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OŠTAR REŽIM RADA NA REFORMINGU U RAFINERIJI NAFTE RIJEKA SMANJUJE SADRŽAJ OKSIGENATA U MOTORNIM BENZINIMA

Sažetak

Nakon izmjene katalizatora na Platformingu 2 Rafinerija nafte Rijeka mogla je voditi proces u oštrom režimu. U pojedinim mjesecima 1999. i 2000. godine kad je način rada na Platformingu 2 rezultirao prosječnim IOB reformata iznad 99, sadržaj oksigenata u eurosuperu BMB 95 kretao se od 0 do 3,23 vol%. Blaži režim rada Platforminga 2 uvjetovao je pak dodavanje preko 7 vol% MTBE. Tijekom oba režima rada platforming procesa zadržana je zadovoljavajuća čistoća proizvedenog vodika za održavanje minimalnog parcijalnog tlaka na hidrodesulfurizaciji dizelskih goriva. Osim značajnih ušteda ostvarenih zbog visoke cijene MTBE smatramo to i doprinosom zaštiti okoliša zbog smanjene opasnosti od zagađenja izvora pitke vode.

Uvod

U svijetu su se oksigenati počeli dodavati u motorne benzine kasnih 70-ih godina radi povećanja oktanskog broja motornih benzina. Desetljeće kasnije dodavanje oksigenata vođeno je s ciljem da se smanji emisija ugljičnog monoksida. Specifikacija reformuliranih benzina polovicom devedesetih godina dopuštavala je sadržaj kisika oko 2 wt%.

Široka uporaba oksigenata u namješavanju motornih benzina dovela je do pozitivnih rezultata u poboljšanju kvalitete zraka. Zahtjevi tržišta za oksigenatima, u čemu je udio etera bio skoro 80%, počeli su rasti a samim tim i njihovi proizvodni kapaciteti. Ova se grana industrije činila vrlo perspektivnom u godinama stagnacije profita u naftnoj industriji.

Tada se dogodilo nekoliko ekoloških incidenata u najosjetljivijem području svijeta, Kaliforniji i na izvjesnu budućnost primjene oksigenata u namješavanju motornih benzina stavljena je ako ne točka a onda ozbiljni upitnik. Incidenti koji su okrenuli razvojni smjer bili su propuštanja podzemnih i nadzemnih spremnika, što je imalo rezultat onečišćenja pitke vode s preko 50 ppb MTBE. Ovaj oksigenat zapravo nije štetan za zdravlje ali njegova prisutnost u količini od svega 2 do 5 ppb čini okus pitke vode neprihvatljivim. Osim problema sa spremnicima u Kaliforniji su kritici MTBE doprinijele i prometne nezgode u kojima je došlo do prolijevanja benzina s MTBE-om, čime je onečišćeno 11 izvora pitke vode do razine 900 ppb. Guverneru Kalifornije je to bilo dovoljno da zabrani uporabu MTBE-a poslije 2002. godine, a i neke druge države kao New Jersey, Connecticut i Maine poduzimaju pravne mjere radi izbacivanja MTBE-a iz uporabe.

1. Gdje je Hrvatska i INA u priči o MTBE-u?

Prema važećim Internim normama INA N 02-001/98 i INA N 02-002/98 INA isporučuje olovne i bezolovne motorne benzine sa sljedećim dopuštenim sadržajem oksigenata:

- etanol, najviše 5% v/v,
- izopropil alkohol, najviše 10%v/v,
- metanol, najviše 3% v/v,
- eteri s 5 ili više C-atoma, najviše 15%v/v,
- butil-alkohol, sek-butil alkohol, viši monoalkoholi vrelišta ispod 215^{0}C, najviše 10%v/v.

U Rafineriji nafte Rijeka se od oksigenata koristi samo metiltercijerni butil eter, koji se počeo nabavljati 1995. godine. Testirale su se najefikasnije i najekonomičnije koncentracije i počele dodavati najprije u bezolovni motorni benzin 95 (5,49 %m/m) a minimalno (2,12 %m/m) u tadašnji olovni super 98 s 0,6 g olova po litri.

Osim za poboljšanje oktana, MTBE je bio i vrlo pogodna komponenta za namješavanje benzina, jer je kao visokooktanski spoj niskog vrelišta (55^{0}C) popravljao oktansku rupu na početku destilacijske krivulje motornih benzina. Kasnije je MTBE dodavan samo u motorne benzine bez olova BMB 95 i MB 98 sa 0.15 g/l.

U sljedećoj tablici prikazane su godišnje količine dodanog MTBE-a u motorne benzine proizvedene u Rafineriji nafte Rijeka u razdoblju od 1995. godine do I.-IX. 2000. godine.

Tablica 1: Utrošak MTBE u Rafineriji nafte Rijeka
Table 1: MTBE consumption at Rijeka Oil Refinery

	PROIZVEDENI MOTORNI BENZINI BMB 95 i MB 98/0.15 T/GOD UMG 95 AND MG 98/0 MOTOR GASOLINE PRODUCED T/YEAR	UTROŠAK MTBE, T/GOD MTBE CONSUMPTION T/YEAR
1995.	318 271	22 674
1996.	320 074	7 546
1997.	287 702	14 038
1998.	341 754	17 804
1999.	341 908	14 531
I.-IX. 2000.	363 078	3 532

Kao što je vidljivo iz tablice 1, ako zanemarimo prvu godinu uporabe MTBE-a kada je potrošena velika količina ovog oksigenata, trend je u sljedećim godinama od 1996. do 1998. rastući, a 1999. godine taj je trend zaustavljen i okrenut naniže.

2. Zamjena katalizatora na Platformingu 2

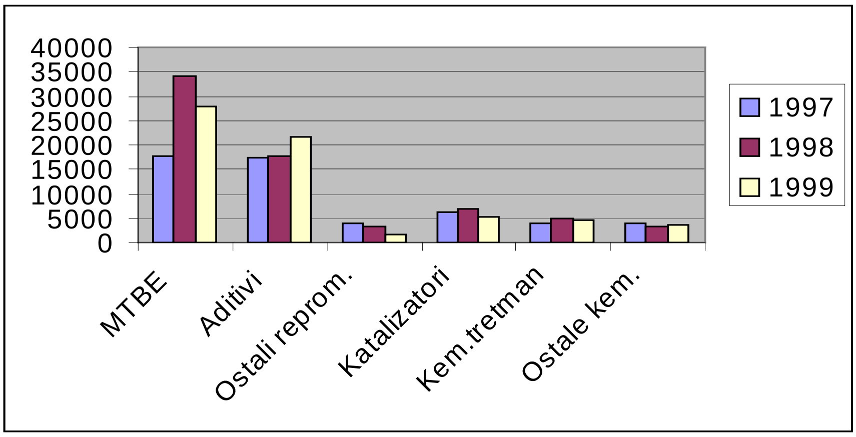
U studenom 1998. godine na Platformingu 2 je zamijenjen katalizator i puni efekti ove promjene vidljivi su tijekom 1999. godine u ukupno smanjenoj količini utrošenog MTBE. Godina 2000. nije pogodna za usporedbu jer je osim MTBE-a za povećanje oktana motornih benzina korišten i novi aditiv za poboljšanje oktanskog broja.

Tablica 2: Prikaz količina i vrijednosti utrošenog MTBE-a u RNR
Table 2: Presentation of volumes and values of MTBE consumed at ROR

	PROIZVEDENI MOTORNI BENZINI BMB 95 I MB 98/0.15, T/GOD UMG 95 AND MG 98/0 MOTOR GASOLINE PRODUCED T/YEAR	UTROŠAK MTBE, T/GOD MTBE CONSUMPTION T/YEAR	UTROŠAK MTBE, $/GOD MTBE CONSUMPTION $/YEAR
1997.	287 702	14 038	3 677 956
1998.	341 754	17 804	5 038 532
1999.	341 908	14 531	3 240 413

Efekt smanjenja potrošnje MTBE-a ne čini se tako značajan kada se prikažu samo naturalni pokazatelji. Kada se usporede apsolutni novčani iznosi plaćeni za MTBE ali i razina troškova nabave MTBE-a u odnosu na troškove drugih materijala - aditiva, katalizatora, kemikalija za kemijski tretman i drugog, dojam postaje potpuniji. Analizom troškova materijala tijekom 1997. i 1998. godine postaje vidljivo da je najveći trošak MTBE-a i nužno se nameće potreba smanjenja njegove potrošnje do potpune eliminacije optimizacijom procesa proizvodnje.

Slika 1: Troškovi materijala u 000 kn u razdoblju 1997.-1999. godine
Figure 1: Material costs in 000 Kn in the 1997-1999 period



MTBE, Additives, Other prod. mater, Catalysts, Chem. treat, Other chemicals

Kako je katalizator Platforminga 2 bio pri kraju svog 9 ciklusa, donijeta je odluka nabave novog katalizatora. Kao glavni ciljevi pred novi katalizator postavljeni su sljedeći zahtjevi:

– Maksimalni oktani - IOB 100.
– Čistoća vodika preko 70 vol%, maksimalni iscrpak vodika.
– Trajanje ciklusa 12 mjeseci.

Ovakve su performance zahtijevane od licencora katalizatora za čistu benzinsku frakciju s toppinga destilacijskih granica 82-185^{0}C, gustoće 0.750g/l, hidroobrađenu do standardnih sadržaja sumpora, dušika i metala. Uvjeti rada na Platformingu 2 s obzirom na mogućnosti i konfiguraciju opreme bili su:

– Tlak na visokotlačnom separatoru 22 bara.
– Maksimalne temperature u reaktorima 549^{0}C.
– Maksimalna količina plina u recirkulaciji 149500 Nm^3/h.
– Protok šarže 90 m^3/h.

Reaktori Platforminga 2 napunjeni su novim katalizatorom u studenom 1998. a pravi efekti različitog režima rada vidljivi su iz ovisnosti prosječnih oktanskih brojeva namiješanih motornih benzina i količine dodanog MTBE-a tijekom 1999. godine. Tekuća 2000. godina, kako je već spomenuto, jest godina u kojoj smo počeli koristiti novi aditiv za povećanje oktanskog broja, te nije moguće razmatrati međusobnu ovisnost samo prosječnih oktana i sadržaj oksigenata.

Uz grafički prikaz oktanskih vrijednosti platformata i dodane količine MTBE-a u motorne benzine treba napomenuti da je na prosječni oktanski broj platformata utjecao i oktanski broj platformata s Platforminga 1. Ipak, ovaj je utjecaj minimalan jer Platforming 1 nije bio u kontinuiranom radu tijekom 1999. godine. Njegov je kapacitet znatno manji -28 m^3/h a projektni oktani su za njegove radne uvjete IOB 97.

3. Efekti zamjene katalizatora i oštrijeg režima

Iz prikazane ovisnosti na slici 2 vidljivi su efekti visokog prosječnog oktanskog broja platformata na potrošnju MTBE u proizvodnji motornih benzina. Kada je prosječni IOB bio iznad 99, udio MTBE-a u benzinima padao je ispod 2 %v/v, dok je u mjesecima nižih prosječnih oktana potrošnja MTBE-a rasla i do 9 %v/v.

Slika 2: Prosječni oktanski broj i utrošak MTBE-a u 1999. godini
Figure 2: Average octane number and MTBE consumption in 1999

MTBE consumed RON (average)

IOB platformata oko 99 postizan je u mjesecima oštrog režima rada na Platformingu 2. Usporedbu procesnih parametara prikazat ćemo na primjeru sljedećih mjeseci.

Tablica 3: Prosječne vrijednosti procesnih parametara na platformingu 2
Table 3: Average Platforming 2 process parameter values

	1.1.2 IOB	WAIT, ^0C	tlak na VTS, bar PRESSURE AT HPS	prostorna brzina, LHSV, h^{-1} SPATIAL VELOCITY
Siječanj 1999.	93.60	508	22	0,95
Veljača 1999.	94.73	508	22	0,95
Ožujak 1999.*	94.49	500	25	1,29
Travanj 1999.	98.77	508	23	1,29
Svibanj 1999.	99.30	517	21	1,55
Lipanj 1999.	98.25	517	23	1,39
Srpanj 1999.	96.79	518	23	1,36
Kolovoz 1999.	97.15	519	22	1
Rujan 1999.	96.25	526	23	1,13
Listopad 1999.*	97.28	480	24	0,95
Studeni 1999.	98.80	506	21,5	1,13
Prosinac 1999.	99.20	507	22	1

*Regeneracije katalizatora/*catalyst regeneration

S obzirom na različite prostorne brzine ovisno o kapacitetu rada Platforminga 2 teško je usporediti oštrinu temperaturnih zahtjeva koji su bili nužni za postizanje zadanog oktanskog broja platformata. Ako se pak temperaturni zahtjev korigira s obzirom na prostornu brzinu odnosno kapacitet rada procesa, dobiva se podatak da je u mjesecima najboljih performanci u pogledu oktanskog broja, znači u četvrtom, petom, te jedanaestom i dvanaestom mjesecu 1999. godine, prosječna ulazna temperatura u reaktore iznosila oko 515^{0}C. Kako su to mjeseci neposredno nakon regeneracije katalizatora a startna temperatura za postizanje maksimalnih oktana je 508^{0}C, vidljivo je da je katalizator radio u oštrom režimu na početku svakog novog ciklusa.

Oštar režim rada u mjesecima kada je prosječni IOB bio oko 99 donio je značajne uštede na potrošnji MTBE-a. Očekivane negativne posljedice rada na visokim temperaturama bile bi svakako niža čistoća proizvedenog vodika, veća produkcija suhog plina i veća brzina stvaranja koksa na katalizatoru, odnosno skraćenje ciklusa. U tablici 4 prikazane su prosječne vrijednosti iscrpka ugljikovodika C1 i C2 i čistoće vodika u recirkulaciji.

Tablica 4: Oktanski broj platformata, iscrpak C1-C2, čistoća vodika
Table 4: Platformate octane number, yield c1-c2, hydrogen purity

	1.1.3 IOB	Čistoća proizvedenog vodika, % v/v Generated hydrogen purity	Iscrpak C1+C2 % m/m Yield
Siječanj 1999.	93.60	65	7,85
Veljača 1999.	94.73	65	7,80
Ožujak 1999.*	94.49	78	7,93
Travanj 1999.	98.77	72	7,20
Svibanj 1999.	99.30	70,5	6,7
Lipanj 1999.	98.25	68,5	7,22
Srpanj 1999.	96.79	67	7,80
Kolovoz 1999.	97.15	67	7,30
Rujan 1999.	96.25	67	7,30
Listopad 1999.*	97.28	78	8,20
Studeni 1999.	98.80	70	7,9
Prosinac 1999.	99.20	70	7,5

Prikazani podaci ne dokazuju očekivanu korelaciju. U mjesecima najboljeg prosječnog oktanskog broja čistoća vodika je bila relativno dobra, oko 70% v/v, dopuštajući normalan rad pogona za hidrodesulfurizaciju plinskih ulja ili blagi hidrokreking. S druge strane, ni produkcija najmanje vrijednog proizvoda platforminga - suhog plina nije bila značajno veća.

4. Zaključci

Korelacija koja je uočena u analizi rezultata rada platforminga su znatno bolji ukupni rezultati procesa nakon regeneracije katalizatora i snažna uvjetovanost rezultata stanjem katalizatora, odnosno danima rada u ciklusu katalizatora. Naknadna saznanja dobivena tijekom regeneracije katalizatora o razini koksa na katalizatorima potvrđuju pretpostavku da oštar režim rada dovodi do značajnog stvaranja koksa i ubrzane deaktivacije katalizatora.

Ipak, smanjeni troškovi dobiveni uštedom količine MTBE-a, te relativno konstantni iscrpci visokovrijednih proizvoda platforming procesa, platformata i ukapljenog naftnog plina, uz zadržavanje čistoće vodika na razini dostatnoj za desulfurizaciju plinskih ulja, dovode do zaključka da je oštar režim rada na platforminzima uz maksimiranje oktana a uz dvije regeneracije katalizatora na Platformingu 2 tijekom godine najprofitabilniji način rada.

HIGH SEVERITY OPERATION MODE ON RIJEKA OIL REFINERY REFORMER REDUCED OXYGENATES CONTENT IN MOTOR GASOLINE POOL

Abstract

After reloading the reformer catalyst on Platforming 2, Rijeka Refinery was able to run the unit in high severity mode. During the months in 1999 and 2000 when average RON of reformate was above 99, consumption of MTBE was kept in range 0 to 3,23 vol% in EUROSUPER gasoline grade. The less severe mode resulted in higher MTBE consumption, so that MTBE content in the same gasoline grade was reaching 7 vol%. During both modes hydrogen quality was preserved to keep required hydrogen partial pressure for hydrodesulphurisation of diesel fuels. By lowering MTBE content in Eurosuper gasoline we reduced production costs due to very high price of MTBE and also prevented possible polution of underground recources of drinking water.

Introduction

The world-wide addition of oxygenates into motor gasoline began in late 70s, in view of increasing the motor gasoline octane number. One decade later, the addition of oxygenates was performed with the purpose of reducing carbon monoxide emission. Specification of reformulated gasoline towards the middle of the 90s permitted oxygen content around 2 wt%.

Wide use of oxygenates in the blending of motor gasoline has lead to positive results in air quality improvement. Market requirements for oxygenates – in which ether share was nearly 80% - started increasing, along with their production capacities. This particular industrial branch seemed very prospecting in the years of oil industry profit stagnation. That is when several environmental accidents occurred in the most sensitive part of the world - California, and the bright future of oxygenate application in the blending of motor gasoline was if not stopped, than at least severely questioned.

Accident that made the turn in the development were leakages of both ground and underground reservoirs, resulting in potable water pollution with over 50 ppb of MTBE. The said oxygenate is not really health-hazardous, but its presence in the volume of only 2-5 ppb makes potable water taste unacceptable. Apart from the problems with reservoirs in California, those criticizing MTBE were also mentioning traffic accidents involving the spilling of MTBE-containing gasoline, thus polluting 11 potable water sources up to the level of 900 ppb. This was enough for the Governor of California to prohibit the use of MTBE beyond 2002, in which decision he was followed by some other states as well, such as New Yersey, Connecticut and Main, now also undertaking certain legal measures in view of banning MTBE use.

1. Where are Croatia and INA in the Story About MTBE?

According to the valid internal standards INA N 02-001/98 and INA N 02-008/98, INA is delivering leaded and unleaded motor gasoline with the following permissible oxygenate content:

- Ethanol, max. 5% v/v
- Isopropyl alcohol, max. 10% v/v
- Methanol, max 3% v/v
- Buthyl-alcohol, *sec* buthyl-alcohol, higher monoalcohols with boiling point below 215^{0}C, max 10%v/v.

The only oxygenate used at Rijeka Oil Refinery is the Methyltertiary Butyl Ether, the supply of which dates back to 1995. The most efficent and the most cost-effective concentrations were tested and then added first in unleaded motor gasoline 95 (5.49% m/m); and minimally (2.12% m/m) in the then leaded Super gasoline with 0.6 g of lead per litre.

Apart from octane improvement, MTBE was also a very favourable component for gasoline blending, because, as a high octane compound with a low boiling point (55^{0}C), it was covering the octane gap at the beginning of motor gasoline distillation curve. Later, MTBE was being added only to UMG 95 unleaded gasoline and MG 98 with 0.15 g/l.

The Table 1 presents annual volumes of MTBE added into motor gasoline produced at Rijeka Oil Refinery in the period from 1995 to 1 Sept., 2000. As may be seen from the Table, if we neglect the first year when MTBE was used, when a large volume of this oxygenate was consumed, the trend in the following years from 1996-1998 was increasing, while, in 1999, it was stopped and started moving downwards.

2. Catalyst replacement at Platforming 2

In November, 1998, catalyst was replaced at Platforming 2, and the results of this change could be noticed in 1999 in the reduced total volume of MTBE consumed. The year 2000 is not apt for comparison, because, except for MTBE, another new octane improving additive was used for the motor gasoline octane increase.

The effect of reducing MTBE consumption does not seem so significant when only natural indicators are shown. When we compare the absolute pecuniary amounts paid for MTBE, but also the MTBE purchase expenditure level with regard to expenditures associated with other materials – additives, catalysts, chemical treatment agents and other – the impression becomes more complete. Through the analysis of material costs in the course of 1997 and 1998, it becomes clear that the costs associated with MTBE are the highest and that there is a need to reduce its consumption down to its complete elimination through production process optimization.

Since the Platforming 2 catalyst was approaching the end of its 9th cycle, the decision was made to purchase a new catalyst. The new catalyst was faced with the following requirements:

- Maximal octanes – RON 100
- Hydrogen purity of over 70 vol%, maximum hydrogen yield
- Cycle duration 12 months

Such performances were required by the catalyst licensor for a pure gasoline fraction from the topping with distillation limits 85-185^{0}C, density 0.750 g/l, hydrotreated up to standard sulphur, nitrogen and metal content. Operating conditions at platforming 2, given the equipment possibilities and configuration, were as follows:

- Pressure at the high pressure separator 22 bar
- Maximum reactor temperature 549^{0}C
- Maximum gas volume in recirculation 149500 Nm^3/h
- Charge flow 90 m^3/h

The Platforming 2 reactors were filled with a new catalysts in November, 1998, while the real effects of the different operating regime are visible from the dependence of average octane numbers of the blended motor gasoline and the added MTBE volume in the course of 1999. The current year of 2000, as we have already mentioned, is the year in which we have started using a new octane improving additive, which is why it is not possible to consider the interdependence of only the average octanes and oxygenate content.

With the graphical presentation of the platformate octane values and the MTBE volume added into motor gasoline, we should point out that the average octane platformate number was influenced also by the octane number of platformate from Platforming 1. Still, the said impact is only minimal, since Platforming 1 was not operating continuously in the course of 1999. Its capacity is much lower -28 m^3/h, while the project octanes for its operating conditions are RON 7.

3. Catalyst replacement and high severity operation mode results

From the dependence shown in Figure 2, we may see the effects of the high average platformate octane number on MTBE consumption in motor gasoline production. When the average RON was above 99, MTBE share in gasoline went down below 2 %v/v, while in the months of lower average octanes, MTBE consumption was going up to as much as 9 %v/v.

Platformate RON of around 99 was achieved in the months of severe operating regime at Platforming 2. The comparison of the process parameters shall be shown on the example of the following months. Given the different space velocities depending on the Platforming 2 operating capacity, it is difficult to compare the severity of temperatural requirements necessary to achieve the desired platformate octane number. If the temperature requirement is corrected given the space velocity i.e. the process operating capacity, we come up with the information that in the months of the best performances when it comes to the octane number – i.e. fourth, fifth, eleventh and twelveth month of 1999, the average input temperature was around 515^{0}C. Since they are the months immediately following catalyst regeneration, while the starting temperature for achieving maximum octanes is 508^{0}C, it may be seen that the catalyst was operating under a severe regime at the beginning of each new cycle.

The severe operating regime in the months with the average RON of 99 has ensured considerable savings in MTBE consumption. The expected negative consequences of operating at high pressure would by all means be lower purity of the hydrogen generated, increased dry gas production and increased velocity of coke generation at the catalyst i.e. cycle shortening. Table 4 presents average values of C1 and C2 hydrocarbons yield, as well as hydrogen purity in recirculation. The data presented do not prove the expected correlation. In the months of the best average octane number, hydrogen purity was relatively good, around 70 %v/v, allowing for a normal operation of the gas oil hydrodesulphurization or mild hydrocracking plants. On the other hand, not even the production of the least valuable Platforming product – dry gas – was considerably higher.

4. Conclusions

The correlation spotted in analyzing the Platforming operation results are the much better total process results after catalyst regeneration and a powerful conditioning of the results by the catalyst condition i.e. days of operation in the catalyst cycle. Additional knowledge acquired during catalyst regeneration on coke level at catalysts confirms the assumption that severe operating regime brings to considerable coke generation and hasty catalyst deactivation.

Still, the reduced costs obtained through saving MTBE volumes, as well as relatively constant yields of the highly valuable Platforming process, platformate and liquid natural gas products – while maintaining hydrogen purity at the level sufficient for gas desulphurization -bring us to conclusion that the severe operating regime at the Platformings with octane maximization and with two catalyst regenerations at Platforming 2 in the course of the year constitute the most profitable mode of operation.

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