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Commercial additives for sulfur control in FCC gasoline: Overall analysis of their impact on LCO and gasoline

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ABSTRACT

Experiments with a commercial equilibrium catalyst and mixtures with a commercial additive for sulphur reduction in gasoline were performed in a CREC Riser Simulator reactor to study the impact of three different proportions of the additive in the overall performance of FCC units. The experiments were performed under conditions similar to those of the industrial operation (510 °C, CAT/OIL 6.4). The reaction effluents were analyzed by on-line gas chromatography using two detectors simultaneously: FID for hydrocarbons and PFPD for sulfur compounds. The highest reduction of sulfur in gasoline (about 10%) without yield penalties was obtained with the smallest amount of additive (10 wt.%); however, the reduction of sulfur in LCO was the least. At higher amounts of additive (40 wt.%) the effectiveness in reducing sulfur in gasoline was lower, but the sulfur reduction in LCO was higher (about 5%). Reducing the gasoline endpoint obviously reduced sulfur not only in the cut, but also in LCO. This positive effect was improved when the gasoline was produced with catalyst–additive mixtures, though a penalty in gasoline yield was observed. If the endpoint reduction of gasoline is very severe, it did not generate a more significant reduction of sulfur in gasoline an LCO, even when additives were present.

1. Introduction

Sulfur is the most important problem in relation to the environmental impact from the use of liquid transportation fuels such as diesel and gasoline. From the various refining processes which contribute to the gasoline pool, the catalytic cracking of hydrocarbons (FCC) is the one which adds the highest amounts of sulfur, up to approximately 90% of the total [1]. Then, different approaches were developed to minimize this problem, which can be applied previously, during, or after the FCC process [2]. The hydroprocessing of the FCC feedstocks, which usually include heavy sulfur compounds, is very effective, impacting on all the FCC products [3]. On the other hand, hydrotreating the FCC gasoline implies dealing with smaller process loads, but olefins in gasoline and aromatics can be hydrogenated to some extent, thus affecting octanes negatively. In the case of the middle distillates cut (LCO), hydrotreating, which is used to increase its quality, is more common. Finally, the heaviest portion of gasoline, where most of the sulfur is present, could be removed and added to the LCO fraction by means of decreasing the final boiling temperature of the cut; actually this means that the problem is placed into another fuel product and, moreover, a loss in gasoline yield is produced. It is clear that the hydrogenation options require extra capacity and hydrogen supply, thus becoming bottlenecks in the refinery operation. Reviews which analyze this problem can be found in the literature [3,4].

Alternatively, the removal of sulfur-containing hydrocarbons during the FCC process can be achieved by means of specific catalyst additives [1,5,6]. Catalyst producers offer a number of them [7,8], which are usually based on Lewis acid materials highly dispersed on proper supports such as alumina. Thus, zinc, gallium and boron oxides, or their mixtures, could perform as proper sulfur control additives. The catalytic mechanism could be based on the fact that sulfur-containing hydrocarbons behave as Lewis bases and, in this way, would adsorb and/or react on the active sites of the additive. In the first case (adsorption), the adsorbed sulfur compounds can be released oxidized in the regeneration section of the unit; in the second case (adsorption and reaction), the additive is supposed to crack the sulfur compounds into H_2S, which can be removed with treatments or conventional scrubbing technologies [7].

The sulfur reduction additives seem to be a simple solution for the sulfur control problem, and one of the most economical for refinery implementation; however, this approach by itself might not be efficient enough to reduce all the sulfur in gasoline and LCO. Taking this into account, these additives could be used in the FCC to decrease sulfur in fuels as much as possible in order to, for example, reduce the severity of LCO and/or gasoline hydrotreating. The effectiveness of the additives to decrease the concentration of sulfur, as well as their impact on product yields and qualities depends on the

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nature of the feed, the operative conditions, the catalysts properties and the amount of additive mixed with the catalyst [9]. In general, the amount of additive in the catalyst inventory could vary from 10 wt.% [5] to 35 wt.% [10] and, considering that it is more expensive than the catalyst itself [4], it is necessary to take into account how the various FCC yields are affected when different loads of additive are used. So far, however, it is to be noted that the effect on the sulfur concentration of the LCO has not been reported in the literature. It is the aim of this work to analyze the effect of a commercial additive for sulfur control on the FCC gasoline and LCO yields, as well as its efficiency in decreasing sulfur, when different amounts of additive are added to an equilibrium catalyst under typical operative conditions, using a high sulfur VGO in the CREC Riser Simulator laboratory reactor.

2. Materials and methods

2.1. Reactor setup

The basic design concept of the CREC Riser Simulator laboratory reactor considers that if a small slice of the environment of an ideal riser reactor, involving certain mass of catalyst and gases and moving along it during a given residence time, is located into this batch reactor, then, the reaction time evolved is analogous to residence time and position change along the ideal riser. The CREC Riser Simulator laboratory reactor, which was designed specifically to address FCC studies [11], has a turbine on top of a chamber that holds the catalyst bed between porous metal plates. The turbine rotates at 7500 rpm, thus inducing a low-pressure area in the upper central zone in the reactor that makes gases to recirculate in the upward direction through the chamber, thus fluidizing the catalyst bed. After the desired experimental conditions are achieved in the reactor, the reactant is fed with a syringe through an injection port and vaporizes instantly, thus setting the initial time. After the desired reaction time is reached, the gaseous mixture is evacuated immediately and the products can be sent to. Additional descriptive details can be found in, e.g., Al-Khattaf [12] and Passamonti et al. [13]. Experiments were conducted at 510 °C, with a mass of catalyst (or catalyst + additive) of 0.8 g, a catalyst-to-oil ratio of 6.4 and reaction times from 6 to 27 s. The amount of coke on the catalysts was determined by means of a temperature programmed oxidation method and the further conversion of the carbon oxides into methane, which was assessed with a FID detector. The analysis of the reactor effluents was performed on-line using an Agilent Technologies 6890 Plus gas chromatograph equipped with two detectors operating simultaneously (FID for the hydrocarbons and PFPD for the sulfur compounds). The column used was a HP-1, 30 m length, 0.25 mm i.d., 0.25 μm phase thickness, with a splitter at the column end that produced two approximately equal-size samples to each detector. Details of the calibration and simultaneous operation of the FID and PFPD detectors can be found in Del Rio et al. [14]. Reaction products were grouped into dry gas (C1–C2), LPG (C3–C4), gasoline (C5 – 221 °C), middle distillates (light cycle oil, LCO, 221–344 °C), unconverted feedstock (VGO, +345 °C) and coke.

The identification of sulfur compounds in the products was performed by comparing their retention times with those of Sigma–Aldrich standards and with elution orders published in the literature [2,15]. In the analysis, the sulfur compounds are considered either individually or grouped according to their family type and boiling point range, following a similar procedure to that of Depauw and Froment [16]. In this work, sulfur compounds/families in gasoline were thiophene, C1-thiophene, tetrahydrothiophene (THT), C2-thiophene, C3-thiophene and benzothiophene (BT); sulfur compounds/families in LCO were C1-benzothiophene (C1-BT), C2-benzothiphene (C2-BT), C3-benzothiophene (C3-BT), dibenzothiophene (DBT), C1-dibenzothiophene (C1-DBT) and C2-dibenzothiophene (C2-DBT). Percentage conversion was calculated as the addition of the yields of dry gas, LPG, gasoline, LCO and coke. Mass balances in the experiments (recovery) closed to more than 92% in all the cases.

Table 1
Properties of the feedstock used.

Density 20/4 °C (g cm ^{3})	0.916
API gravity (°)	22.3
Conradson carbon (wt.%)	0.11
Aniline point (°C)	80.1
V (ppm)	0.73
Ni (ppm)	0.1
Sulfur (wt.%)	2.03
Basic nitrogen (ppm)	400
Total nitrogen (ppm)	1441

2.2. Materials

The catalyst used was a commercial equilibrium FCC catalyst formulated to process heavy feedstocks and to minimize bottoms yields (Ecat), kindly provided by Petrobras (Brazil). Its properties were: surface area, 178 m^2g^{-1}; zeolite content, 18.1 wt.%; unit cell size, 2.426 nm; rare earth oxides content, 2.43 wt.%; nickel content, 1204 ppm and vanadium content, 894 ppm. The additive used was Resolve 950, gently supplied by Fábrica Carioca de Catalisadores FCC SA, Brazil. The additive was composed mainly by magnesium oxide (up to 60 wt.%), aluminum oxide (up to 20 wt.%), amorphous silica (up to 10 wt.%) and other metal oxides (up to 10 wt.%). The additive was used as received and mixed with the equilibrium catalyst in three different proportions: 10 wt.%, 25 wt.% and 40 wt.% The characteristics of the VGO feedstock, which was paraffinic in nature, with high content of sulfur, are shown in Table 1.

3. Results and discussion

3.1. Conversion and product yields

The activities of the catalyst and of the different mixtures with the additive are shown in Fig. 1, expressed as the conversion of the VGO as function of time. The activity of the catalyst is higher than those of the catalysts–additive mixtures, which were similar at the three different proportions of additive. It has been reported by various authors that the additives have lower cracking activity than the catalysts and when they are added to a given catalyst

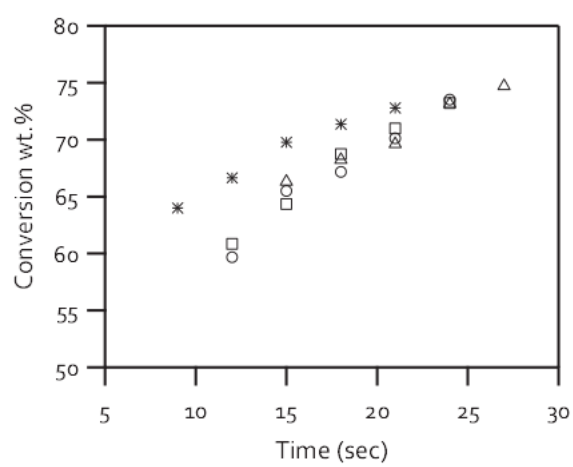

Fig. 1. Comparison of catalyst and catalyst–additive activity. (*) Ecat, (○) Ecat + 10% additive, (□) Ecat + 25% additive, and (△) Ecat + 40% additive.

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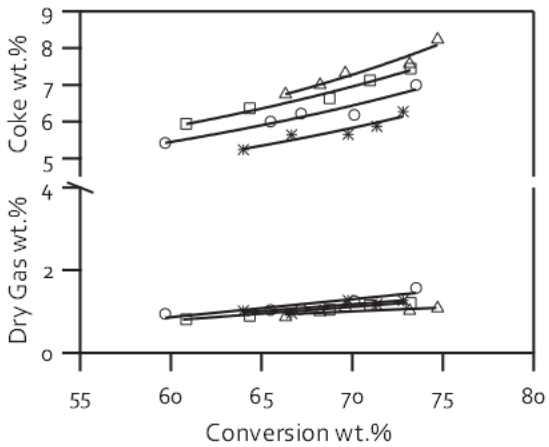

Fig. 2. Dry gas and coke yields of catalyst and catalyst–additive mixtures. (*) Ecat, (○) Ecat + 10% additive, (□) Ecat + 25% additive, and (△) Ecat + 40% additive.

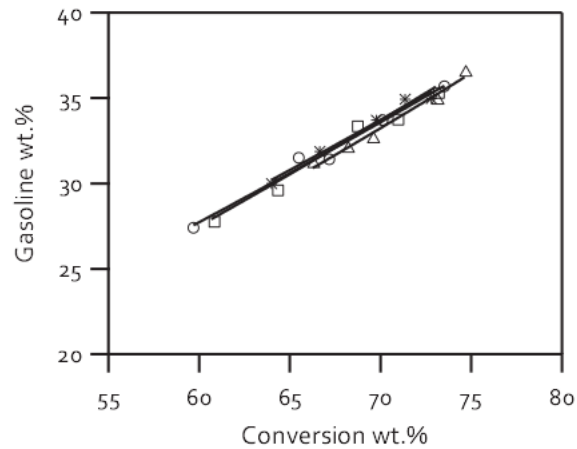

Fig. 4. Gasoline yield of catalyst and catalyst–additive mixtures. (*) Ecat, (○) Ecat + 10% additive, (□) Ecat + 25% additive, and (△) Ecat + 40% additive.

a dilution effect is observed [6,17], a fact which is coincident with these results. However, the noticed decrease in activity seems to be independent of the amount of additive added, at least in the range evaluated. It is interesting to observe that the difference in conversion between the catalyst and the catalyst–additive mixtures is higher at shorter reaction times, but activities tend to similar values at longer reaction times.

The yields of dry gas and coke are shown in Fig. 2. In the case of dry gas there is no appreciable effect of the additive on the group's yield; however, the impact is apparent on coke yield, where the higher the proportion of additive, the higher the yield. This is consistent with previous reports about higher coke yields when sulfur reduction additives were added to FCC catalysts in the laboratory operation [1,18], probably due to their relatively high Lewis acidity, which may be responsible of coke formation [19]. In some cases, due to its impact on the key thermal balance in the FCC process [20], this fact can represent a drawback of the use of additives. Moreover, this stresses the need for reliable laboratory tools to evaluate the implementation of process' options or adjustments.

The yields of LPG are shown in Fig. 3. When 10 wt.% of additive is used, a slight increase in the LPG yield is observed in comparison to the catalyst alone. If higher amounts of additive are used, the LPG yields are lower as the amount of additive increases. This observation could be due the fact that some alkyl-thiophene compounds whose productions are enhanced by similar additives [8], could be then cracked by the FCC catalysts into H_2S and light hydrocarbons,

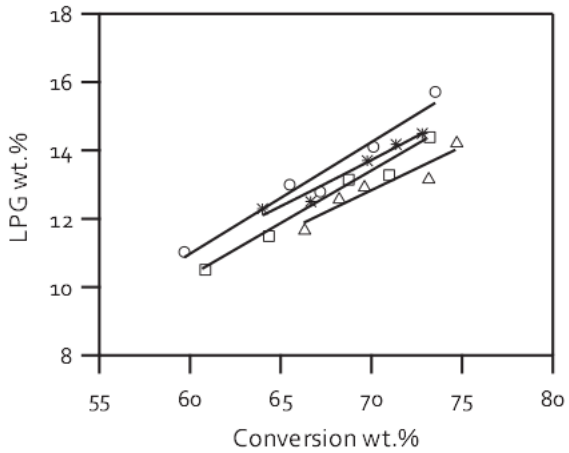

Fig. 3. LPG yield of catalyst and catalyst–additive mixtures. (*) Ecat, (○) Ecat + 10% additive, (□) Ecat + 25% additive, and (△) Ecat + 40% additive.

thus increasing LPG yields; nevertheless, the dilution effect on the catalyst at higher additive concentrations, would impede it to favor these reactions.

It is clear that the incorporation of sulfur control additive to the catalyst at the levels which were evaluated in this work does not induce changes in the yield of gasoline, as it can be seen in Fig. 4. This is one of the most important characteristics of the additive, considering that gasoline is in general the most valuable product in the FCC process, and that the additive should reduce the sulfur content in gasoline without yield penalties.

3.2. Gasoline composition

The selectivities of gasoline and LCO at 70% of VGO conversion, which can be considered typical of the commercial operation, are shown in Table 2 together with the composition of gasoline. It can be seen that n-paraffins are essentially not affected by the presence of the additive. At 10 wt.% of additive an increase in iso-paraffins is clearly observed, while a decrease in olefin content is also found; at higher additive loads, the amount of iso-paraffins decrease at levels similar to those when the additive is not present and the

Table 2
Concentration of sulfur compounds and gasoline yield and composition for different additive additions, at 70% of VGO conversion.

	Additive (wt.%)			
	0	10	25	40
Gasoline (wt.%)	34	33.5	33.7	33
P (%)	3.8	3.8	3.9	3.9
I (%)	36.4	38.3	36.1	36.4
O (%)	10.4	9.0	11.1	11.0
N (%)	8.4	8.1	8.8	8.7
A (%)	38.8	38.7	37.8	37.2
Sulfur in gasoline (ppm)	1339	1209	1292	1325
Tiophene	152	122	119	122
C1-tiophene	203	180	181	197
THT	55	50	49	49
C2-thiophene	271	245	263	279
C3-thiophene	228	209	270	269
BT	430	403	410	409
LCO (wt.%)	15.4	14.3	15.4	15.9
Sulfur in LCO (ppm)	35,383	34,721	34,782	33,607
C1-BT	4285	4093	4103	3823
C2-BT	5971	5878	5996	5594
C3-BT	8810	8767	8996	8716
DBT	1535	1356	1410	1410
C1-DBT	5852	5437	5539	5352
C2-DBT	8930	9190	8738	8712

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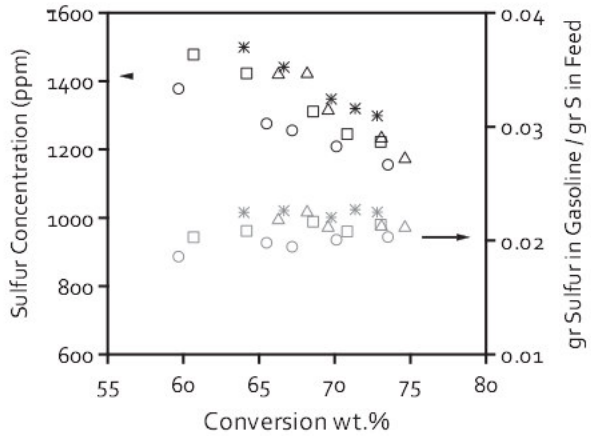

Fig. 5. Sulfur concentration in gasoline and sulfur in gasoline-to-sulfur in feed ratio for the catalyst and additive–catalyst mixtures. (*) Ecat, (○) Ecat + 10% additive, (□) Ecat + 25% additive, and (△) Ecat + 40% additive.

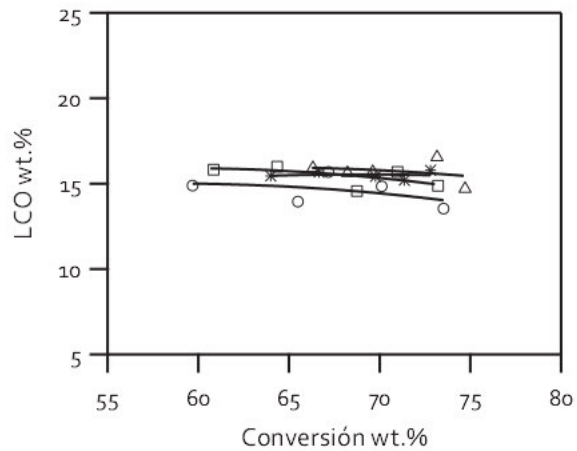

Fig. 6. LCO yield of catalyst and catalyst–additive mixtures. (*) Ecat, (○) Ecat + 10% additive, (□) Ecat + 25% additive, and (△) Ecat + 40% additive.

concentration of olefins increases as compared with the experiments without additive. This can be rationalized considering that at high concentration the additive promotes the alkylation of aromatic compounds, particularly the sulphur-containing ones, a reaction which is olefin demanding, and the hydrogenation reactions, which consume olefins and produce paraffins [9]. The concentration of aromatics in gasoline shows a slight decrease when the amount of additive increases, possibly as the consequence of selective adsorption on the Lewis sites of the additive, finally ending up in coke [17].

3.3. Sulfur compounds in gasoline

The concentration of sulfur due to the various sulfur compounds in gasoline is shown in Fig. 5, where it can be seen that it decreases as the conversion increases. Moreover, the concentration of sulfur in gasoline is lower with the catalyst–additive mixtures than with the catalyst alone in all the range of conversions, particularly with the 10 wt.% mixture. It is observed that increasing the amount of additive over this level does not produce further reductions in the concentration of sulfur. It can be seen in the same figure that the relationship between sulfur in gasoline and sulfur in the feedstock shows that approximately 2% of the sulfur in the feed ends in the gasoline cut, and that this amount is appreciably stable in the range of conversions studied. This relationship is slightly lower for the mixture of catalyst and 10 wt.% of additive, a fact which is in line with the results mentioned previously, considering that the gasoline yield is practically not affected by the incorporation of the additive.

The contributions to the concentration of sulfur from each of the individual sulfur compounds or families in gasoline at 70% conversion are also shown in Table 2. The reductions in sulfur concentrations can be observed in almost all the compounds or families in the range of gasoline. However, it is apparent that when the additive is present at 10 wt.%, the decrease of total sulfur concentration as compared to the catalyst alone is higher; specifically, at 70% of conversion, the concentration of sulfur in gasoline drops from 1339 to 1209 ppm (about 10% of sulfur reduction), while other amounts of additive produce lower reductions. These values of sulfur in gasoline are close to the values reported and expected taking into account that 2–5% of the sulfur in feed ends up in the gasoline range [2,17]. In the case of the C3-tiophene family, the concentration of sulfur increases when the additive is loaded at 25 and 40 wt.% This increase is consistent with the reaction mechanisms proposed for this type of materials [8], which admit that the additives promote the alkylation of thiophene and alkyl-thiophenes to produce other alkyl-thiophenes and alkyl-tetrahydrothiophenes, which would be more easily transformed by the FCC catalyst into hydrogen sulfide and sulfur-free hydrocarbons, or heavier sulfur compounds out of the gasoline range. In these cases (high additive loads), the concentration of sulfur in C3-thiophene family would increase because the remaining FCC catalyst mass is not enough to provide for active sites able to perform this last transformation of the sulphur containing molecules.

3.4. LCO

In all the cases (pure catalyst and additive–catalyst mixtures) the yields of LCO are essentially stable through the range of conversions obtained, as can be observed in Fig. 6. It can be seen that the yield of LCO is slightly decreased by the addition of 10 wt.% of additive, while the other proportions of additive essentially do not exert effects. Commercial additives have been reported in the literature as effectively reducing the amount of sulfur in the full range of gasoline and even in the light portion of the LCO cut [10]; however, the effect on LCO (sulfur concentration and yield) has not been reported.

3.4.1. Sulfur in LCO

The concentration of sulfur in LCO is shown in Table 2, where it can be seen that it decreases as a function of the proportion of additive and that the reduction in sulphur was observed in most of the families/compounds. This behavior is similar for all the families/compounds, and the maximum total sulfur reduction is about 5%, when the additive proportion is 40 wt.% At 10 wt.% of additive, which was the condition to achieve the maximum sulfur reduction in gasoline, the reduction in sulfur concentration in LCO is the lowest (approximately 2%). The higher difficulty in reducing the concentration of sulfur in LCO than in gasoline can be rationalized on the fact that sulfur compounds in that boiling range have a more aromatic, and consequently refractory, character. The concentration of sulfur in LCO at 70% of VGO conversion represents about 26 wt.% of the sulfur in feed, and seems to be in line with the value reported in the literature [2].

3.5. Reduction in the end boiling point of gasoline

The design and operation of the experimental setup used in this work allow for the sulfur and hydrocarbons simultaneous on-line analysis with the help of two detectors [14]. This is also useful in analyzing the effect of reducing the gasoline end boiling point, both

Table 3
Effect of gasoline end boiling point reduction (70 wt.% conversion) on gasoline and LCO yields and sulfur concentrations.

Gasoline end point	Ecat			Ecat + 10% additive			Ecat + 25% additive			Ecat + 40% additive		
	221 °C	196 °C	174 °C	221 °C	196 °C	174 °C	221 °C	196 °C	174 °C	221 °C	196 °C	174 °C
Gasoline yield	34.0	31.3	28.6	33.5	31.3	29.1	33.7	30.8	28.5	33.0	30.3	27.7
Yield reduction (%)		7.9	15.9	1.5	7.9	14.4	0.9	9.4	16.2	2.9	10.9	18.5
Total sulfur	1339	987	996	1209	871	865	1292	961	950	1325	1009	1000
% sulfur reduction		26.3	25.6	9.7	35.0	35.4	3.5	28.2	29.1	0.9	24.6	25.3
LCO yield	15.4	18.0	20.8	14.3	17.0	19.3	15.4	17.9	20.6	15.9	18.7	21.3
Yield increase (%)		17.3	35.3	−7.0	10.6	25.0	0.0	16.1	34.1	3.5	21.4	38.4
Total sulfur	35,383	30,932	34,722	34,722	30,478	27,010	34,781	30,145	26,571	33,607	29,830	26,306
% sulfur reduction		12.6	23.3	1.9	13.9	23.7	1.7	14.8	24.9	5.0	15.7	26.7

on gasoline and LCO yields and sulfur contents, in a very simple and accurate way. As it was mentioned in the introduction, another option to decrease the amount of sulfur in gasoline is to modify the final boiling point of the cut to lower values. This may have a double impact: first, directly reducing the concentration of sulfur in gasoline, which is due to compounds with the highest boiling points in the range, and additionally, it may improve the overall effect of the additive. It is obvious that this action will have a penalty on gasoline yield, where a loss will occur. However, it has to be taken into account that LCO yield will increase, with changes in the sulfur balance. Various options for the reduction of the gasoline end point are shown in Table 3, which were chosen to be coincident with n-decane and n-undecane boiling points only to ease calculations. By reducing the end point of the gasoline cut, the heaviest sulfur compounds (particularly C3-tiophene and BT) are now sent to the LCO cut, together with some hydrocarbons. The simple reduction of the gasoline end boiling point from 221 °C to 196 °C, even when the additive is not present, leads to a considerable reduction in the gasoline sulfur concentration (about 26%). Further reductions in the end point do not produce major changes in the concentration of sulfur and cause a high loss of gasoline yield, independently of the amount of additive present. The conditions of maximum sulfur reduction in gasoline with minimum yield penalty are achieved with the combination of 10 wt.% of additive and final boiling point of gasoline at 196 °C, producing a significant reduction (35%) in the concentration of sulfur in the cut, with a yield loss of approximately 8 wt.% The consequences of these exercises on the gasoline end boiling point are to increase LCO yield and decrease its sulfur concentration by means of a dilution effect on the middle distillate cut. The conditions of maximum sulfur reduction in gasoline with minimum yield penalty (10 wt.% additive, 8% gasoline yield loss) imply that LCO yield will increase about 10% and that the sulfur concentration in the cut will simultaneously reduce in about 14%.

4. Conclusions

The laboratory methodology and tools employed in this study made it possible to assess the effectiveness and impact on FCC yields of different concentrations of a commercial additive for sulfur control, added to a commercial catalyst. It was observed that the reduction of sulfur occurs differently in gasoline and LCO cuts, and that, depending on the goal of the refiner, it may be more efficient to use a low (10 wt.%) or high (40 wt.%) percentage of the additive. At 70% of VGO conversion, the lower additive content showed a greater efficiency in the reduction of sulfur in the gasoline boiling range, with some loss of LCO to LPG, while the higher concentrations of additive showed more efficiency to decrease sulfur in the LCO boiling range without changes in their yield, with little loss of GLP.

In cases where apart from using the additive there is a chance of reducing the final boiling point of gasoline, a fact that will cause a loss in its yield, the reduction of sulfur in gasoline as well as in LCO may become more attractive.

The CREC Riser Simulator reactor coupled to a GC with parallel FID and PFPD detectors showed neat advantages for the analysis of sulfur in FCC products.

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