FÁBRICA CARIOCA DE CATALISADORES

Technical Moment

EXPERTS' HINTS

Optimization of antimony additive and catalytic solutions for nickel passivation

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Introduction

In scenarios of geopolitical instability with global economic repercussions, refineries tend to intensify strategies aimed at maximizing production, optimizing processes, controlling costs, and increasing profitability. In this context, changes in the processed crude slate tend to become more frequent, resulting in changes in the feedstock composition of units of fluidized catalytic cracking (FCCU).

Among the main metallic contaminants present in crude oil, nickel (Ni), vanadium (V), iron (Fe), and sodium (Na) stand out. Variations in the concentrations of these metals and in the characteristics of the crude oil require continuous monitoring of the feedstock quality, and it is the role of process engineering to act proactively to mitigate impacts on the unit and the catalyst.

Although nickel does not promote degradation of the zeolite crystal structure, as occurs with vanadium, its high dehydrogenating activity can cause significant operational impacts, imposing restrictions associated with the gas compressor, leading to a reduction in processed feed and requiring a decrease in operational severity. For more information on the impacts of nickel on FCCU performance, please visit the FCC S.A. technical brief: *"H2 and its impacts on FCCU performance"* [1].

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OPTIMIZATION OF ANTIMONY ADDITIVE AND CATALYTIC SOLUTIONS FOR NICKEL PASSIVATION

1. Additives for Ni passivation

One of the strategies used to mitigate the effects of nickel is the injection of passivating additives, the most common being those based on antimony (Sb) or, to a lesser extent, bismuth (Bi). In industrial practice, solutions containing antimony pentoxide (Sb_2O_5) are predominantly used due to their greater passivation efficiency. Bismuth-based compounds are already being used as an alternative to antimony, due to the potential occupational and environmental risks associated with this element [2].

Operationally, controlling the dosage of this additive is critical. Improper injection can compromise both operational performance and unit costs. When dosed in excess, antimony tends to preferentially migrate to the bottom stream of the main fractionator, which can cause:

- **Financial losses** associated with non-optimized dosage;
- **Increased rate of coke deposition at the bottom of the main fractionator,** especially in heat exchangers: the antimony present in the slurry acts as a catalyst for secondary reactions, favoring the formation of heavy compounds that tend to deposit in the piping of the bottom system;
- **Increased coke deposition in the reactor and riser:** Operational reports indicate the occurrence of "coke agglomerates" formation, associated with high levels of antimony;
- **Hydrotreating catalyst (HDT) poisoning:** Antimony can be carried into hydrotreating streams and promote the deactivation of Co-Mo and Ni-Mo catalysts, directly affecting hydrodesulfurization (HDS) and hydrodenitrogenation (HDN) reactions [3];
- **Environmental and combustion impact:** it can cause an increase in NOx and SOx emissions and interfere with the performance of combustion promoters.

One important aspect is that excess antimony is not detectable through e-cat analysis, as its deposition to the catalyst depends on the presence of nickel available for complex formation. In the absence of nickel available for the reaction, antimony migrates to the slurry oil stream.

Practical indicators of overdose:

- Keeping the Ni concentration in the feed constant, increase the additive dosage and check if there has been an increase in the Sb content of the e-cat. If this does not occur, it indicates an overdose and that there is no more nickel available to react with the antimony;
- Perform antimony analysis on the slurry periodically. If the dosage is controlled, significant quantities of this metal are not expected to be found in this stream.

2. Calculation of antimony solution dosage

To avoid problems caused by excess additive, the necessary amount of antimony to react with nickel must be calculated in order to maintain the balance of the reactions, without excesses. To calculate the optimal injection rate of this additive, it is important to consider the following factors:

- **Nickel content in the feedstock:** The amount of antimony solution added should always be based on the amount of nickel in the feedstock. The optimum dosage ratio corresponds to an Sb/Ni ratio of 0.3 to 0.5 in e-cat [2];
- **Presence of nickel trap in the catalyst:** Catalysts with nickel passivation technology, such as those from FCC S.A., reduce the need for antimony additive, and the recommended Sb/Ni ratio becomes 0.1 to 0.2;
- **Antimony retention:** the efficiency of antimony deposition on the catalyst depends on the solution composition and the injection method. Antimony retention in the catalyst is 75 to 85%, without recycling the slurry in the riser. If the unit practices recycling, retention usually rises to 90% [2]. The manufacturer usually indicates the retention rate of their product, generally 85%, however it is recommended that each user calculate their actual retention rate;
- **Solution concentration:** the amount of antimony in the solution is crucial for the calculation and is provided by the manufacturer. Whenever there is a change of supplier, it is important to check the concentration and adjust the calculation accordingly. Concentrations generally range from 17 to 30%.

Formula for calculating the flow rate of antimony solution in L/d:

$$Q_{sb}(L/d) = \frac{\dot{m}_{feedstock} \times Ni_{feedstock} \times \frac{Sb}{Ni}}{1000 \times \eta_{sb} \times C_{sb} \times \rho_{sb}}$$

Where:

$\dot{m}_{feedstock}$ = Mass flow rate of feedstock in t/d;

$Ni_{feedstock}$ = Nickel concentration in the feedstock in ppm;

$\frac{Sb}{Ni}$ = Ratio according to theory, from 0.1 to 0.5;

η_{sb} = Retention of the antimony solution (from 0.65 to 0.85);

C_{sb} = Mass concentration of the antimony solution (from 0.17 to 0.30);

P_{sb} = Density of the antimony solution in kg/L.

The calculated value should always be validated and adjusted based on the responses of the unit's operational variables, primarily: H_2 concentration in the fuel gas, coke formation, and Sb/Ni ratio in the e-cat.

3. Considerations on the use of Nickel passivating additives

- There is evidence that the presence of chlorine can lead to the reactivation of oxidized nickel species, increasing the unit's coke and hydrogen yields [4]. This fact reinforces that monitoring and controlling chlorine in the feedstock is also essential to minimize the effects of nickel on FCC units;

- Antimony reacts with the platinum present in combustion-promoting additives based on platinum, reducing their efficiency. Therefore, in units that use platinum-based combustion promoters, a higher dosage of promoter may be necessary to maintain afterburning control;
- According to the literature, the use of antimony is generally advantageous in units that have a nickel concentration in the e-cat above 1000 ppm [2]. However, the indication for use involves other variables, one of which is the quality of the catalyst employed. Furthermore, maintaining inventory quality through the fresh catalyst makeup and the use of catalysts with nickel trap in their composition significantly reduces the need for antimony additive.

4. Nickel passivation technology in FCC S.A. catalysts

The components incorporated into the catalyst formulation to suppress the dehydrogenating activity of nickel are called "nickel traps". FCC S.A. catalysts contain a high-performance nickel trap called ADM-60 in their formulation, the concentration of which can be adjusted according to the specific needs of each unit.

The mechanism of action occurs through the formation of spinel-like structures, in which nickel reacts with the catalyst matrix, forming nickel aluminate ($NiAl_2O_4$), a stable spinel-like crystalline phase (Figure 1). Under typical FCC operating conditions, the formation of the non-stoichiometric phase $Ni_xAl_2O_{3+x}$ is also common. These reactions occur primarily at high regenerator temperatures in an oxidizing atmosphere (680-730 °C). Due to the high thermodynamic stability of spinel compounds, their reduction to the Ni^0 metallic phase is significantly hindered under riser conditions, characterized by a reducing atmosphere and temperatures between 500–550 °C. Consequently, greater tolerance of the catalyst to nickel contamination is observed, with a significant reduction in dehydrogenating activity associated with this contaminant. This behavior has been reported in the literature through temperature-programmed reduction (TPR) analyses.

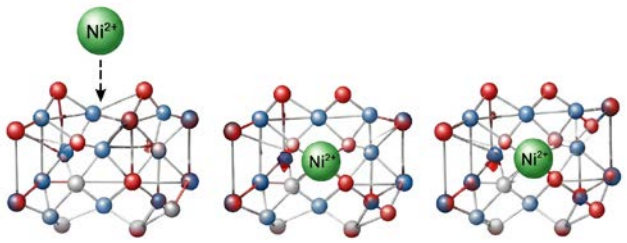

Figure 1 - Formation of a spinel-like structure.
Source: FCC S.A. Database

This trap approach (formation of a spinel structure) is superior when compared to other passivation approaches, such as those based on adsorption by Lewis acid-base interaction (Figure 2). In this model, the Ni^{2+} cation (Lewis acid) is retained by coordination interactions with basic sites of the catalyst matrix, such as surface groups, commonly based on MgO or La_2O_3 [5]. Although this interaction limits the mobility and sintering of nickel, the stability of the species formed is considerably lower than that of spinel: under the reducing conditions of the riser, an atmosphere rich in hydrocarbons and hydrogen at 500–550 °C, adsorbed Ni^{2+} can be partially reduced to metallic Ni^0, a species highly active in dehydrogenation reactions. Thus, although the Lewis acid-base model reduces the mobility and sintering of nickel, its effectiveness in suppressing dehydrogenating activity is inferior to that provided by the spinel model, in which nickel is incorporated into a thermodynamically stable crystalline structure that is difficult to reduce.

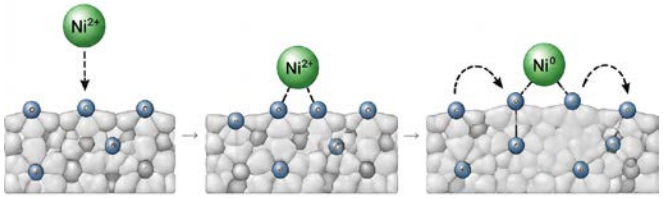

Figure 2 - Lewis model of adsorption or acid-base binding
Source: FCC S.A. Database

To illustrate the effectiveness of nickel passivation of FCC S.A. catalysts, Table 1 shows the results related to this subject from a catalyst evaluation with an 80% inventory exchange from a catalyst from another manufacturer to UPGRADER™, which contains a Ni passivator in its formulation.

	Base Catalyst	UPGRADER	DELTA
Ni equivalent (ppm)	3000	4260	42% ↑
H_2/CH_4 (%mol)	0,74	0,63	-15% ↓

Table 1 - Resultado de la evaluación del UPGRADER™
Source: FCC S.A. Database

The FCC S.A. catalyst, when compared to the base catalyst, showed a significant reduction in the H_2/CH_4, ratio, even with an increase in the amount of nickel equivalent, indicating better product performance, which incorporates FCC S.A.'s ADM-60 nickel passivation technology. In addition to UPGRADER™, technologies such as DENALI® and SaFeGuard™ have also been developed for processing heavy feed and possess high resistance to metals.

Final considerations

Nickel passivation through antimony injection is an important tool for reducing the effects of nickel in FCC units, especially in scenarios involving the processing of more contaminated feedstock. However, its application requires strict control, since overdosing can result in significant operational impacts, as well as economic losses. Therefore, periodic review of the dosage calculation and adjustment of the product addition is recommended.

Additionally, the use of catalysts with integrated passivation technologies represents an effective and well-established strategy to reduce dependence on additives, increase operational robustness, and improve process profitability. To achieve maximum performance from catalysts, it is important to preserve inventory quality through optimal and continuous make-up with fresh catalyst.

FCC S.A., through its Technical Services team, offers total support to refineries in evaluating, optimizing, and maximizing the performance of FCC units. Our solutions include, for example, antimony dosage and metal balance studies, as well as constant analysis of the suitability of the catalyst formulation aligned with the specific operational challenges and objectives of each unit.

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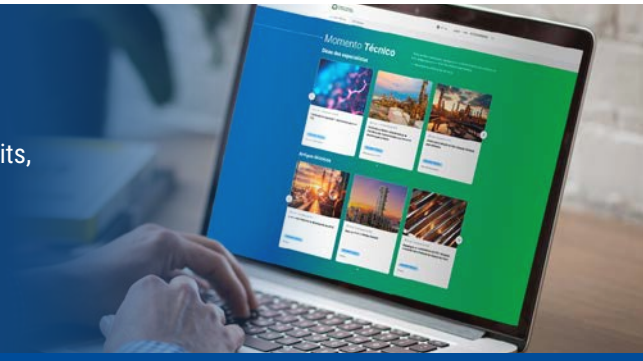

About FCC S.A.

FCC S.A is a leading-edge technology company, with headquarters in Rio de Janeiro, comprising the Petrobras S.A. and Ketjen companies. Being the sole manufacturer of catalytic cracking catalysts and additives for petroleum refining in the South-American market, its consumer customers are the refineries of the Petrobras Systems, as well as the petroleum refineries of South-American countries.

For more information, contact the FCC S.A. Technical Services team

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