained by doing a hydrogen sparge to refresh the weakly held sites?

**A 17.** Adequate hydrogen pressure is needed to maintain the catalyst's activity and preventing a hydrogen deficient environment. High hydrogen pressure can help stabilize the adsorbed hydrogen at the catalyst surface. An alkaline/water rinse (or solvent) will aid removal of any organic material on the catalyst surface that can lead to deactivation via adsorption on active sites.

#### Q 18. Can you also comment on the metal leaching (Ni, Mo, Cr ...) in those liquid phase reactions?

**A 18.** Metal leaching occurs when the Sponge nickel catalyst is subjected to an acidic environment (typically below pH 5). The amount of metal leaching is determined by time and pH. Any promoters, including Mo, Cr, or Fe, would also be affected by the acidic environment and result in soluble metal ions.

#### **Q 19.** Is there a particular test that can be used to judge catalyst activity?

A 19. We have an activity instrument in the lab that measures activity of the catalyst.

#### Q 20. Is there a beach titration test due to bleach being used to deactivate the catalyst?

**A 20.** We have a procedure in our sponge handling brochure that shows how to deactivate the catalyst using various methods and can be supplied to you.



#### Q 21. What is the difference between A-5000 and A-4000 series?

**A 21.** A-5000 is a non-propmoted Ni-Al catalyst. A-4000 is a Fe/Cr promoted Ni-Al catalyst, typically used in dinitrile reactions.

### Q 22. Can the deactivation aspect be used to judge activity? My site uses an excess of bleach to deactivate.

**A 22.** We have a procedure in our sponge handling brochure that shows how to deactivate the catalyst using various methods and can be supplied to you.

# **Q 23.** Best practice(s) to avoid the catalyst from getting "sticky" from the leaching out of the aluminum?

**A 23.** Adequate agitation with excess water should break-up any loose agglomerates. Maintaining high alkaline environment will keep the Al within the supernatant.

#### **Q 24.** What is HMDA?

A 24. Hexamethylenediamine used for production of polymers and more specific nylons

**Q 25.** Are there any case to apply CSTR for sorbitol production?

**A 25.** There are a number of slurry-phase units operating worldwide on a continuous, rather than batch, basis. In this type of operation, catalyst, feedstock, and hydrogen are passed upwards co-currently through a reaction zone.

#### **Q 26.** What is the optimal temperature of the reaction regarding to recycling of the catalyst?

A 26. The optimal reaction temperature is typically determined through catalyst testing.

### We at JM would like to thank you for participating in this webinar