**Characterisation of the FCC catalyst for fluid catalytic cracking of atmospheric residue: application to the SORAZ FCC catalyst**

**Abstract**

This paper presents a brief summary of the study on the characterisation of the fluid catalytic cracking catalyst used at the Zinder refining company (SORAZ) for cracking the atmospheric residue from the atmospheric distillation unit (CDU). The main objective of this study is to characterize characterise the catalyst used by the FCC unit of the Société de Raffinage de Zinder (SORAZ) for cracking atmospheric residue into lighter products such as high-octane gasoline. The catalyst used for upgrading heavy feedstocks (RA, RSV, heavy diesel, etc.) by fluid catalytic cracking (FCC) must have a number of characteristics that meet the standards for best product yields. Characterisation is therefore just as important as the FCC unit's performance. The fine-grained, white powder catalyst plays a crucial role in the environmental context, as it eliminates or reduces the content of atmospheric pollutants and increases the production of gasoline and other high-value cracking products. The C230 LECO is a device for measuring the carbon content accumulated on the catalyst. Malvern Laser (HYDRO2000). This device is used in the laboratory (SORAZ) to determine catalyst particle size. There are a multitude of catalyst characterization techniques available, such as experimental characterization (DRX, BET, TPD-NH3, FTIR-pyridine, ICP-OES, SEM/TEM, TGA), catalytic tests (micro-activity (MIA), mini-FCC, selectivity), aging analysis and essential catalyst parameters that can be determined, namely specific surface area (BET), total pore volume, pore size distribution, zeolite crystallinity, acidity, metal contaminants such as nickel, vanadium, iron and calcium, residual carbon content (or residual coke), hydrothermal stability and bulk density. The study revealed correlation between the catalyst activity index and residual carbon content is practically insignificant. In fact, residual carbon contents of 0.03% and 0.05% (minimum and maximum values, respectively) correspond respectively to activity indices of 56% and 59% (approx.). On the other hand, the 0.04% contents were obtained at a variable activity index (55, 56, 57 or 59%). The study also showed that the micro-activity index of the fresh catalyst was 57, with zero residual carbon content. The regenerated catalyst had a residual carbon content of 0.4%. Overall, the characterisation results showed that the fresh or regenerated catalyst met the specification standards, enabling it to follow the cracking operations, leading to the best results. At the end of this study, the catalyst characterisation results (fresh, to be regenerated and regenerated) were satisfactory. Characterisation of the fresh catalyst revealed an activity index averaging 61% and an acceptable average size distribution.

Keywords: Catalyst; Microactivity index; Heavy diesel; Fluid catalytic cracking ; atmospheric residue

1. **Introduction**

The Fluid Catalytic Cracking unit (FCC) is a key process that plays an important technical and economic role in the refining industry. Over the past years, there has been an increased interest in applying advanced process modelling and control that would lead to a significant economic benefit of FCC operation. This has resulted in a substantial number of contributions within this field (Khaldi et al., 2023). It is one of the most important techniques used in the refining industry for upgrading heavy feeds such as atmospheric residue (AR), vacuum residue (VRS), heavy gasoil, etc, with high boiling points into lighter products (with low boiling points) of high market value using a catalyst (Amadou et al., 2025; Speight, 2019; Jarullah et al.,2017; Leprince, 1998). The FCC catalyst is widely used in the oil refining industry, and its performance affects the unit operation and the economic benefits of the refinery. Nickel, vanadium, iron, calcium, sodium, copper, and other metals exist in heavy crude oil in the form of porphyrin complexes, naphthenates, or inorganic compounds, which have toxic effects on the catalysts. With the increasing trend of heavy and poor quality of FCC feedstock, the influence of heavy metal deposition, especially iron, on the catalyst performance has gradually increased (Liao et al., 2021 ;Oloruntoba et al., 2022). As these heavy feedstocks or heavy petroleum residues are difficult to transform by processes operating at low severity (low temperature, for example), their upgrading often calls for much more severe techniques (high temperature ≥500°C) such as catalytic cracking, hydrocracking, etc., of which fluid catalytic cracking using a catalyst is the most widespread worldwide. It is the workhorse of modern crude oil refinery. Its regenerator plays a critical role in optimising the overall profitability by efficiently restoring the catalyst activity and enhancing the heat balance in the riser reactor (Amadou et al., 2025).

Thus, the main objective of this study is to characterise the catalyst used by the FCC unit of the Société de Raffinage de Zinder (SORAZ) for cracking atmospheric residue into lighter products such as high-octane gasoline.

The atmospheric residue in general can contain various hetero-atomic compounds containing elements such as sulfur (S), nitrogen (N), oxygen (O), VOCs, etc. and traces of metals such as Nickel (Ni), Vanadium (V), Iron (Fe), etc., considered as atmospheric pollutants (Amadou et al., 2025), responsible for the corrosion of refinery equipment and some of which are considered poisons for the catalyst.

In order to reduce the content of these compounds, which are harmful to human health and/or atmospheric pollutants, in cracking products, and to protect the environment, specific zeolite-type catalysts are used during cracking operations.

However, in order to better ensure the catalyst's role in these operations and/or its suitability to meet the needs of refiners during RA cracking, it needs to be characterised. The characterisation in question will therefore concern both the catalyst to be regenerated and the regenerated catalyst.

1. **Materials and methods**

The FCC catalyst used at SORAZ consists of fine particles of different diameters (or sizes) in the form of a white powder. In order to determine the essential characteristics of the different types of catalysts used by the SORAZ FCC unit for cracking RA, a number of materials and techniques were employed.

In order to carry out this study, we used all the products and materials detailed below:

* ***Equipment:***
* ***Malvern Laser (HYDRO2000).***

This device is used in the laboratory (SORAZ) to determine catalyst particle size. The use of this equipment requires strict adherence to the technique detailed below:

* Switch on the instrument and let it warm up for 15 minutes; start the computer system and enter the laser particle size analysis software.
* After cleaning the sample cell, add distilled water to 2/3 of the sample cell, then shake.
* Selection of measurement conditions: stirring speed must be 3000 rpm and ultrasonic intensity must be 20J;Saisir le nom et le numéro de lot des échantillons ;
* Light calibration: laser intensity should be over 70%.
* Test background intensity;
* Slowly add uniformly mixed samples to the sample cell using a spoon and maintain the shading rate at 10-20%; Ultrasonic exposure time should be 10 seconds (ultrasonic exposure time can be increased for kaolin depending on dispersion condition), to disperse particles.
* Call up the results of the analysis according to particle size (0-45.8) microns, (45.8-111.0) microns and over 111.0 microns;
* Once the measurement is complete, add clean distilled water to the sample cell to be used for the following determination;
* Switch off instrument power supply, computer power supply, printer power supply and main power supply;
* Ratio: round to three significant figures the mathematical mean of two results per parallel determination for the ratio.
* **The RIPP, WFS-1D:**

This is a Chinese instrument also used in the SORAZ laboratory to determine the micro-activity index of the catalyst. Use of this instrument follows the operating procedure detailed below:

* Catalytic cracking reaction: weigh out 5.00g of the pore-forming agent and place it in the reactor filled with glass wool; fix the reactor ;
* Place the reactor in the reaction furnace to heat it up; when the furnace temperature has stabilised at 460±5, connect the reactor to the reaction system; when no leaks are detected on checking, purge the reactor with nitrogen for 10 minutes using a flow rate of 20 ml/min;
* Adjust the flow rate of the inlet pump, and turn off the nitrogen; improve the oil inlet passage, plunge the oil recovery device into the beaker in the cold bath, and prepare for the oil inlet reaction ;
* Start the reaction button on the reaction unit. The time should be 70 minutes, and the oil input should be 1.56±0.04 g ;
* Once the reaction is complete, purge with nitrogen for 10 minutes.

When the purge time has elapsed, dismantle the oil recovery device and weigh it immediately after cleaning. Then place it in the icebox for chromatographic analysis.

* For the chromatographic analysis method, refer to Q/SY LS1290-2008.
* Calculation: Mark the gasoline and diesel oil cut-off points and calculate the proportion of gasoline and diesel oil yields using the peak area, then calculate the microactivity index.

(Eq.1)

Where:

MAI is the microactivity index, % ;

M1 is the weight of liquid products, g ;

Y is the percentage by weight of liquid products, % ;

m is the weight of the oil stream, g.

* **Le C230 LECO :**

The C230 LECO is a device for measuring the carbon content accumulated on the catalyst. The procedure used to carry out this test comprises the steps listed below:

* Mettez l'instrument sous tension et attendez que l'instrument soit réchauffé et stabilisé pendant 30 minutes ;
* Set oxygen and nitrogen cylinder outlet pressure to 3.0kg/cm2 ;
* Set the oxygen flow rate to 200 ml/min, making the necessary adjustments;
* Set the time required for the instrument to analyse a sample to 40 seconds;
* System leak detection: after entering the system leak detection state, wait 60s; if the instrument's system pressure change value is between -4mmHg and +44mmHg, the system has no gas leak. If this is not the case, the necessary treatment to stop the leak must be carried out before proceeding again with system leak detection; proceed with the following operation when the above conditions are met.
* *Analyse the correction coefficient of standard sample storage:* weigh 0.200 g standard sample (to be rounded off to the nearest 0.001 g) and 1.5 g fluxing agent into the crucible, and analyse the carbon content of the standard sample as described above; record the result. Repeat the operation so as to take at least 7 analysed data points with approximate results to calculate the correction coefficient; store the correction coefficient of the standard sample in the instrument and incorporate the codes for future use;
* *- Revision and selection of correction coefficient:* depending on the carbon content of the sample being analysed, select the recorded correction coefficient of the standard sample with approximate carbon content as the external standard correction coefficient during sample analysis. Once the correction coefficient has been selected, the corresponding standard sample is first analysed; if the difference between the actual result and the analysed result is within the tolerance range, the sample is analysed; if the difference is not within the tolerance range, the sample is corrected again, and a new correction coefficient is obtained.
* *Sample analysis:* weigh 0.2 g of sample into the crucible, to an accuracy of 0.001 g, then add 1.5 g of fluxing agent; switch on the instrument and send the sample to the combustion furnace for analysis; after analysis, the instrument automatically reports the result.

*At the end of the test, the carbon content accumulated on the catalyst noted X was calculated using (Eq.52) as follows:*

(Eq.2)

Where X represents the weight percentage of carbon in the sample, % (weight percentage), F the correction coefficient obtained from the external standard object, A the sample concentration detected by the IR detector, % (weight percentage) and W the sample weight (g).

* **Products :**
* During the various analyses, the different products that were used are classified as follows:
* Fresh catalyst;
* Catalyst to be regenerated, the carbon content of which is to be determined;
* Regenerating catalyst for which the carbon content is to be determined;
* Iron and/or aluminium used as standard metal;
* The water used by the measuring instrument;
* The oil used by the measuring instrument.

However, there are a multitude of catalyst characterization techniques available, such as experimental characterization (DRX, BET, TPD-NH3, FTIR-pyridine, ICP-OES, SEM/TEM, TGA), catalytic tests (micro-activity (MIA), mini-FCC, selectivity), aging analysis and essential catalyst parameters that can be determined, namely specific surface area (BET), total pore volume, pore size distribution, zeolite crystallinity, acidity, metal contaminants such as nickel, vanadium, iron and calcium, residual carbon content (or residual coke), hydrothermal stability and bulk density. Most of these techniques or parameters have been the subject of numerous studies (Oumarou, 2020; Speight, 2014; Sadeghbeigi, 2012; Bartholomew, 2006; Marcilly, 2001). Thus, only the parameters and characterisation techniques used during this work have been detailed in the paragraphs above. Table I illustrates some of the catalyst's characteristics.

Table I: Physico-chemical properties of fresh catalyst

|  |  |
| --- | --- |
| Paramètres | Values |
| Al203 (% by weight) | 52 |
| SiO2 (% by weight) | 0,33 |
| Re2O3 (% by weight) | 3,3 |
| Specific surface area (m2/g) | 315 |
| Pore volume (cm3/g) | 0,41 |
| Apparent density (g/cm3) | 0,77 |
| Fire loss | 7 |
| Attrition index | 5 |

The parameters shown in Table I were supplied by the manufacturer.

1. **Results and discussion**

This section presents all the results and discussion.

The catalyst used in the FCC unit is one of the most important elements in the cracking process. It is of the zeolitic type and consists of fine grains of different sizes or dimensions in the form of a white powder (fresh catalyst). Being a key element in obtaining good yields of cracked products, the FCC catalyst must have a certain number of essential characteristics, such as activity index, for example, which meet standards in order to be used. The FCC unit can use either fresh catalyst or catalyst regenerated after analysis. The properties of these types of catalysts are discussed in the following paragraphs.

**III.1 Characterisation of fresh catalyst**

The activity or micro-activity index of the fresh catalyst is an essential parameter for assessing or evaluating the performance of said catalyst before it is introduced into the FCC unit. This parameter has been studied by numerous researchers, including Letzsch (2015), Speight (2015), Gary et al.(2007). In principle, the fresh catalyst is the most effective for good performance of the FCC unit. This is because it has the required characteristics that make it suitable for a successful cracking operation. For this type of catalyst, the most sought-after characteristic is its activity or activity index, which averaged 61%. In principle, as reported in the literature, fresh catalyst has an activity index above 61%. This depends on the composition and type of catalyst. For example, for fresh catalysts of the USY zeolite type, their activity index is generally between 70 and 80% (Marcilly, 1991; Gary et al., 2007; Speight, 2014). According to Gary et al.(2007), this activity index can have an initial value of 75% for modern catalysts. So the value found in the case of this study can only be due to the composition of the catalyst studied, but also to the conditions of its manufacture.

As the catalytic converter is fresh from the factory, it rarely fails to meet marketing standards. As a result, it possesses almost all the necessary characteristics to comply with usage standards, giving it a high level of performance. But this does not rule out its characterisation.

**III.2 Characterisation of regenerated catalyst**

The regenerated catalyst is one that has participated in FCC cracking reactions (during which it is covered with coke), separated from the cracking products in the reactor by a system of cyclones, then sent to the regenerator, where it undergoes incineration (or combustion) during which all the coke accumulated on its surfaces is burned off. In this way, combustion products such as CO, CO2, H2 and a quantity of heat are released as black smoke (not totally black) through the chimney. It's important to mention that the heat recovered from the regenerator is subdivided into two parts, one of which is sent up the chimney with the smoke, and the other is sent to the boiler for the production of dry steam, which in turn is sent to unit 5 for the production of electricity to power the entire plant.

After regeneration, it is assumed that the catalyst is fit to be returned to the riser (where the majority of cracking reactions take place). This is why it is called regenerated catalyst. Depending on the results of the analysis (carbon content, for example), it can be reused for new cracking operations or stored as spent catalyst. Table II illustrates the desired properties.

Table II: physico-chemical properties of regenerated catalyst

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| Days | Carbon content(%mas) | Activity Index | Particle size () | | | | |
| **0.01-18.5** | **18.5-39.5** | **39.5-83.5** | **83.5-111** | **>111** |
| Day 1 | 0.05 | 57 | 4.01 | 21.96 | 43.44 | 14.85 | 15.74 |
| Day 2 | 0.04 | 57 | 3.92 | 21.91 | 43.36 | 14.74 | 16.07 |
| Day 3 | 0.04 | 56 | 3.67 | 21.45 | 43.63 | 14.96 | 16.29 |
| Day 4 | 0.04 | 55 | 3.96 | 22.31 | 43.68 | 14.63 | 15.42 |
| Day 5 | 0.04 | 57 | 3.70 | 21.99 | 44.07 | 14.72 | 15.52 |
| Day 6 | 0.04 | 59 | 4.52 | 22.48 | 42.79 | 14.41 | 15.80 |
| Day 7 | 0.03 | 56 | 3.58 | 21.12 | 44.04 | 15.07 | 16.19 |
| Day 8 | 0.05 | 59 | 4.97 | 22.25 | 42.59 | 14.47 | 15.72 |
| Maximum values | 0.05 | 59 | 4.97 | 22.48 | 44.07 | 15.07 | 16.29 |
| Minimum values | 0.03 | 55 | 3.58 | 21.12 | 42.59 | 14.41 | 15.42 |
| Average values | 0.0389 | 57 | 4.0412 | 21.9338 | 43.45 | 14.7312 | 15.8438 |

Analysis of Table II shows that the correlation between the catalyst activity index and residual carbon content is practically insignificant. In principle, as the carbon content increases, the activity index decreases. This is practically not the case if we observe the values obtained correctly. In fact, residual carbon contents of 0.03% and 0.05% (minimum and maximum values, respectively) correspond respectively to activity indices of 56% and 59% (approx.). On the other hand, the 0.04% contents were obtained at a variable activity index (55, 56, 57 or 59%). This observation is valid for the 0.05% grades obtained. This variation in activity index at the same grade, such as 0.04% or 0.05%, could have several explanations. It could be due either to the performance of the analysis equipment (leading to slightly erroneous values if it is faulty), or to contamination during analysis, or even to the source (the manufacturing plant or FCC unit) of the catalyst.

Residual carbon and catalyst activity index have average values of 3.89% and 57% respectively, which gives a more precise idea of the quality of the regenerated catalyst, as well as its resistance to deactivation. In fact, these two parameters are actually linked and can also be related to particle size distribution. The lower the residual carbon content, the higher the activity index.

Table II also illustrates the size distribution of catalytic particles. The size of FCC catalyst particles plays a very important role. According to several researchers (Amadou et al.,2025; Sadeghbeigi, 2012; Gary et al., 2007; Sadeghbeigi, 2000; Leprince, 1998; Marcilly, 1991, ...), the activity index is greater the smaller the particle size (≤60 μm). The average catalyst size distribution.

This would also enable catalytic pores to be easily accessed by the sites, resulting in high exchange surfaces and rapid diffusion of hydrocarbons within the pores. This obviously favours the cracking of these hydrocarbons, and hence the yield of cracked products.

**III.3 Characterisation of the catalyst to be regenerated**

The catalyst to be regenerated is that resulting from catalytic cracking reactions, which leaves the reactor for the regenerator to be free of the coke it carries. Indeed, this catalyst is loaded with coke containing all possible impurities such as sulfur, nitrogen, oxygen and some metals such as vanadium, iron, nickel, calcium, etc. So, prior to regeneration, the said catalyst needs to be characterised to ensure the type of adjustment of operating variables such as hot air flow, temperature and pressure that needs to be adopted. However, the most sought-after characteristic for this catalyst is the carbon content accumulated on the catalytic surfaces. As the micro-activity index progressively decreases with the number of cracking operations carried out, this parameter is not of as much concern to refiners as the accumulated carbon content, which is generally strictly greater than 1. This directly indicates the need to regenerate the catalyst before reuse.

Furthermore, the activity index and residual carbon content vary progressively with catalyst particle size. According to our laboratory analyses, the larger the catalyst particles, the more coke they accumulate. Thus, coke, being a catalyst deactivator, acts on the catalyst's activity, making it less active, leading to rapid deactivation and hence regeneration.

1. **Conclusion**

At the end of this study, the catalyst characterisation results (fresh, to be regenerated and regenerated) were satisfactory. Characterisation of the fresh catalyst revealed an activity index averaging 61% and an acceptable average size distribution. The regenerated catalyst had an activity index of 57% on average, and residual carbon contents of 0.03% and 0.05%, minimum and maximum values, respectively. These values indicated that the regenerated catalyst was of average quality, justifying its performance in further cracking operations until it reached its maximum deactivation threshold. The characterisation results also showed that the catalyst to be regenerated contained contaminants such as Nickel, Vanadium, Iron, ..., as well as accumulated carbon, giving clear indications of the need for regeneration operations. This also makes it possible to take precautions (severity settings) for a successful regeneration operation.

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