

# Simultaneous use of CO and naphthalene for the reduction of NO on potassium promoted copper catalyst supported on Ce/TiO<sub>2</sub>-SiO<sub>2</sub> and in the presence of oxygen

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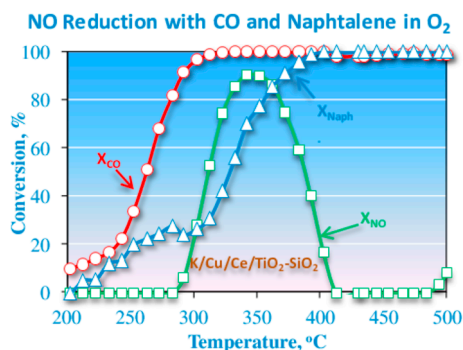
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## HIGHLIGHTS

- CO and naphthalene can be used simultaneously to reduce NO in the presence of oxygen.
- Full conversion of CO and naphthalene are at 330 °C and 400 °C, respectively.
- A maximum 90% conversion of NO is achieved at 342 °C.

## GRAPHICAL ABSTRACT



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## ABSTRACT

A set of Cu/Ce/TiO<sub>2</sub>-SiO<sub>2</sub> and K/Cu/Ce/TiO<sub>2</sub>-SiO<sub>2</sub> catalysts was used for the simultaneous use of CO and naphthalene as reducing agents for the reduction of NO in the presence of oxygen. Only 5% of TiO<sub>2</sub> greatly increase the activity of the catalysts. It was also found that the addition of potassium is able to tune down these oxidation reactions allowing the reduction of NO. The Cu/Ce/TiO<sub>2</sub>-SiO<sub>2</sub> catalyst achieves the full conversion of naphthalene at 302 °C, 96% of CO conversion at the same temperature, and negligible conversion of NO. On the other hand, the K/Cu/Ce/TiO<sub>2</sub>-SiO<sub>2</sub> catalyst reaches the full conversion of CO at 330 °C, 90% conversion of NO at 342 °C, and 70% conversion of naphthalene at the latter temperature.

## 1. Introduction

Incomplete fuels combustion, both natural and man-made, involves the generation of a great variety of polycyclic aromatic hydrocarbons

(PAHs). These compounds are well known for their negative effect on the human immune system because they are carcinogens, mutagens, and teratogens [1]. Among all PAHs, naphthalene is the smallest and less toxic molecule although it can cause the breakdown of red blood

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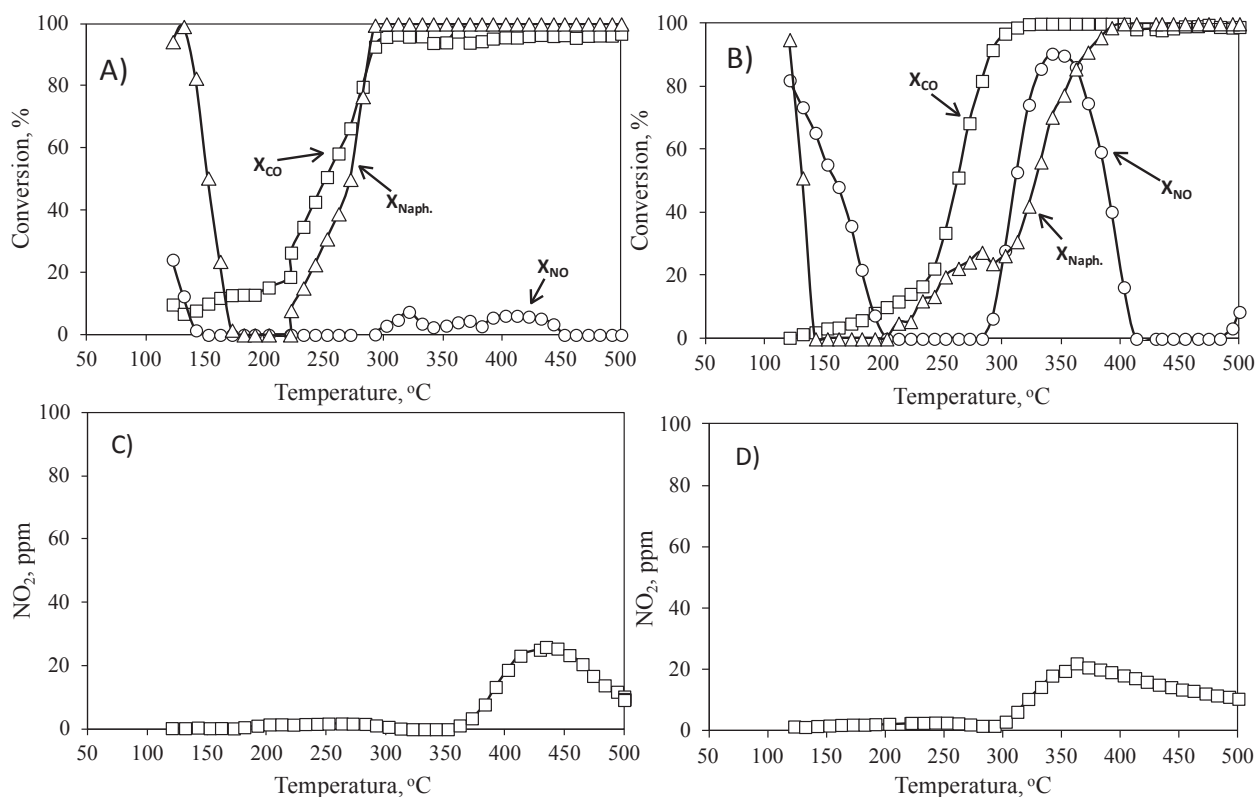
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**Fig. 1.** Activity of the (A) Cu/Ce/TiO<sub>2</sub>-SiO<sub>2</sub> and (B) K/Cu/Ce/TiO<sub>2</sub>-SiO<sub>2</sub> catalysts on the reduction of NO with CO and naphthalene. The presence of NO<sub>2</sub> in the reactor outlet is presented for the (C) Cu/Ce/TiO<sub>2</sub>-SiO<sub>2</sub> and (D) K/Cu/Ce/TiO<sub>2</sub>-SiO<sub>2</sub> catalysts. The conversion of CO ( $X_{CO}$ ), conversion of NO ( $X_{NO}$ ), and conversion of naphthalene ( $X_{Naph}$ ) are correspondingly labeled.

cells if ingested or inhaled in large quantities [2]. Nevertheless, the lower naphthalene toxicity is an advantage and it is frequently used as probe molecule representative of more complex PAHs.

Several methods have been used for naphthalene elimination being the most common the photocatalytic abatement [3–5] and catalytic oxidation using metal oxides and supported-metal oxides [6–17]. Among the latter, Pt-based catalysts are known to be highly active in the naphthalene catalytic oxidation [6–11]. Despite the high efficiency of noble metals, efforts are being put forth in finding more cost-effective materials. In fact and just to mention some examples, cerium oxide has demonstrated to be active in naphthalene oxidation [12–14,18,19], and copper supported on metal oxides as well [14,15,20]. Even copper supported on ceria has achieved high conversions of naphthalene oxidation [17].

Considering that the effluents from combustion processes consist of a pollutants mixture, other common compounds might be present when catalytically oxidizing naphthalene, such as CO and NO, two compounds whose presence in wood burning emissions is well documented [21]. In this study, a copper based catalyst supported on a Ce/TiO<sub>2</sub>-SiO<sub>2</sub> support is investigated on the simultaneous reduction of NO with CO and naphthalene in the presence of oxygen. The challenge on the latter reaction is to avoid the oxidation of CO and naphthalene on high oxygen concentrations and, therefore, eliminate the reducing agents of NO. Moreover, since the combustion of several energy sources, especially biomass, can lead to the detrimental accumulation of alkaline compounds on the catalyst surface, the addition of potassium to the catalyst is also studied.

## 2. Experimental

A mixture of 5 wt% TiO<sub>2</sub> (Sigma-Aldrich, < 25 nm particle size, 99.7% trace metal basis) and 95 wt% SiO<sub>2</sub> (Sigma-Aldrich, 5–15 nm particle size, 99.5% trace metal basis) was used as support. The

appropriate amounts of as-received TiO<sub>2</sub> and SiO<sub>2</sub> were weighed and mixed in a beaker for 1 h. An 8 wt% Ce (cerium nitrate hexahydrate, Sigma Aldrich) was wet-impregnated onto the TiO<sub>2</sub>-SiO<sub>2</sub> mixture and further calcined at 500 °C for 1 h. The obtained Ce/TiO<sub>2</sub>-SiO<sub>2</sub> was wet-impregnated with 3 wt% copper (copper nitrate hydrate, Sigma Aldrich) and calcined at 500 °C. Part of the previous sample was wet impregnated with 5 wt% potassium and again calcined at 500 °C. The catalysts activity (0.42 g) was determined in a 1 cm I.D. plug-flow quartz reactor prior reduction at 500 °C for 1 h in a flow of 10 cc/min of 5% H<sub>2</sub>/Ar. The gaseous mixture consisted of 500 ppm naphthalene, 500 ppm NO, 1% CO, 10% O<sub>2</sub>, balanced in He, flowing at 50 cc/min (GHSV = 10,000 h<sup>-1</sup>). A water bath was used to vaporize the solid naphthalene and the reacting gases through heated lines carried the generated vapor into the reactor. An ice bath was used after the reactor to prevent downstream condensation by naphthalene. The outlet gases from the reactor were analyzed by an infrared spectrometer (Shimadzu, IRPrestige21) equipped with a 2.4 m Pike gas cell. A chromatograph (Series 580, Gow-Mac) equipped with a 60/80 molecular sieve 5A (Supelco) was also used. DRIFTS analyses were performed on the same infrared spectrometer using a temperature-controlled stainless steel reactor (Pike) equipped with gas and refrigerant ports, and a KBr window. The crystal structure of the different catalysts was determined by X-ray diffraction on a Siemens D-5000 diffractometer using Cu K $\alpha$  radiation at a scan rate of 0.02° per minute.

Samples of each catalyst were additionally studied by temperature programmed reduction (TPR) experiments in a 5% H<sub>2</sub>/Ar stream using a 10 °C/min ramp. In these experiments, 0.1 g of catalyst was loaded into the reactor and oxidized in situ in a 20 cc/min stream of pure O<sub>2</sub> at 350 °C for 1 h. The surface area (BET) and pore size distribution of various prepared samples were obtained from N<sub>2</sub> adsorption isotherms using a Micromeritics 2010 adsorption apparatus.

### 3. Results

#### 3.1. Activity results

The activity results on the reduction of NO with CO in the presence of oxygen and the naphthalene simultaneous oxidation is shown in Fig. 1. For the Cu/Ce/TiO<sub>2</sub>-SiO<sub>2</sub> catalyst, the conversion of CO and naphthalene was similar at higher temperatures with full conversion of naphthalene at 302 °C and 96% conversion of CO at the same temperature. It can be observed in Fig. 1A that almost negligible conversion of NO was obtained with the Cu/Ce/TiO<sub>2</sub>-SiO<sub>2</sub> catalyst.

On the other hand, for the K/Cu/Ce/TiO<sub>2</sub>-SiO<sub>2</sub> catalysts, in Fig. 1B can be observed that full CO conversion is reached at 330 °C, whereas complete conversion of naphthalene is achieved at 400 °C. In the case of the NO conversion, a volcano-shaped curve is obtained with a maximum of 90% NO conversion at 342 °C. The naphthalene conversion for this latter temperature corresponded to 70%.

Both catalysts showed activity on the NO conversion, and naphthalene below 150 °C, but it rather corresponded to physical absorption of these compounds on the catalysts surface. Also, it should be noticed that NO<sub>2</sub> resulting from the NO oxidation, should not leave the reactor because of its reactivity into other harmful pollutants. As shown in Fig. 1C, small amounts of NO<sub>2</sub> are eluted out of the reactor above 300 °C when using the Cu/Ce/TiO<sub>2</sub>-SiO<sub>2</sub> catalyst, which is also at the temperature where full conversion of CO and naphthalene is reached. The maximum NO<sub>2</sub> concentration is observed at 430 °C. On the other hand, for the K/Cu/Ce/TiO<sub>2</sub>-SiO<sub>2</sub> catalyst, NO<sub>2</sub> appears above 350 °C and reaches a maximum at 360 °C, Fig. 1D. Nevertheless, the amount of NO<sub>2</sub> in the gaseous outlet is rather low with both catalysts. It should also be noticed that both catalysts led to full oxidation of CO and naphthalene with no other compounds present in the gaseous outlet.

Fig. 2 compares the effect of the presence of TiO<sub>2</sub> on the reduction of NO with CO, in the presence of oxygen using the catalyst containing potassium and copper. It is clearly observed that the activity is much higher when TiO<sub>2</sub> is present. The use of higher TiO<sub>2</sub> loadings did not lead to higher activities (not shown) and thus the 5% loading of TiO<sub>2</sub> was used in this work.

#### 3.2. X-ray diffraction

In order to study the catalysts crystalline phases, XRD analyses were performed on the calcined catalysts. Fig. 3 shows the SiO<sub>2</sub> diffraction with a broad peak centered at 2θ = 25°, which is assigned to amorphous silica. When mixed with TiO<sub>2</sub> and impregnated with CeO<sub>2</sub>, the Ce/TiO<sub>2</sub>-SiO<sub>2</sub> support retains the diffraction of the SiO<sub>2</sub> support, with small contributions of the TiO<sub>2</sub> phase at 25°, 38°, 48°, 55°, 63°, and 75°, corresponding to the (101), (112), (200), (211), (204) and (301) planes of the anatase phase (JCPDS #84–1286). A small shoulder at 29°

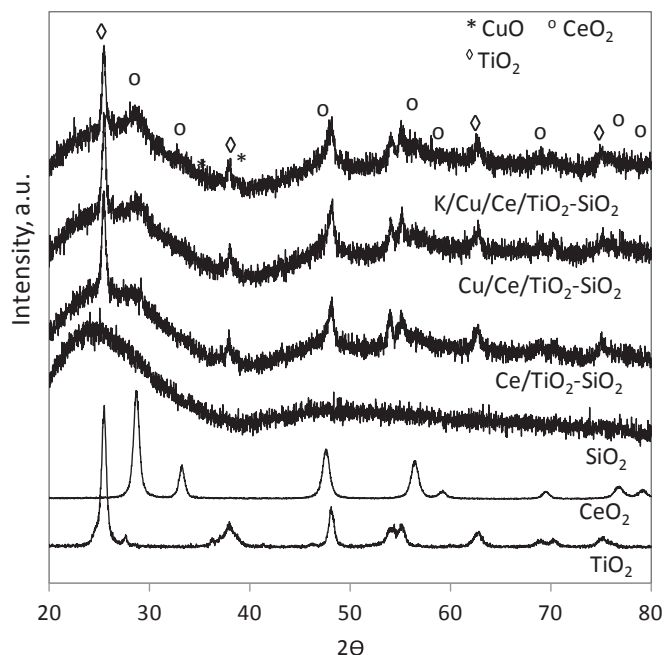


Fig. 3. X-ray diffraction spectra from the K/Cu/Ce/TiO<sub>2</sub>-SiO<sub>2</sub> and Cu/Ce/TiO<sub>2</sub>-SiO<sub>2</sub> catalyst. For comparison, the spectrum of the Ce/TiO<sub>2</sub>-SiO<sub>2</sub> support is included, as well as SiO<sub>2</sub>, CeO<sub>2</sub>, and TiO<sub>2</sub>.

evidences the existence of the (111) plane of the fluorite CeO<sub>2</sub> phase (JCPDS 34–0394). Regarding the spectra of the K/Cu/Ce/TiO<sub>2</sub>-SiO<sub>2</sub> and Cu/Ce/TiO<sub>2</sub>-SiO<sub>2</sub> catalysts, all diffractions are similar to the one of Ce/TiO<sub>2</sub>-SiO<sub>2</sub> support. The latter suggests either a high dispersion of the copper and potassium phases or their amorphous character.

#### 3.3. Temperature programmed reduction with hydrogen

In order to study the catalyst reducibility, temperature programmed reduction of the catalyst was carried out. Fig. 4 shows the reduction profile of the Cu/Ce/TiO<sub>2</sub>-SiO<sub>2</sub> catalyst, which shows the typical copper reduction with different degrees of aggregation: i) highly dispersed CuO species in close contact with the support (235 °C), ii) small and dispersed oxide clusters not forming crystallite yet (265 °C), and small crystallites of CuO (291 °C) [22,23]. The reduction at 389 °C would correspond to either the reduction of labile oxygen from the copper-ceria interface or the reduction of dispersed bulk CuO not detected by XRD.

After adding potassium, the K/Cu/Ce/TiO<sub>2</sub>-SiO<sub>2</sub> catalyst shows an abrupt shift of reduction profile up to higher temperatures. Rather than larger aggregations of copper, this behavior has been ascribed to the

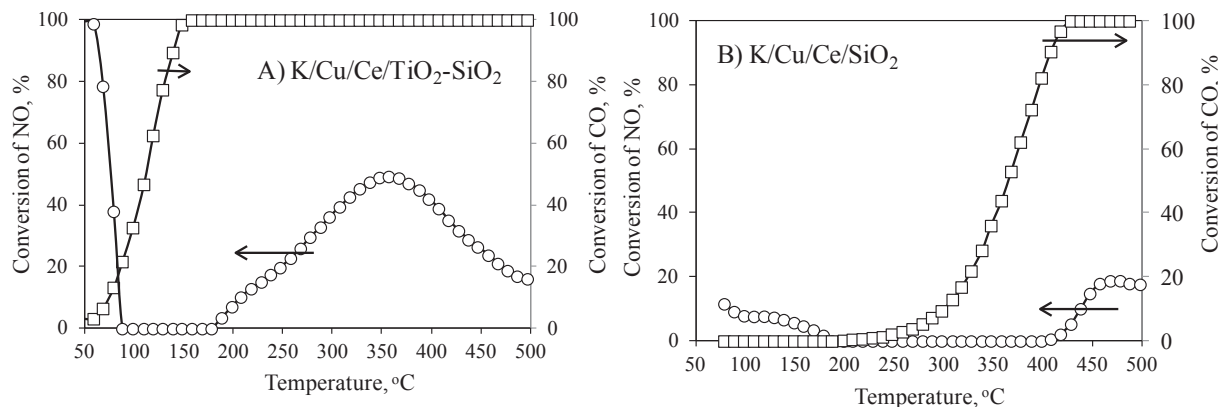


Fig. 2. Activity of the (A) K/Cu/Ce/TiO<sub>2</sub>-SiO<sub>2</sub> and (B) K/Cu/Ce/SiO<sub>2</sub> catalysts on the reduction of NO with CO in the presence of oxygen.

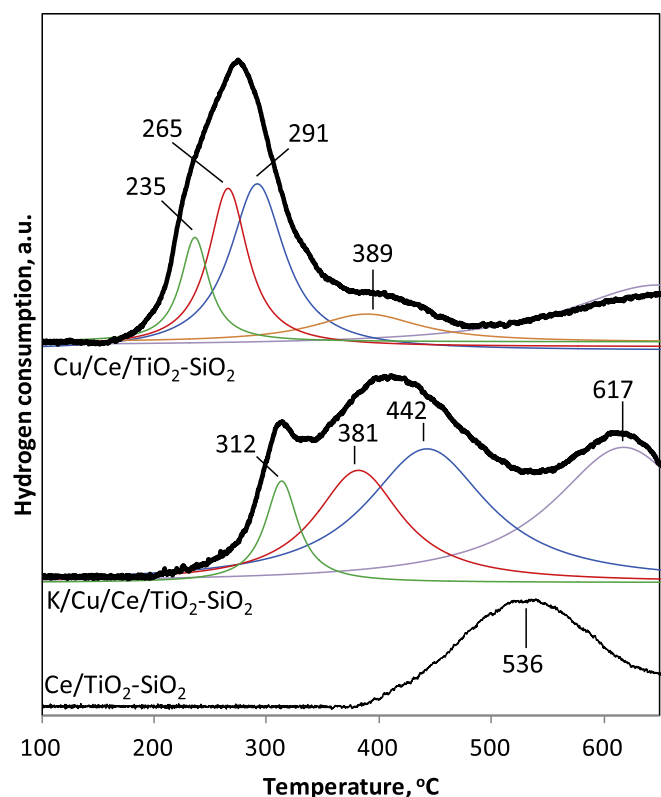


Fig. 4. Temperature programmed reduction with hydrogen of the K/Cu/Ce/TiO<sub>2</sub>-SiO<sub>2</sub> and Cu/Ce/TiO<sub>2</sub>-SiO<sub>2</sub> catalyst. For comparison, the Ce/TiO<sub>2</sub>-SiO<sub>2</sub> support is also included.

electronic effect of potassium over copper. The last interaction results in a less reducible copper phase and the shifting of the reduction peaks to higher temperatures [24]. It has been reported that simultaneous presence of copper and ceria leads to the formation of a CuO-CeO<sub>2</sub> interface, which contributes to hydrogen consumption during the reduction process [25]. In fact, the hydrogen consumption on the K/Cu/Ce/TiO<sub>2</sub>-SiO<sub>2</sub> catalyst is 60% higher (first three reduction peaks) than the Cu/Ce/TiO<sub>2</sub>-SiO<sub>2</sub> catalyst. This hydrogen overconsumption suggests that the presence of potassium not only affects the copper reduction, but it also increases the reduction of labile oxygen. Although the nature of such oxygen species is not clear at the moment, they could be associated with the interaction of copper with the ceria and/or titania phases. Again, due to the presence of potassium, the high-temperature reduction peak at 617 °C might correspond to the reduction of bulk copper particles, very small in size since their diffraction does not appear in the XRD results. The reduction broad peak at 536 °C shown by the Ce/TiO<sub>2</sub>-SiO<sub>2</sub> support has been associated with the reduction of labile surface oxygen species from ceria support [16,22,23]. The latter broad peak is also observed on the Cu/Ce/TiO<sub>2</sub>-SiO<sub>2</sub> catalyst, which shows that part of the CeO<sub>2</sub> surface is exposed after copper impregnation, what results in the reduction of labile surface oxygen from that phase.

### 3.4. Diffuse reflectance infrared Fourier transform spectroscopy (DRIFTS)

Given the good activity results obtained with the K/Cu/Ce/TiO<sub>2</sub>-SiO<sub>2</sub> catalyst, DRIFTS analyses were performed on this catalyst under different reaction conditions, Fig. 5A. The common band observed at 935 cm<sup>-1</sup> corresponds to infrared absorption from silanol groups from the SiO<sub>2</sub> support. A strong absorption band located at 1379 cm<sup>-1</sup> during the NO oxidation is assigned to ionic nitrates [26,27]. On the other hand, only a weak band at 1525 cm<sup>-1</sup> appears, which is associated with monodentate carbonate [28]. When the reduction of NO with CO was conducted, in the presence of oxygen, only a weak band is

observed at 1373 cm<sup>-1</sup> which is linked to ionic nitrate absorbed mostly on alkaline sites [29]. In the case of adding naphthalene to the latter reaction, i.e. the reduction of NO with CO and naphthalene (with oxygen), the ionic nitrate band was weaker than the case of the NO oxidation only, with a stronger contribution of monodentate carbonates at 1520 cm<sup>-1</sup>. The broadband in the range of 1700–1600 cm<sup>-1</sup> corresponds to different vibrations from surface water evolved during the naphthalene oxidation [30]. Similar absorption bands were observed on the Cu/Ce/TiO<sub>2</sub>-SiO<sub>2</sub> catalyst, Fig. 5B, except for the weaker intensity of all bands during the reduction of NO with CO and naphthalene, and the appearance of a weak broadband at 1500–1400 cm<sup>-1</sup> during the NO oxidation only and during the reduction of NO with CO. The latter broad absorption band is assigned to monodentate nitrates [31].

In order to elucidate the character of the active site under reaction conditions, the CO oxidation reaction was performed starting from room temperature. DRIFTS results shown in Fig. 6, shows the development of a band at 2094 cm<sup>-1</sup>, which has been ascribed to the absorption of CO on Cu<sup>+</sup> [32]. This band slowly decreases in intensity at higher temperatures, which is possibly due to the increase in the reaction rate making it not possible to observe the adsorption of CO at higher temperatures.

## 4. Discussion

It is clear that potassium addition changes the interaction of reacting species on the catalyst surface. The potassium-containing sample shows higher infrared absorption of carbonates and lower infrared absorption from ionic nitrates. These effects are reflected in the activity of the catalysts, where the potassium-containing sample shows lower activity on the oxidation of naphthalene than the potassium-free catalyst but higher NO reduction than the last sample. In other words, it seems that in the absence of potassium, copper sites are highly active oxidizing either CO or naphthalene, but not reducing NO. These results suggest that the presence of potassium changes the electronic copper environment [27], which is reflected in the TPR results: the potassium-free catalyst is able to be reduced to much lower temperatures than the sample containing potassium. It seems that the electropositive character of potassium leads to a higher interaction with surface oxygen atoms, in particular with those of copper, which ends up in a catalyst being more difficult to reduce. In terms of activity, the presence of potassium is able to tune the CO and naphthalene oxidation to provide species that are able to reduce NO, what does not occur in the potassium free catalyst where the direct CO and naphthalene oxidation proceeds without reduction of NO. It seems that the high activity on the oxidation of both CO and naphthalene is associated to redox cycle of Cu<sup>+2</sup>/Cu<sup>+</sup>. The latter might be favored by the presence of ceria due to the simultaneous presence of redox Cu<sup>+2</sup>/Cu<sup>+</sup> and Ce<sup>+3</sup>/Ce<sup>+4</sup> pair [33]. The highly dispersed copper on these catalysts would contribute to the readily redox cycles of this species which in turn favors the NO reduction and also the reactions of CO and naphthalene. In summary, the addition of potassium to a highly dispersed copper catalyst is able to tune down the oxidation reactions of CO and naphthalene, which in turn are used as reducing agents of NO in the presence of oxygen.

On the other hand, in the absence of TiO<sub>2</sub> the activity in the reduction of NO is rather poor compared to the case of adding TiO<sub>2</sub>. It has been reported that the ability of oxygen storage and transfer from TiO<sub>2</sub> is greatly increased by CeO<sub>2</sub> [34], as well as the generation of active oxygen species [35]. These effects would contribute to the improvement in the catalytic reduction of NO with CO and the increase in hydrogen consumption observed in the TPR experiments, which requires only small amounts of TiO<sub>2</sub> to occur.

Regarding the BET surface area, the Ce/TiO<sub>2</sub>-SiO<sub>2</sub> support had 324 m<sup>2</sup>/g after calcination at 500 °C (pore volume of 3.4 × 10<sup>-1</sup> cm<sup>3</sup>/g). After copper impregnation the Cu/Ce/TiO<sub>2</sub>-SiO<sub>2</sub> catalyst had an area of 251 m<sup>2</sup>/g after calcination at 500 °C (pore volume of 3.0 × 10<sup>-1</sup> cm<sup>3</sup>/g). On the other hand, the impregnation of potassium

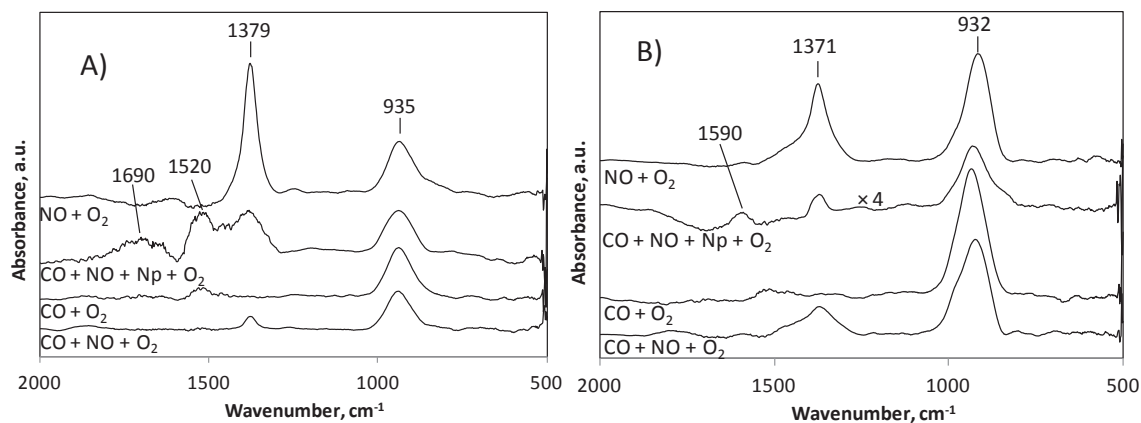


Fig. 5. DRIFTS results on the (A) K/Cu/Ce/TiO<sub>2</sub>-SiO<sub>2</sub> and (B) Cu/Ce/TiO<sub>2</sub>-SiO<sub>2</sub> catalysts under reaction conditions. From bottom to top: reduction of NO with CO in the presence of oxygen, CO oxidation, reduction of NO with CO and naphthalene (Np) in the presence of oxygen, and NO oxidation.

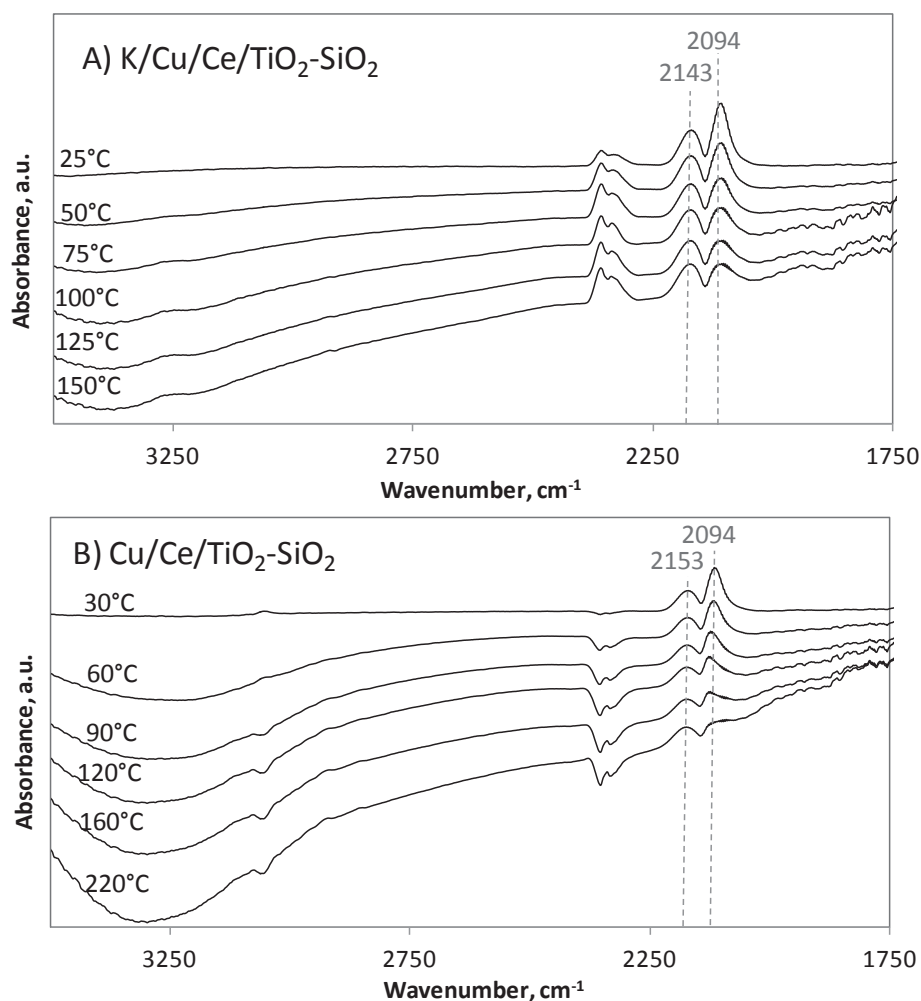


Fig. 6. DRIFTS results during the CO oxidation reaction at different temperatures on the A) K/Cu/Ce/TiO<sub>2</sub>-SiO<sub>2</sub> and B) Cu/Ce/TiO<sub>2</sub>-SiO<sub>2</sub> catalysts.

led to an abrupt decrease of the area of the calcined K/Cu/Ce/TiO<sub>2</sub>-SiO<sub>2</sub> catalyst with 44 m<sup>2</sup>/g (pore volume of 1.1 × 10<sup>-1</sup> cm<sup>3</sup>/g). The latter decrease in the area and pore volume has been assigned in previous work to the occlusion of small pores by potassium, which does not occur with the impregnation of copper [27,36]. In fact, the K/Cu/Ce/TiO<sub>2</sub>-SiO<sub>2</sub> catalyst is able to sustain a high activity being able to fully convert CO and naphthalene, Fig. 1B.

## 5. Conclusion

The simultaneous reduction of NO with CO and naphthalene was studied on a Cu/Ce/TiO<sub>2</sub>-SiO<sub>2</sub> and K/Cu/Ce/TiO<sub>2</sub>-SiO<sub>2</sub> catalysts. It was found that the highly dispersed copper phase is active oxidizing CO and naphthalene. The addition of potassium suppressed the oxidation reactions, specifically naphthalene oxidation, which allows CO and naphthalene to be used as reducing agents of NO. More important, it was found that only a small amount of TiO<sub>2</sub> is able to induce a great



change in the catalytic activity of a low-active copper catalyst.

The Cu/Ce/TiO<sub>2</sub>-SiO<sub>2</sub> catalyst is able to achieve full naphthalene conversion at 302 °C, 96% of CO conversion at same temperature, and negligible conversion of NO. On the other hand, the K/Cu/Ce/TiO<sub>2</sub>-SiO<sub>2</sub> catalyst reaches the full conversion of CO at 330 °C, 90% conversion of NO at 342 °C, and 70% conversion of naphthalene at the latter temperature. The use of potassium acts effectively to restrain the oxidation reactions to favor the NO reduction, which ends up favoring the elimination of all three pollutants.

## Acknowledgements

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