

Conversion of Residual Feedstocks in FCC. Evaluation of Feedstock Reactivity and Product Distributions in the Laboratory

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A method was developed to evaluate the conversion and product distribution of a resid that could be added to conventional VGO feedstocks for FCC processing. An atmospheric tower resid was dissolved into toluene and methylnaphthalene at 15% and 25%, respectively, and converted at 550 °C over an equilibrium conventional FCC catalyst in a CREC Riser Simulator laboratory reactor. Reaction times were from 5 to 25 s. The pure solvents were also converted under the same conditions to provide background information. The method was based on the careful assessment of the masses of the reaction products. Results showed that, considering that an interaction is developed between resid and solvent in relation to the catalyst surface, it is possible to evaluate the specific contribution of the resid to the product slate in terms of hydrocarbon groups or compounds of particular interest. In this way, different catalysts could be compared for their performance with different resids and VGOs, or the impact of various factors on the FCC operation could be predicted as a function of resid-related changes.

Introduction

A number of factors, including the decreasing demand for fuel oil and the high price of crude, are contributing to the increasing use of residual feedstocks in refineries, where various commercial processes (hydrocracking, catalytic cracking, coking) are able to use them. This trend is particularly perceivable in the process of catalytic cracking of hydrocarbons (FCC), where the incorporation of resids into conventional VGO feedstocks has been increasing steadily since about a decade ago,^{1,2} based on the process versatility and high efficiency in the conversion of heavy molecular weight mixtures.³ Moreover, some FCC technologies were specially developed to process only residual feedstocks.

It is always interesting for refineries to know about resid feedstock's reactivity and contribution to the product slate.⁴ This issue is important for many reasons, including the definition of the economic balance of the whole refinery, the evaluation and selection of catalysts, and the expected changes in operations when a new feedstock is to be processed. However, most of those features or decisions need to be based on previous knowledge, which is usually generated in the laboratory, about the particular resid.

Resids impose a number of effective limitations for handling, processing, and evaluation, since they are very complex mixtures of hydrocarbons with singular characteristics. Their molecular weights may be over 1000 g/gmol, and the boiling points are above 530 °C.² Resids include higher amounts of contaminant metals, polynuclear aromatics, and heteroatom (S and N) species

than conventional FCC feedstocks.⁵ Viscosities are higher, too. One of the consequences is that the Conradson coke residue (CCR) is high, exceeding 2%. As a consequence, the resid FCC catalysts are specially formulated to have more metals tolerance, to resist to higher regeneration temperatures, and to provide a better accessibility to the catalyst active sites.^{1,6}

Because of their properties, it is very difficult to manage residual feedstocks in the laboratory, from both the handling and result analysis standpoints. Particularly, if the target in the evaluation of a resid is to elucidate its reactivity and specific contribution to the product slate when added to a VGO feedstock, then it is not possible to discriminate how much it converts and what is produced by the resid itself. It is the objective of this work to develop a methodology for the laboratory evaluation of commercial residual feedstocks over commercial FCC catalysts and conditions in a CREC Riser Simulator reactor.

Experimental Section

An equilibrium conventional commercial FCC catalyst was used; its main properties were unit cell size 2.423 nm, specific surface area 139 m²/g, zeolite load 16.9%, rare earth oxides 1.26%, and Akzo accessibility index⁷ (AAI) 5.5. The residual feedstock (refer to Table 1) was an atmospheric tower resid from a naphthenic crude. Operating refineries provided both the catalyst and the feedstock.

The conversion experiments were performed in a CREC Riser Simulator laboratory reactor⁸ at 550 °C for 5, 10, 15, 20, and 25 s, with a catalyst-to-oil ratio of 5.8 achieved with a catalyst mass of 0.8 g. The reactor was designed specifically for tackling studies about FCC issues. Its basic design concept considers that a small

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Table 1. Properties of the Residual Feedstock

distillation (vol %)		
initial point	°C	294.8
5	°C	385.2
10	°C	411.7
20	°C	448.8
30	°C	473.1
40	°C	500.2
final point	°C	513.9
yield	%	45.6
density	g cm ⁻³	0.95
viscosity at 50 °C	sSU	350
iron	ppm	28
nickel	ppm	15.6
vanadium	ppm	38.2
nitrogen	ppm	3336
sulfur	%	1.4
CCR	%	8.11

slice of an ideal riser, carrying the mixture of catalyst particles and hydrocarbons that “see” each other while moving along the riser after being put into contact, can be located into a batch reactor with internal recirculation. Thus, residence time and position along the ideal riser can be considered analogous to reaction time evolved in the CREC Riser Simulator (see Figure 1). An impeller rotating at very high speed on top of the chamber that keeps the catalyst between two metal porous plates induces the internal circulation of the reacting mixture in an upward direction through the chamber, thus fluidizing the catalyst. When the reactor is at the desired experimental condition, the reactant is fed through an injection port, and immediately after the reaction time is attained, products are evacuated instantly.

For each experimental condition, the feedstocks injected were pure toluene (referred as TOL when used as a solvent) and methylnaphthalene (referred as Me-N when used as a solvent) solvents and solutions of the resid at 15% and 25% into TOL and Me-N, respectively. Products were analyzed on-line in a Hewlett-Packard 6890 Plus gas chromatograph equipped with a FID detector and a HP-1, 30 m column using CO₂ as cryogenic gas to obtain an initial analysis temperature of 273 K. The product identification was performed with PIANO standards. An internal standard was used for the assessment of yields and conversion. Coke yields were assessed by means of a method with temperature-programmed oxidation and further methanation of the carbonaceous deposits on the catalysts. Products from the resid or the solvents were classified into five main groups—dry gas (C1–C2), LPG (C3–C4), gasoline (C5–C12), light cycle oil (LCO, C12–C20), and coke—but it is possible to follow the yield of individual hydrocarbons. Typical standard deviations in the yields of products were close to 1%. Conversion was defined as $x_{\text{Res}} = (m_{\text{Res}}^0 - m_{\text{Res}})/m_{\text{Res}}^0 \times 100$, and selectivities for each product were calculated as the ratio between individual product and total products yields. The yield of H₂S due to sulfur in the feedstock was considered negligible in the mass balances in the experiments.

Results and Discussion

The dissolution of very viscous or solid reactants into proper solvents allows processing them more easily in the laboratory and can help to discern the products formed from their conversion. For example, the approach resulted very useful in the determination of the products from polymers recycled to FCC units as a means to decrease their postuse environmental impact.^{9–11} The technique is particularly useful if the solvent used were in turn inert because in that case the products observed would only be formed by the reactant object of study.

For example, under FCC conditions, benzene is essentially inert and produces only a small amount of coke;⁹ then, if a polymer is dissolved into benzene and converted over FCC catalysts, all the products in the reactor gas phase would be produced by the polymer. Considering that an universal, ideal solvent does not exist, the same approach can be applied to other solvents as long as they convert to a low level; in those cases, background experiments with the solvent alone might provide a basis (conversions, product distributions) to deduct from the results with the solution, that is, when the reactant and the solvent are present.¹¹

1. Experiments with Pure Solvents. Among the various solvents that dissolve the resid completely and that can be used to apply this approach, toluene and methylnaphthalene were chosen. The background experiments with TOL and Me-N showed that they convert to a certain extent at 550 °C, as shown in Table 2 for the example of 5 s reaction time. The main products in the conversion of TOL were benzene and xylenes from the disproportionation reaction and very small amounts of hydrocarbons with six or fewer carbon atoms per molecule (C6-), aromatics with nine carbon atoms per molecule, naphthalene, and methylnaphthalene. On the contrary, Me-N showed higher conversions, and the main products were naphthalene, hydrocarbons in the range of LCO, and small amounts of C6-. This solvent yields very small amounts of aromatic compounds in the range C6–C9. Concerning coke production, TOL shows a maximum yield of 1.9% at 25 s reaction time, while Me-N yields about 12% for the same reaction time. This is consistent with the stronger coke forming trend of aromatic molecules with more condensed rings.¹² The fact that TOL converts less and yields less products than Me-N makes it a better choice for assessing conversion and product distribution produced by the resid, as shown below.

2. Experiments with Resid–Solvent Solutions. Assessment of Conversion. Considering that the total mass of hydrocarbons injected to the reactor, m_{Tot}^0 , is conserved during the experiment

$$m_{\text{Res}}^0 + m_{\text{Solv}}^0 = m_{\text{C20-}} + m_{\text{Res}} + m_{\text{Coke}} \quad (1)$$

The lhs and rhs of eq 1 represent the initial and final instants of the experiment, respectively. $m_{\text{C20-}}$ includes hydrocarbon boiling up to 344 °C (dry gas, LPG, gasoline, and LCO) and contains the mass of unreacted solvent and its products as well as the products from the resid. This mass is calculated with the help of an internal standard. Then, it is possible to calculate the mass of unreacted resid as

$$m_{\text{Res}} = m_{\text{Res}}^0 + m_{\text{Solv}}^0 - (m_{\text{C20-}} + m_{\text{Coke}}) \quad (2)$$

and the conversion of the resid, as defined in the Experimental Section, can be assessed as

$$x_{\text{Res}} = \frac{m_{\text{C20-}} + m_{\text{Coke}} - m_{\text{Solv}}^0}{m_{\text{Res}}^0} \times 100 \quad (3)$$

3. Experiments with Resid–Solvent Solutions. Assessment of Product Distributions. The yields obtained in the conversion of the mixtures of the resid with the solvents are shown in Table 3 with the example of 15 s reaction time. If compared with the results obtained in the conversion of the pure solvents, a much higher proportion of C6- compounds can be observed, which indicates that these compounds are generated essentially by the conversion of the resid. This increase is mainly due to the most

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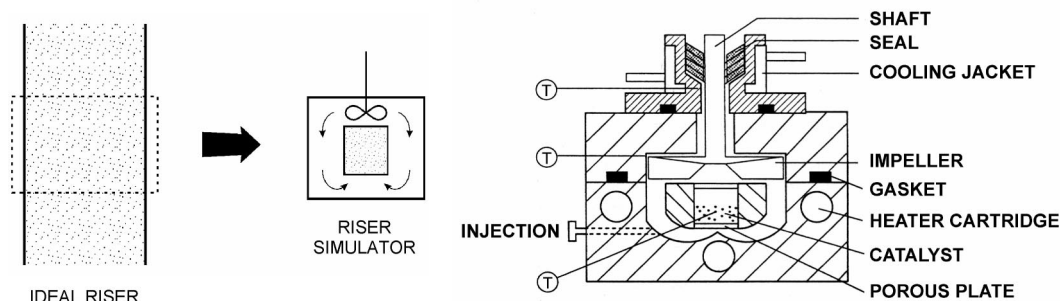


Figure 1. Schematic representation of the CREC Riser Simulator reactor.

Table 2. Conversion (%) and Product Yields (%) of TOL and Me-N at 550 °C and 5 s Reaction Time

solvent	TOL	Me-N
conversion	9.5	31.2
C6-	0.2	0.5
benzene	3.0	0.3
toluene	90.5	1.1
C8 aromatics	4.6	0.5
C9 aromatics	0.3	0.2
naphthalene	0.0	11.3
methylnaphthalene	0.0	68.8
LCO	0.0	11.2
others	0.0	0.2
coke	1.3	5.9

important yield of olefins and isoparaffins in the C3–C5 range, which represents more than 70% of all the hydrocarbons in the C6- group.

If it is assumed that there is no interaction between the solvent and the resid (for example, that there is no competition for the available catalyst's surface active sites), the mass of a given product that is generated exclusively by the resid can be calculated from the difference between the total mass of the product and the mass formed by the solvent, if any:

$$m_{i,\text{Res}} = m_i - m_{i,\text{Solv}} \quad (4)$$

The mass of a given hydrocarbon that is formed exclusively from the solvent, $m_{i,\text{Solv}}$, can be calculated from the experiments with the pure solvent under the same conditions, according to

$$m_{i,\text{Solv}} = y_{i,\text{Solv}} m_{\text{Solv}}^0 \quad (5)$$

where $y_{i,\text{Solv}}$ is the yield of the product i from the solvent.

However, this procedure leads to inconsistent results for the yields of those hydrocarbons that are the main products observed in the conversion of the solvent. For example, when TOL was the solvent, the yields of benzene and xylenes from the resid calculated according to eqs 4 and 5 resulted negative, and the yield of toluene was excessively high. These results suggest that the solvent converts to a lesser degree when the resid is also present, and thus the assumption of no solvent–resid interaction would not be valid. Indeed, it is expected that the resid would adsorb more than the solvent on the catalytic surface.¹³

Then, if the true conversion of the solvent when resid is present is known, the actual yields of hydrocarbons produced by the solvent could be interpolated from the corresponding yield curves (yield vs conversion) obtained with the pure solvent, at the true conversion, and used in eq 4 to calculate the masses produced by the resid.

In the case of TOL as the solvent, its true conversion could be estimated if it is known how much toluene is formed by the

resid. The experiments under the same conditions with resid dissolved into Me-N would allow to know this because Me-N produces only small amounts of toluene, and then all the toluene observed in those experiments would be produced essentially by the resid.

In summary, if the total mass of toluene and the mass of toluene produced by the resid are known, then their difference will yield the mass of unreacted solvent TOL; consequently, it is possible to calculate the true conversion when the resid is present. Consistently, the conversions of the solvent calculated in this way for each experiment were lower than those that considered that TOL and resid did not interact. In the example shown in Table 3, the calculated true conversion for TOL was 6.0%, while the conversion in the experiment with pure toluene for the same conditions was 15.1%.

In the same way, the true conversion of solvent Me-N when the resid is present can be assessed if it is known how much methylnaphthalene is formed by the resid. Since TOL does not produce methylnaphthalene, the experiments with resid–TOL solutions could provide this information. In the example shown in Table 3, the calculated true conversion for Me-N was 31.5%, while the conversion in the experiment with pure methylnaphthalene for the same conditions was 38.3%.

As discussed in section 1, TOL was chosen as the solvent to assess resid conversion and product distributions. The mass of a given compound obtained from the resid when the true conversion of the solvent is known, can then be calculated using eq 4. Results are shown in Table 3 (see column corresponding to mass fractions of products from the resid, calculated from the resid–TOL experiments, $w_{i,\text{Res}}^{\text{TOL}}$). These results, obtained considering the interaction between resid and solvent TOL, showed consistency, except in the cases of C6–C9 aromatics, which are the main products from the conversion of TOL and which did not show the typical relationships observed for these hydrocarbons in VGO-FCC¹⁴ (particularly for the case of benzene to toluene relationship). Since Me-N yields small amounts of those hydrocarbons, the experiments with resid dissolved in Me-N can provide the information about selectivity for the C6–C8 aromatics.

In summary, in order to assess the true mass fractions for products from the resid, $w_{i,\text{Res}}$, the observations in resid–TOL experiments for all the products except C6–C9 aromatics, and the observations in resid–Me-N for C6–C9 aromatics, can be combined using the group of C6- hydrocarbons as the nexus for the mass balances. Note in Table 3 that, as it was mentioned, the group C6- is produced in important amounts when the resid is present and in very low proportions by both solvents.

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Table 3. Conversion (% , Coke Excluded) of Solvents, Resid–Solvents Mixtures, and Resid^a

	reactant					
	solvent		resid–solvent mixture		resid (calculated)	
	TOL	Me-N	Res + TOL	Res + Me-N		
	$w_{i,TOL}$	$w_{i,Me-N}$	$w_{i,TOL}^{Res}$	$w_{i,Me-N}^{Res}$	$w_{i,TOL}^{Res}$	$w_{i,Res}$
C6-	0.3	0.9	8.8	6.1	36.5	51.5
benzene	6.2	0.5	3.0	0.6	7.7	2.4
toluene	84.9	1.3	75.5	1.7	9.9	7.0
C8 aromatics	7.6	0.8	6.8	1.5	21.3	8.3
C9 aromatics	0.7	0.3	1.8	0.7	7.3	6.2
naphthalene	0.1	17.6	0.3	9.9	1.4	2.0
methylnaphthalene	0.1	61.7	0.8	63.8	3.1	4.4
LCO	0.0	16.6	1.0	14.1	4.4	6.2
other	0.1	0.3	2.0	1.6	8.5	12.0
conversion	15.1	38.3			80.5	80.5
true solvent conversion (calcd)			6.0	31.5		

^a Product distributions (% , coke excluded). Temperature 550 °C, reaction time 15 s.

In order to make the calculations, three hydrocarbon groups were defined: (i) the main products from the conversion of TOL (group A, including benzene, toluene, and C8–C9 aromatics); (ii) main products from the conversion of Me-N (group B, including naphthalene, methylnaphthalene, LCO, and others (nonaromatic hydrocarbons in the C7–C12 range)); and (iii) products that are not formed significantly by the solvents (group C6-).

Considering the conversion of the resid, particular product mass fractions (mass of a given hydrocarbon or group formed by the resid, in reference to total mass of hydrocarbons formed by the resid) are

$$w_{i,Res} = \frac{m_{i,Res}}{m_{C20-,Res}} \quad (6)$$

According to the previous definitions, it is possible to state the following experimental relationships

$$R_{A,Res} = \frac{w_{A,Res}^{Me-N}}{w_{C6-,Res}^{Me-N}} = \frac{w_{A,Res}}{w_{C6-,Res}} \quad (7)$$

where $w_{A,Res}^{Me-N}$ and $w_{C6-,Res}^{Me-N}$ are the mass fractions of the groups A and C6-, respectively, formed by the resid in the experiments with resid and Me-N as the solvent. Since groups A and C6- are not formed significantly by Me-N, $R_{A,Res}$ is the actual relationship for that group produced by the resid itself.

Analogously

$$R_{B,Res} = \frac{w_{B,Res}^{TOL}}{w_{C6-,Res}^{TOL}} = \frac{w_{B,Res}}{w_{C6-,Res}} \quad (8)$$

where $w_{B,Res}^{TOL}$ and $w_{C6-,Res}^{TOL}$ are the mass fractions of the groups B and C6-, respectively, formed by the resid in the experiments with resid and TOL as the solvent. Since groups B and C6- are not formed significantly by TOL, $R_{B,Res}$ is the actual relationship for that group produced by the resid itself.

Moreover, it must always be kept that

$$w_{A,Res} + w_{B,Res} + w_{C6-,Res} = 1 \quad (9)$$

and, then, eqs 6 and 9 allow to calculate the mass fractions, $w_{i,Res}$, for each of the hydrocarbon groups formed by the resid.

Since the chromatographic analysis provides the concentrations of the individual hydrocarbons, it is also possible to calculate the yields and selectivities of all of them in each experiment. For example, as mentioned, the experiments with resid in Me-N are used to calculate the yields and selectivities for benzene, toluene, and xylenes (group A). For all the other individual products (included in groups B and C6-), the experiments with resid in TOL

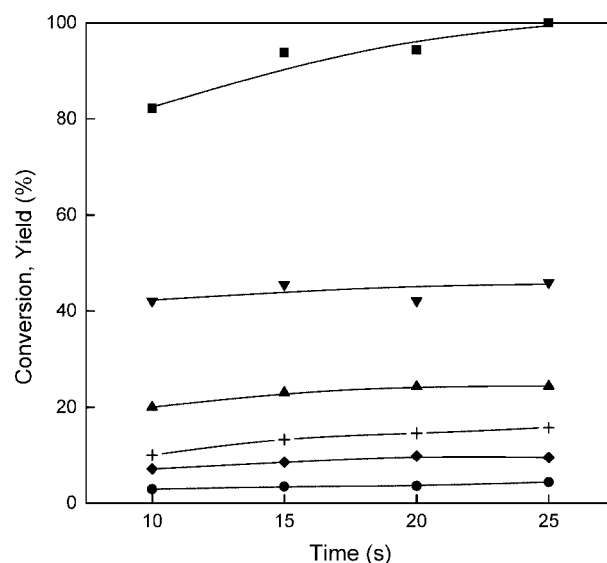


Figure 2. Resid conversion (■) and dry gas (●), LPG (▲), gasoline (▼), LCO (◆), and coke (+) yields as a function of the reaction time. Temperature = 550 °C.

are used. Since the conversion of the resid is calculated from the experiments with TOL as the solvent, selectivities correspond to that resid conversion level. The results for the case example of Table 3 are shown in the last column.

4. Resid Conversion: Analysis of Results. Some results derived from the application of the methodology described above are presented as examples. The conversion and the yields of the main groups of products (dry gases, LPG, gasoline, LCO, and coke) produced by the resid are shown in Figure 2 as a function of contact time. It can be seen that the conversion is very high, a fact that can be attributed to the high effective catalyst to reactant relationship (note the resid is dissolved to 25% in this example) and the relatively long contact times. It can be noticed that, as expected, the yield of coke is higher than those of conventional VGO-FCC; in effect, at almost complete conversion, it was as high as about 15%. These values are in line with commercial observations.^{15,16}

Considering the product distribution from the conversion of the resid, it can be significant to observe the yields of important raw materials for the petrochemical industry and for other derived

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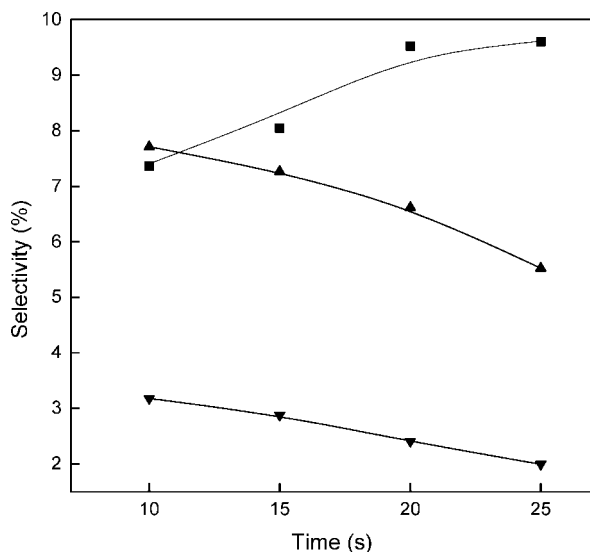


Figure 3. Isobutane (■), butenes (▲), and isoamylenes (▼) selectivity in the resid conversion as a function of the reaction time. Temperature = 550 °C.

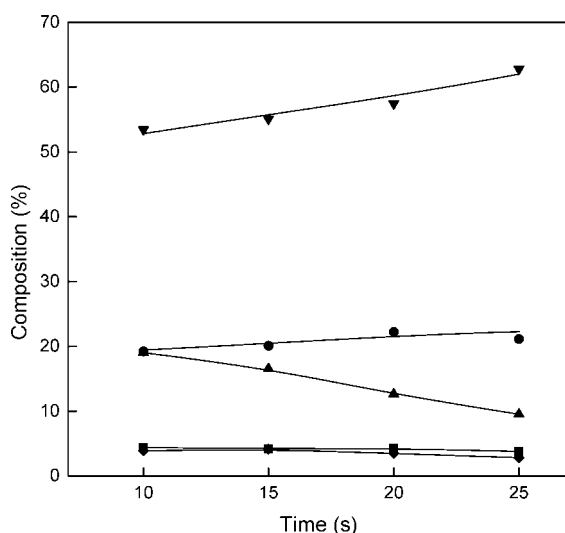


Figure 4. Gasoline composition in the resid conversion as a function of the reaction time. Symbols: aromatics (▼), n-paraffins (■), isoparaffins (●), olefins (▲), and naphthenics (◆) compounds. Temperature = 550 °C.

processes for fuel production, like, for example, isobutane and olefins for alkylation or isoolefins for MTBE or TAME synthesis. The selectivities of isobutane, butenes, and isoamylenes are shown in Figure 3. Olefins decrease as a function of time, and conversion, due to the increasing incidence of secondary hydrogen transfer reactions.¹⁷ Isobutane, on the contrary, which is one of the indexes of the extent of catalytic cracking reactions,¹⁸ increases steadily as conversion increases, as expected.

For given catalyst and process conditions, the composition of the gasoline cut should reflect the characteristics of the resid feedstock. This is an interesting problem because it must be taken into account that the addition of resid to conventional VGOs will certainly affect the quality of gasoline.¹⁹ The composition of the gasoline cut for these test experiments is shown in Figure 4 in terms of most important hydrocarbon groups. For this specific

resid, the resulting gasoline cut shows a proportion of aromatic compounds about 25% higher than that usually observed with VGOs,^{19,20} while the amount of isoparaffins is about 30% lower.

These examples show which other issues that, according to the objectives of the evaluation of resid conversion could be given particular importance, can be analyzed.

Conclusions

By means of a simple methodology based on the careful assessment of the masses of the reaction products in the experiments of conversion of a resid feedstock dissolved into toluene and methyl-naphthalene, which combines those results with background information from the conversion of the solvents themselves, it is possible to evaluate various subjects concerning the incorporation of resids into conventional FCC feedstocks.

The use of the CREC Riser Simulator laboratory reactor that mimics FCC commercial conditions (contact time, temperature, catalyst-to-oil ratio) allows to predict changes in important factors that may have a strong economical impact on refinery operation. For example, different catalysts could be compared for the same feedstock, or different resids could be compared for a given or various catalyst options. Moreover, the impact of various amounts of a resid added to a certain VGO could be evaluated about the changes in product distribution, yields, and selectivities of hydrocarbon groups or individual products.

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Nomenclature

<i>m</i>	mass (g)
Me-N	methylnaphthalene solvent
<i>R</i>	experimental relationship defined by eqs and
TOL	toluene solvent
<i>w</i>	mass fraction, defined in relation to total compounds with boiling points lower than 344 °C, $w_i = m_i/m_{C20} \times 100$
<i>x</i>	conversion (%)
<i>y</i>	yield (%)

Subscripts

A	group of hydrocarbons including benzene, toluene and C8–C9 aromatics
B	group of hydrocarbons including naphthalene, methyl-naphthalene, LCO, and others
Coke	refers to coke
C6-	group of hydrocarbons with 6 or fewer carbon atoms per molecule, except benzene
C20-	hydrocarbons with boiling point lower than 344 °C
<i>i</i>	index, compounds with boiling point lower than 344 °C
Res	produced by resid
Solv	produced by solvent
Tot	total amount

Superscripts

0	initial condition
Me-N	refers to experiments with methylnaphthalene as a solvent
TOL	refers to experiments with toluene as a solvent

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