

JM ProcessWise Webinar

Edible Oils and Oleochemicals webinar questions

October 2020

Questions and Answers

Q1. Would you have guidance for best practice to ensure there is no carry over of the catalyst into the finished product?

A1. The catalyst can carry over into the finished product in two ways:

- a. Solids carry-over
- b. Dissolved nickel e.g. nickel salts / soaps

The carry-over of solids can be prevented by a rigorous filtration strategy. The filter type and specification is the first important choice, with candle or pressure-leaf filters being popular choices (although other types are available). You should work with your filter provider to ensure an appropriate mesh grade is used, and Johnson Matthey can provide information on catalyst particle size distribution to ensure that your filter is well-matched. You may also wish to consider the use of a filter aid to ensure finer filtration, or circulating your feed through the filter several times to allow a filter cake of catalyst to build up on your mesh.

Dissolved nickel is best avoided by process optimisation to minimise the dissolved nickel content in your product. Please reach out if you would like to discuss process optimisation options for a specific process. Furthermore a downstream cleaning step may be desirable, in which an acid e.g. citric or phosphoric acid is mixed with the finished product to chelate to dissolved nickel as part of a bleaching step.

Q2. Apart from Ni, what other promoters are vital for a high performing full hydrogenation catalyst?

A2. It is important to use a catalyst with an appropriate pore structure to ensure the best activity possible for a full hydrogenation.

Chemical promoters can also be used in catalysts to modify such parameters as nickel dispersion, pore size and pore structure. The exact nature of promoters used in the PRICAT range is proprietary. Please contact us directly if further information is required.

Q3. I assume full hydrogenation and non-selective hydrogenation is controlled by the activity and thus Ni content of the catalyst, how are catalysts tailored for selectivity or steep melting curve applications.

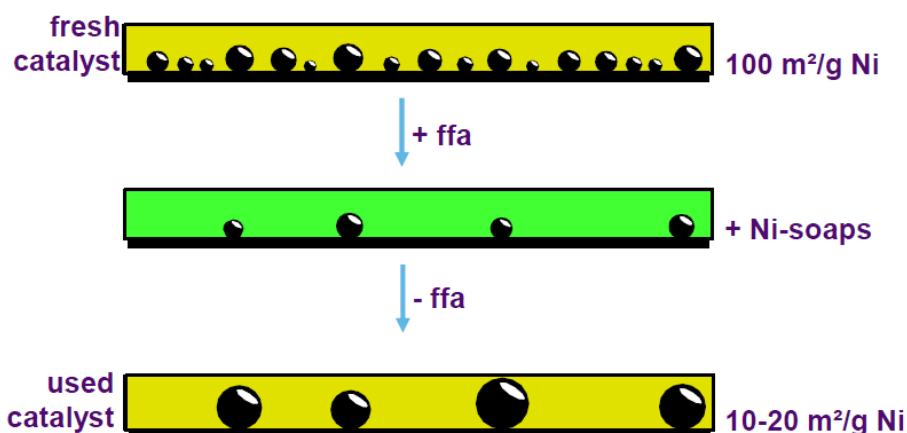
A3. The tailoring is different in each case depending on the application.

Full / non-selective catalysts are designed to have deep and easily accessible pore networks to promote maximum access to the active nickel surfaces and so maximum activity.

Selective catalysts have wide but shallow pores to promote single-hydrogenation interactions between oil and catalyst; in other words, the oil molecule can interact with the catalyst and then easily become dispersed in the bulk oil again without further interaction, in contrast to the deep pore networks of the full hydrogenation catalysts.

The steep melting curve catalysts a.k.a. trans-selective catalysts are sulphided, and this light dispersion of sulphur across a proportion of the available nickel surface area promotes trans-isomerisation. As explained in the session, it is the presence of these trans-fats that creates the steep melting curve characteristic.

- Q4. Sorry, I didn't properly understand why the catalyst cannot be reused for oleochemicals. Is it too poisoned? Please could you explain again.
- A4. Nickel is soluble in fatty acids feeds, and so when hydrogenating fatty acids for oleochemical applications, some of the nickel is inevitably dissolved in the feed. The amount of nickel dissolved in the feed changes over the course of the hydrogenation e.g. some nickel dissolves when catalyst is charged to the feed, and then re-deposits on the support when hydrogen pressure is applied. These dissolution-redeposition cycles cause a loss in nickel surface area:



Although the catalyst is capable of carrying out a single hydrogenation without any significant effect on performance, the loss of surface area means that performance in multiple hydrogenations is likely to degrade.

- Q5. What is the best way to review if we are using the right dosage of catalyst, especially if there are slight changes in the feed and conditions?
- A5. If changes in your feed characteristics and conditions cause a change in the process or the final product, catalyst dose is just one parameter that you may need to adjust. The change in process or final product composition will give you a clue as what you need to change e.g. if your final product melts too quickly, you may need increase the saturate content of the product by increasing the catalyst dose. The same effect could also be achieved by an increase in temperature, pressure or agitation.

It is also worth considering that hydrogenation parameters are interrelated, and a single isolated change is rarely possible. For example, if you increase your catalyst dose to increase saturate content of the final product, you may also find that trans-fat content becomes higher and reaction time becomes shorter. Also increasing catalyst dosage may not have the desired effect if there is insufficient hydrogen pressure to make use of the extra catalyst, and/or too little agitation to ensure contact between catalyst, oil and hydrogen.

Please reach out for a custom evaluation of your catalyst dosage.

- Q6. What level of poisons can the catalyst handle?
- A6. As a rule of thumb, you should not see appreciable detriment to catalyst performance if the maximum poison concentration is observed as below:

Sulphurs: ≤ 2 ppm
Phosphatides: ≤ 5 ppm

Successful hydrogenations are possible with poison concentrations greater than this, but adjustments in catalyst dosage and perhaps process conditions would need to be considered.

Q7. Do you also supply a Kosher certified catalysts?

A7. The entire PRICAT 99xx range is Kosher certified, and we can provide the appropriate certificates to demonstrate this.

Q8. We've had occasional issues with nickel soap even though we've tried a few different catalyst, is there anything new we should consider?

A8. In order to minimise dissolved nickel, it is advisable to:

- a. Minimise nickel dissolution by process optimisation
- b. Carry out downstream bleaching to remove dissolved nickel from the product.

The former is the best approach, because prevention is better than the cure!
Dissolved nickel is minimised by:

- Using maximum hydrogen pressure
- Reducing the free fatty acid content of streams (not possible for fatty acid feeds of course!)
- Preventing water ingress into the reactor, and/or pre-drying feed to minimise moisture
- Minimising oil-catalyst contact time pre- and post-hydrogenation e.g. prewarming oil before charging catalyst, filtering as soon as practicable following hydrogenation...
- Selecting an appropriate catalyst.

Altering these parameters may have other effects on the process and the final product characteristics, some of which may be undesirable, so a balance is required based on optimisation.

If these measures do not sufficiently reduce dissolved nickel content, downstream nickel removal is also possible, usually in the form of a bleaching step.

Q9. Can fatty acids molecules block active sites on the Ni pellets surface? In other words, is there an optimum with respect to catalyst loading?

A9. The deactivation mechanism of fatty acids is the dissolution of nickel. This differs from the poisoning mechanisms of sulphurs and phosphatides, which can block active metal surfaces or block pores respectively.

The optimum catalyst loading with respect to fatty acid deactivation varies depending on process conditions and desired process outcomes.

Q10. Are full and partial hydrogenation catalysts the same metal type?

A10. Yes, the entire PRICAT 99xx range is based on nickel. The variation between the different catalyst types comes not from the identity of the metal, but from the pore structure of the support and the use of modifiers.

Q11. How do we know that we have the right dose of catalyst?

A11. If your hydrogenation delivers the outcomes that you want, then you are probably using the right dose of catalyst! If the aim is to reduce the catalyst dose to be as economical as possible, it may still be possible to minimise the dose by altering other process parameters e.g. better agitation can enable a lower catalyst dose. Please reach out to discuss the options for your individual circumstances.

Q12. What is the best way to deal with nickel soap?

A12. See A8.

Q13. Do you have a catalyst type that is suitable for kosher and halal applications?

A13. Yes, the entire PRICAT 99xx range is certified for Halal and Kosher applications.

Q14. What is the main poison for these catalysts that we should worry about and what levels can the catalyst cope with?

A14. The most common poisons are sulphurs and phosphatides, although other catalyst poisons do exist which are uncommon in oil feeds e.g. citric acid, halogens, carbon monoxide. See A6 for tolerances.

Q15. What are pros and cons of hydrogenation on triglycerides compared to on fatty acids?

A15. The choice is most often a matter of practicality. For example, not every producer of hydrogenated fatty acid products has splitting capability on site, so they may have no choice but to source and directly hydrogenate fatty acids.

Where the choice does exist, process safety considerations favour the hydrogenation of triglycerides, because the required hydrogen pressure is generally lower for triglycerides compared to fatty acids, perhaps 10 bar vs. 20 bar respectively.

Q16. Please could you clarify what is the form and size of the catalyst?

A16. The catalyst arrives as a pellet, which consists of nickel powder catalyst encapsulated in solid fat. The encapsulation means that the nickel powder can arrive pre-reduced without the danger of exposure to air, which could cause heating due to oxidation. These pellets are amorphous but as a guide are approximately 5 mm in diameter.

The particle sizes of the nickel powder in the pellets varies slightly by product, but as a guide, the powder size generally falls in the range of 6 – 14 microns.

Q17. How to determine the right amount of spent catalyst to be reused back in the process?

A17. This will depend on how much activity remains in the spent catalyst after hydrogenation, which in turn varies depending on poison content, free fatty acid content, hydrogenation time and some process factors.

To give a simple example, if 1kg of fresh catalyst was charged, and that 1kg of spent catalyst was recovered, retaining 80% of the activity of fresh catalyst, you would need to charge 1.25kg of spent catalyst to observe the same performance. Alternatively, you could charge the 1kg of recovered spent catalyst and top up with 0.2kg fresh catalyst.

This is a simple example for demonstrative purposes, and factors other than activity need to be considered before reusing. Please reach out for a discussion on reuse tailored to your specific circumstances.

Q18. Not a question but more of a comment: I used to work for JM and had experience with this catalyst range. Clear presentation and I trust this product range is in great hands with Callum Campbell.

A18. Thank you for your valuable feedback.

Q19. In the oleochemicals production, is nickel dissolution the deactivation mechanism then?

A19. Nickel dissolution is the main deactivation mechanism of fatty acids, yes.

Q20. Does the process operate in batch or continuous mode?

A20. The PRICAT 99xx range are designed for slurry processes operating in batch mode.

Q21. How can make the melting curve steep?

A21. By using a trans-selective catalyst in a partial hydrogenation process, isomerisation of double bonds to their trans-form is promoted. The presence of these trans-fats gives the fat the steep melting curve property.

Q22. Is the support used on the nickel catalysts relevant?
A22. Yes. Different supports offer different pores structures, filterability and resistance to different feed types.

Q23. Can any metal other than nickel be used for the catalyst if we have a soap issue?
A23. Yes. Platinum and palladium metals are popular soap-free choices where the presence of dissolved nickel is unacceptable. The choice to use nickel is often an economical one, as nickel catalysts are usually approximately 10 - 100x cheaper, accounting for relative dose requirements and differing reuse capabilities.

Johnson Matthey offers a range of suitable precious metal catalysts, which we can discuss with you if nickel is not appropriate for your circumstances.

Q24. Does each catalyst type have different optimum operating conditions or is the optimum determined by the process?

A24. The catalysts have an operating temperature of approximately 120 – 250 °C under normal use.

Other than this temperature range, most of the choices made around operating conditions will be based on process requirements and desired product characteristics.

Q25. What is most beneficial cost wise. Hydro and split or split and hydro for final production of fatty acids?

A25. The process costs depend upon a wide variety of factors which could not be adequately summarised here. See A15 for a short summary which may help to inform cost calculations.

Q26. What physically is the difference between selective and non selective catalysts if they are they are both nickel based?

A26. See A3.

Q27. What are the issues of using spend catalyst?

A27. Spent catalyst will have lower relative activity than fresh catalyst. A higher charge of spent catalyst or a top-up with fresh catalyst is required to achieve the same results with each cycle.

If there are catalyst poisons present in the oil feed, there may also be additional consequences e.g. reusing a spent catalyst which is partially poisoned by sulphur will have the same effect as using a trans-selective catalyst, which will promote trans isomerisation in a partial hydrogenation. This would affect the melting characteristics of the final product.

Q28. Can spent catalyst be rejuvenated in some way?

A28. Options to rejuvenate nickel catalysts ultimately prove to be uneconomical in most cases. The most common way to reclaim value from spent catalysts is not to rejuvenate for further use, but rather to refine the waste to recover the nickel. The question refers to rejuvenating the waste for further use as a catalyst, and refining does not offer this, but it is a way to at least recover value from the waste.

Q29. Are the Ni-powder mechanically stable? Or can their be an effect on particle size depending on stirring conditions?

A29. The catalysts are designed with low attrition in mind. The typical particle size is 6 – 14 microns, and at this particle size the mechanical stability is good. In addition, particles enjoy some protection from attrition because of the viscous oils and fatty acids in which they are used.

Under normal use, the particle size will be roughly the same at the start and at the end of the process, but to minimise the risk of attrition, it is recommended to avoid especially strong stirring (normal agitation is fine) or sharp edges etc.

- Q30. For the hydrogenation of palm kernel stearin, what type of catalyst you recommend?
A30. This depends very much on what properties you require from your final product. Please reach out for a recommendation based on your individual circumstances.

Further Information

Please contact your local Johnson Matthey representative for further information or send your enquiry to polly.murray@matthey.com

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