Article

Emissions of Criteria and Non-Criteria Pollutants by a Flex-Fuel Motorcycle

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While criteria pollutants have established emission limits for motorcycles in Brazil, aldehydes limits have not been established and conclusive studies have not been found in the existing literature, despite the growing number of motorcycles using flex fuel in Brazil. This work presents results for the emissions of criteria pollutants, such as carbon monoxide, hydrocarbons and nitrogen oxides, acetaldehyde and formaldehyde. The motorcycle was tested with 22, 61, and 100% of ethanol in gasoline blends. Fourier transform infrared (FTIR) and high performance liquid chromatography (HPLC) were used to determine the pre- and post-catalytic converter emissions. Aldehyde emissions directly increased with the ethanol content in the fuel blend. The tailpipe aldehyde emissions for 22, 61, and 100% of ethanol in gasoline were 3.9, 8.5, and 38.8 mg km^{-1}, respectively. These results demonstrated that aldehyde emissions in motorcycles are not negligible, and higher emissions are observed during the cold phase of the engine.

Keywords: vehicular, emission, aldehyde, motorcycle, flex fuel

Introduction

The internal combustion engines will remain, in the coming years, the main propulsion system for vehicles. Thus, the impact on the environment by emissions from mobile sources assumes great importance, mainly in urban centers.[1-3]

In Brazil, concern over vehicular emissions is not new. In 1986, the Control Program of Air Pollution from Motor Vehicles (PROCONVE) was created, and PROCONVE imposed a schedule to gradually reduce pollutant emissions for light and heavy vehicles.[4,5] With regard to motorcycles, legislation for emission control is newer, and in 2002, the Control Program of the Air Pollution by Motorcycles and Similar Vehicles (PROMOT) was created in Brazil. In countries with a tradition in the control of vehicle emissions such as the United States and European countries, legislation was established in the early 1960s and 1970s, respectively,[6,7] and the establishment of limits for motorcycle emissions has been established since 1990,[8] with a marked restriction from 1999.[9] However, the Brazilian legislation basically considered compounds legislated by the European Union.

The emission of aldehydes is relevant to the Brazilian context,[10,11] being legislated for vehicles in terms of total emissions since 1992.[12] The basis for this control was due to the Brazilian automotive energy matrix, where fuel differs from other countries by the addition of ethanol to gasoline and its use in the hydrated form. The use of ethanol has many advantages such as a renewable fuel, generation of less carbon monoxide, hydrocarbons and particulates, and higher octane numbers compared to gasoline, which leads to an increase in the engine compression ratio, promoting greater efficiency and engine power. Disadvantages stand out because of its lower calorific value and the increased emissions of carbonyl compounds, especially aldehydes.[13,14]

Because of the relationship between aldehyde emissions and the use of ethanol as a fuel,[10,15-17] emission limits have been implemented for these pollutants, as aldehydes are the result of partial oxidation of alcohols during the combustion process.[18-20]

These compounds cause damage and irritation to the respiratory tract and eyes and are classified as carcinogenic (formaldehyde) and potentially carcinogenic (acetaldehyde).[21-23] Due to their chemical reactivity, these compounds can participate in various reactions in the troposphere, forming secondary pollutants such as

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ozone, which, besides being a photochemical oxidant, is a greenhouse gas.[21,22-24]

Another aspect that has been discussed in the literature refers to the criteria and non-criteria emissions according to the ethanol content and, more recently, the concern about the cold phase emissions.[25,26] In the initial minutes, the emissions of most pollutants are known to be greater because the beginning of engine operation is associated with less efficient burning.[27,28] In addition, the catalyst is cold and therefore less effective. In this period, it is also common to observe significant emissions of ethanol and unconverted hydrocarbons, in addition to criteria pollutants.[29] Overall, this scenario commonly occurs in short paths and becomes worrisome in the context of the medium and large cities.

In Brazil, with the implementation of the Euro III limits in 2009, motorcycles need to be equipped with a three-way catalytic converter (TWC). In the same year, the first flex-fuel motorcycle was created. In 2010, Brazil occupied the fifth position in motorcycle production globally, trailing only the Asian countries. In 2009/2010, motorcycles constituted 25% of Brazil's total fleet.[30] The attractive price, financing facilities, and an inefficient public transport system in a favorable economic environment have boosted the market. According to ABRACICLO,[30] in 2011, more than two million units were sold. Compared to the automobile market, one motorcycle is sold for every 1.3 cars sold. In the following years, 1.5 and 2.0 million motorcycles were sold *per* year, confirming the importance of the motorcycle fleet in Brazil.

Some articles about aldehyde emissions are found in the literature,[31-35] but virtually there are no articles about carbonyl emissions during the cold phase or about the action of the catalyst under these conditions.

This work determines the emissions of criteria pollutants (carbon monoxide, carbon dioxide, total hydrocarbons and nitrogen oxides) and non-criteria pollutants (aldehydes) from a flex fuel motorcycle in the cold phase and the steady state by evaluating the role of the catalyst in both periods. The NBR 12026[36] regulation was followed, based on the CARB procedures.[37,38] For the determination of aldehydes, high performance liquid chromatography (HPLC) with UV detection and Fourier transform infrared (FTIR) spectroscopy were used to follow the aldehyde emission behavior and the influence of the catalyst. The tests were conducted on a chassis dynamometer in accordance with the European rules.

Experimental

A motorcycle with a 150 cm³ engine, representative of the Brazilian fleet (manufactured in 2011, standard Euro III,

with three-way catalyst (TWC), electronic fuel injection, flex fuel and 135 kg of inertia), was used.

Gasoline (78%) and anhydrous ethanol (22%) were used on a volume basis, called emission standard gasoline (E22), hydrous ethanol fuel (E100) and an intermediate mixture, called E61, containing both standard gasoline (E22) and E100 in the proportion of 50% by volume of the two fuels.

Brazil adopted the standard chassis dynamometer bench testing with the driving cycle from European legislation 97/24/EC,[9] which allows simulation of load changes during emission testing. The predetermined path can be divided into one or two stages, according to the motor volumetric capacity. Motorcycles up to 150 cm³ perform just one phase, called the urban cycle.

The test includes approximately 6.05 km in the urban cycle. The resistive power applied to the motorcycle by the dynamometer (AVL Zöllner AN 40770, 648 mm, 100 kW) along the way was determined by reference to the mass, adding up to 75 kg standard weight for the driver. The cooling of the motorcycle engine was performed by a fan positioned in front of the vehicle (AVL Zollner AN 40770, 52,000 m³ h⁻¹) at a speed directly proportional to the motorcycle velocity. The temperature of the testing room remained between 20 and 30 °C, and the average temperature value for the tests was 24 ºC. The temperature of the lubricating oil was maintained at ± 2 °C to room temperature before beginning the test.[9]

During the test, the emissions were diluted with ambient air and collected in 90 L Tedlar bags by a constant volume sampler (CVS Horiba 7200S) (Figure 1).

The unburned hydrocarbons were determined by a flame ionization detector (FID) analyzer (model FIA-720, 0-1000 ppmC), FID for CH_4 (model GFA720, 0-50 ppm), CO and CO_2 by non-dispersive infrared (NDIR) analyzers (model AIA-721A, 0-5000 ppm, and AIA-722, 0-5 vol%, respectively) and NO_x by a chemiluminescence analyzer (model CLA-720A, 0-300 ppm), all from the Horiba MEXA 7200 bench.

Aldehydes were determined following ABNT NBR 12026 methodology,[36] by the reaction with 2,4-dinitrophenylhydrazine (DNPH) in 25-mL impingers at a constant flow of 2.0 mL min⁻¹ of the diluted exhaust gas during the entire test (1200 s). Samples were analyzed immediately after the tests. For chemical analysis, an Agilent 1200 HPLC was used with a reverse phase column Agilent ZORBAX ODS C18 column (4.6 × 250 mm × 5 μm) operating with 70% acetonitrile in water and UV detection at 365 nm.

Supelco 47651-U standard solution was used at 0.210 to 5.250 mg L⁻¹ for formaldehyde and at 0.153 to 3.828 mg L⁻¹ for acetaldehyde. Five replications were analyzed with
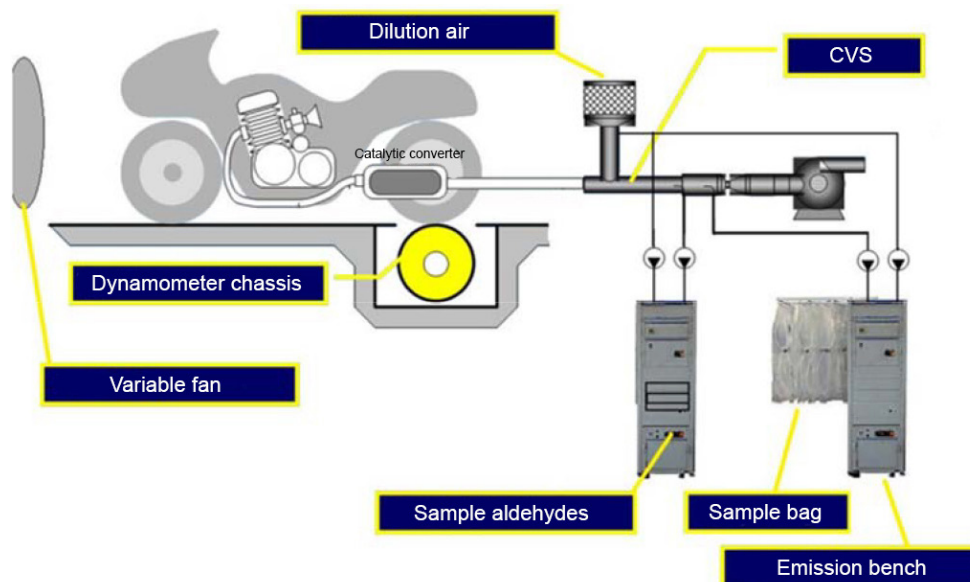
Figure 1. Basic configuration of the motorcycle test facility.

deviations below 3% and a determination coefficient better than 0.99.

Additional tests were performed using FTIR (AVL SESAM4), a technique that allows an on-line evaluation of compounds difficult to detect with a wide application for non-criteria compounds. The device has adjustments (methods) that optimize its operation in accordance with the tested fuel and was operated according to the methods for gasoline (E22 and E61) and ethanol (E100). The exhaust system of the motorcycle was instrumented for the collection of raw emissions before the catalytic converter (pre-cat) and after (tailpipe) at a frequency of 1 Hz.

To provide a statistical analysis and data assessment, three tests were conducted in each condition.

Results and Discussion

Vehicle emissions are known to depend on parameters such as ethanol content, the air/fuel ratio, engine operating conditions, and type of vehicle, among others.[39] In addition, most studies have been devoted to vehicular emission from ethanol-gasoline blends.[40] There are few studies related to the emissions from flex-fuel motorcycles,[15,18,32] and among these studies, there is little discussion of cold phase emissions.

Criteria pollutants emissions

Figures 2 and 3 depict the trend of criteria emissions (CO, NO_x, and THC-total hydrocarbons), measured using the FTIR technique, before and after the catalyst, allowing for the identification of emissions in the cold phase (transient region). To support the data, the temperature profile over time is present in Figure 4 because it contributes to a better understanding of the transient region. Note that the steady state is reached after 200 s. Furthermore, increasing the ethanol content reduces the operating temperature of the system.

Figure 2 shows that the CO and THC emissions are highest before the catalyst during the first 200 s (cold phase), justified by the incomplete combustion. In this stage, NO_x emissions are also observed to be lower in that same time frame because the lowest engine temperatures reduce the NO_x thermal pathway.

After the catalyst, the CO and THC emissions are also higher in the initial 200 s, explained by the low efficiency of the catalyst, which needs a minimum temperature to start the conversion reactions. For NO_x, few changes in emissions are observed, compared to the emissions during the steady state (after the initial 200 s) because at the beginning of the operation, emissions arriving in the catalyst are lower, and the catalyst is cold with a lower conversion rate.

To understand the effect of the ethanol content on the emissions, the results are presented before (Figure 5a) and after (Figure 5b) the catalyst during the two periods of analysis (before and after 200 s).

Regarding the results before the catalyst in the steady state (Figure 5a after 200 s), the criteria emissions follow the expected trend, namely, a reduction with increased ethanol content, in a less extent for CO using E100. The results for the steady state after the catalyst (Figure 5b after 200 s) are lower than the Euro III emissions limits (2.0; 0.8 and 0.15 g km^{-1} for CO, THC and NO_x,
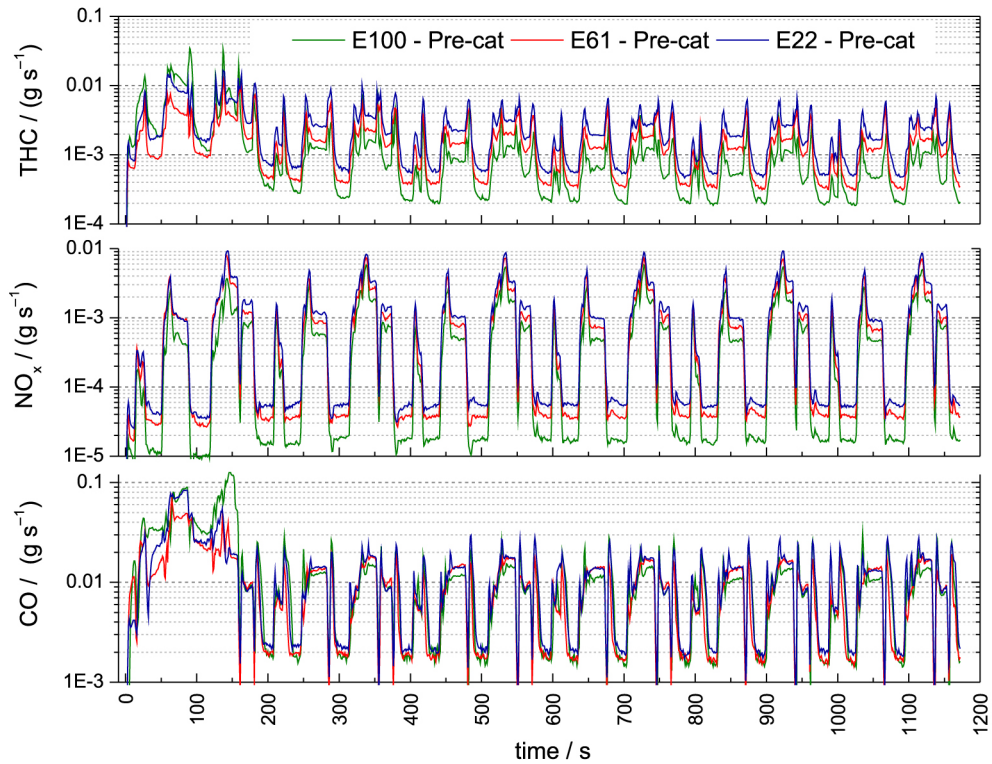
Figure 2. CO, THC (total hydrocarbons) and NO$_x$ emissions over time before the three-way catalytic converter (TWC).
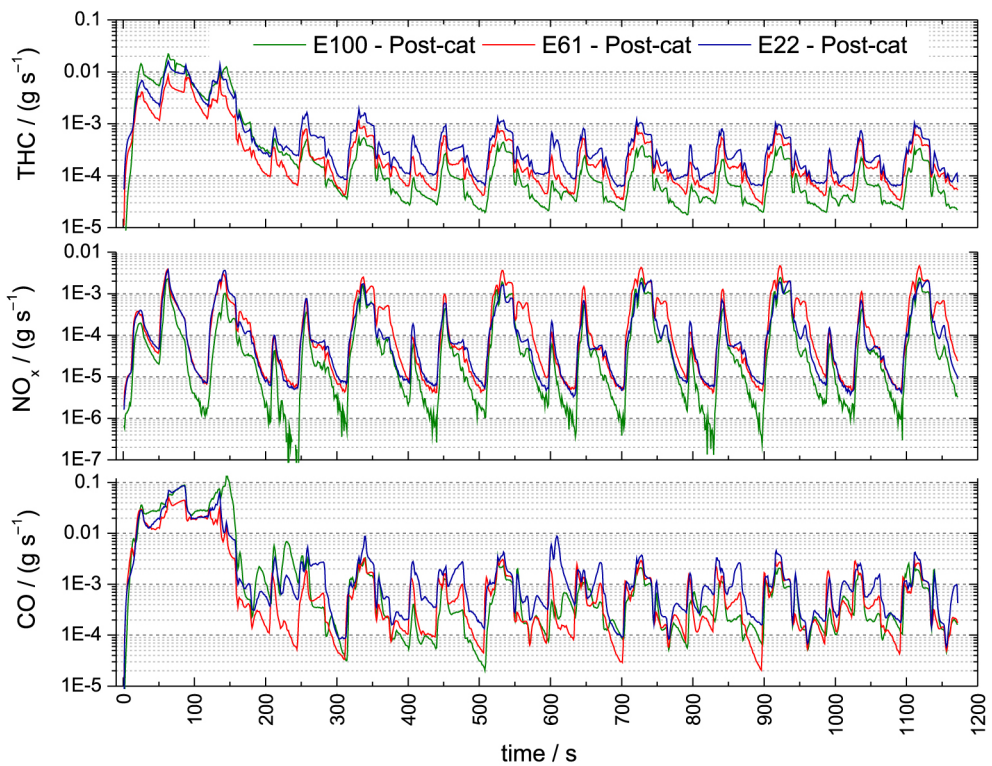

Figure 3. CO, THC (total hydrocarbons) and NO$_x$ emissions over time after the three-way catalytic converter (TWC).

respectively), regardless of the fuel used. Figure 5b clearly indicate the efficiency of the catalyst after the cold phase.

With regard to the cold phase before and after the catalyst, there is a clear correlation between the ethanol content and emissions. In this step, the engine components
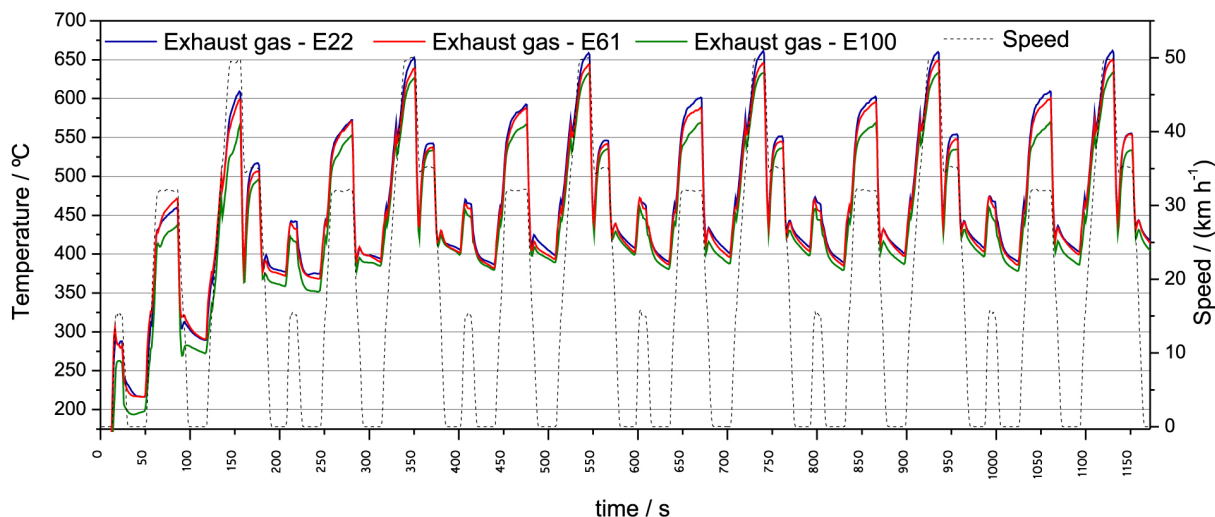
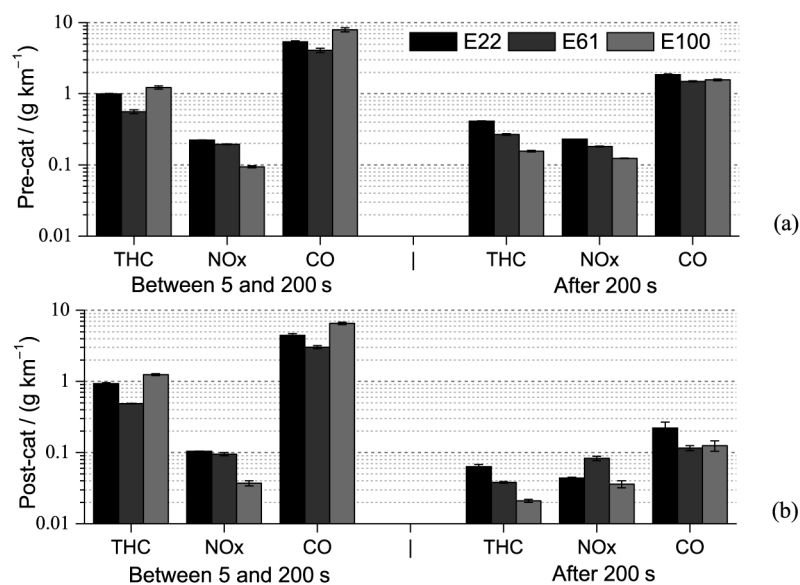**Figure 4**. Temperature profile during the experiment.

Figure 5. Average values for criteria emissions (a) before and (b) after the TWC, for cold phase and steady state.

are at ambient temperature. This period may also be defined as the time required for the engine to operate without suffering performance variations.

However, it is worth mentioning the issue of NO_x for E100. According to the literature,[41] when hydrated ethanol is used (E100), such as in Brazil, NO_x emissions decrease because water absorbs heat during vaporization, reducing the temperature peaks and hence the NO_x emissions.

A global analysis of these results after the catalyst for NO_x also indicates some differences that are mainly related to E61. This behavior can be attributed to the mapping criteria adopted by the fuel injection system manufacturer and the lower air/fuel ratio control for motorcycles.[42] The lower THC and CO emissions for E61 and the highest NO_x value after the catalyst can be explained by the

unforeseen change in the air/fuel ratio during the tests with E61. An analysis of the mean lambda value indicates values of 1.052, 1.104 and 1.065 for tests using E22, E61 and E100, respectively, and shows E61 tests with a more oxidizing medium, favoring oxidation reactions (increasing the conversion of CO and THC) instead of the reduction (decreasing NO_x conversion).

The catalyst conversions during the cold phase are very low (Figures 2 and 3), and they present null values for THC and values as low as 17% for CO and 51% for NO_x. The catalyst performance after 200 s shows good efficiency in most cases. With the use of E22, an average conversion of 85, 88 and 81% was observed for THC, CO and NO_x, respectively. With the use of E100, the average values are close to the above values: 87, 92, and 71% for THC, CO

and NO_x, respectively. Finally, for E61, 86, 92 and 54% are obtained for THC, CO and NO_x, respectively. Again, there is a low conversion of NO_x for E61, assigned to a higher air/fuel ratio, as discussed above.

As discussed in the literature, the catalytic efficiency values for motorcycles in most cases are lower than the catalytic efficiency value for automobiles that have required efficiency values above 90% for the criteria pollutants[43] and are assigned to the lower air/fuel ratio control for motorcycles.[42] Table 1 shows the average values of the criteria emissions by CVS analysis. With the exception of methane using A22 and E61, the hypothesis of no significant differences among the compounds is set aside. THC emissions are significantly different between the fuels; they are higher for E100 because the FID detector also measures the unburned ethanol as THC.

Non-criteria pollutants emissions

For the non-criteria emissions, emphasis is given to the aldehydes, specifically acetaldehyde and formaldehyde, as shown in Figure 6, before and after the catalyst for each of the fuels used. In the cold phase, higher formaldehyde and acetaldehyde emissions, especially with E100, are observed. The emission stabilization occurs approximately 3 and 6 min for acetaldehyde and formaldehyde, respectively.

During the cold phase, the unconverted hydrocarbons are partially oxidized, generating OHCs (oxygenated hydrocarbons), and intermediate species, such as aldehydes (formaldehyde and acetaldehyde), form during low temperature reactions. Specifically, formaldehyde is generated by the decomposition of alkyl peroxides formed at low temperatures and thus occurs primarily in the early stages of combustion, reaching a maximum value at approximately 1100 K when high temperature combustion starts, and formaldehyde tends to be consumed thereafter.[44]

The formaldehyde formation process on motorcycles using ethanol shows a lack of information, with reports presenting discordant values about mixtures with a low percentage of ethanol in gasoline.[45] Research on vehicles using blends up to 85% ethanol disclose increased

Table 1. Results for the criteria pollutant emissions

Fuel	Result	THC / (g km^{-1})	CO / (g km^{-1})	NO_x / (g km^{-1})	CO_2 / (g km^{-1})	CH_4 / (g km^{-1})	NMHC / (g km^{-1})
E22	average	0.182	0.978	0.068	50.969	0.010	0.172
	SD	0.006	0.093	0.003	0.218	0.001	0.005
E61	average	0.126	0.596	0.107	50.364	0.009	0.117
	SD	0.008	0.042	0.004	0.226	0.001	0.008
E100	average	0.246	1.198	0.050	49.262	0.020	0.227
	SD	0.006	0.073	0.004	0.252	0.002	0.006

THC: total hydrocarbons; SD: standard deviation; NMHC: non-methane hydrocarbons.
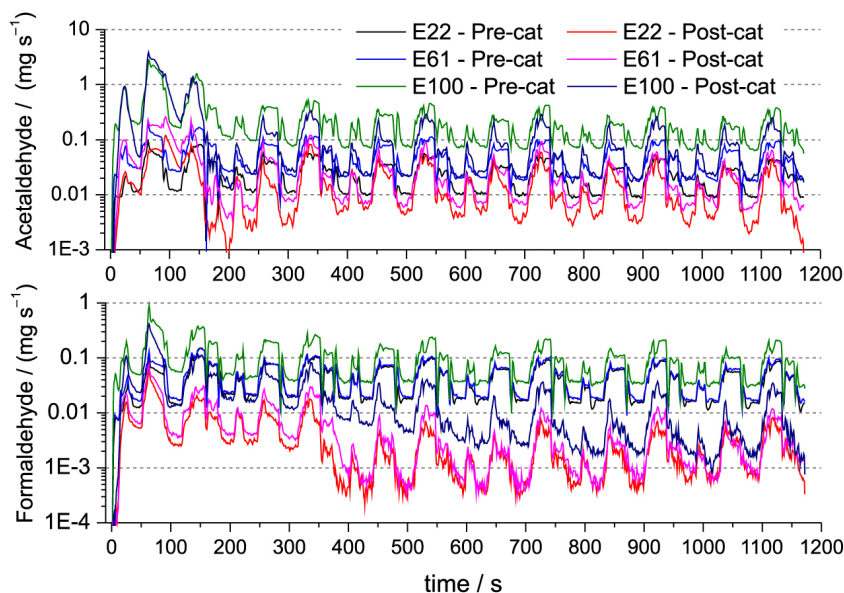

Figure 6. (a) Formaldehyde and (b) acetaldehyde before and after the catalyst, for different fuels.

emissions of formaldehyde.[46-48] However, in the literature, there are no reports of formaldehyde formation mechanisms with the use of hydrated ethanol (E100). In the case of acetaldehyde, all articles regarding the use of ethanol confirm its formation from the oxidation reaction during the combustion of ethanol.[49]

Table 2 shows the emission of aldehydes measured by the FTIR technique before and after the catalyst. The emissions before the catalyst during the cold phase for E22 present more formaldehyde than acetaldehyde. With an increase in the content ethanol (E61), the acetaldehyde emissions exceed formaldehyde, and using E100, acetaldehyde emissions are five times greater than formaldehyde emissions. This behavior can be explained by the fact that the engine is still cold, and combustion can be incomplete, releasing larger amounts of unburned alcohol from the combustion chamber. Figure 7 shows the emission of unburned ethanol at low temperatures.

According to Table 2, during the cold phase, the catalyst level of performance is lower. Formaldehyde conversions achieved values between 66% using E100 and 83% using E22. However, in the case of acetaldehyde for the same conditions, there was no conversion but an increase of this compound after the catalyst was observed. The formation of acetaldehyde in the catalyst is explained by the operating conditions. At the beginning of engine operation, combustion is inefficient due to low temperatures.[50] Thus, unburned ethanol can be oxidized and also dehydrogenated to acetaldehyde in CeO_2, a major component of the automotive catalyst, which is favored in the presence of platinum or palladium, the noble metals also traditionally used in these catalysts.[51-54] Thus, there is a clear correlation between acetaldehyde after the catalyst and the presence of unburned ethanol in the emissions (Figure 7).

In a steady state and after the catalyst, emissions are lower for formaldehyde and acetaldehyde. Formaldehyde

Table 2. Aldehyde emissions (in mg km^{-1}) measured before and after the catalyst by FTIR, during the cold phase and steady state and in the diluted emissions by HPLC

Fuel / SD	FTIR-raw exhaust-cold phase / (mg km^{-1})				FTIR-raw exhaust-steady state / (mg km^{-1})				HPLC / (mg km^{-1})	
	Pre-catalyst		Tailpipe		Pre-catalyst		Tailpipe		Diluted air	
	F	A	F	A	F	A	F	A	F	A
	5-390 s	5-200 s	5-390 s	5-200 s	> 390 s	> 200 s	> 390 s	> 200 s		
E22	9.17	6.22	1.58	6.33	7.58	4.42	0.35	2.78	0.93	3.00
SD	0.09	0.18	0.04	0.25	0.08	0.03	0.04	0.06	0.09	0.13
E61	10.12	13.48	2.45	16.11	8.23	8.78	0.50	4.26	1.54	6.92
SD	0.21	0.47	0.14	0.79	0.09	0.10	0.11	0.03	0.27	0.18
E100	27.35	130.9	9.41	135.71	15.68	33.57	1.62	14.29	3.31	32.50
SD	0.40	5.18	0.93	3.12	0.21	0.27	0.19	0.11	0.29	0.56

SD: standard deviation; FTIR: Fourier transform infrared; HPLC: high performance liquid chromatography; F: HCHO; A: CH_3CHO.
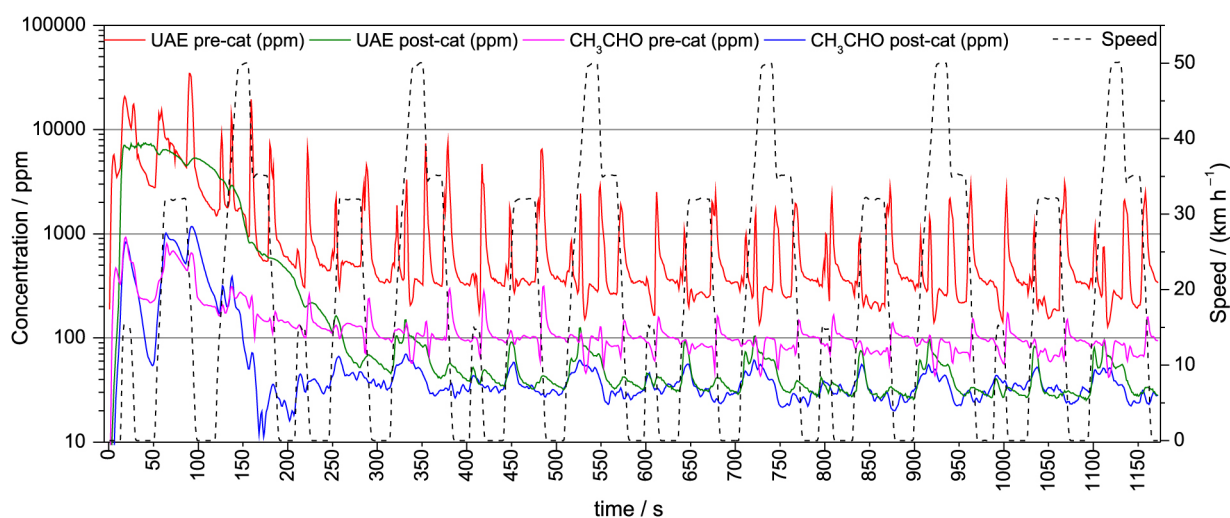

Figure 7. Unburned alcohol emissions (UAE) for E100.

conversion values are 90% for E100 and 95% for E22 and for acetaldehyde conversions are much lower, between 37% for E22 and 57% for E100, indicating the possibility of acetaldehyde being produced in the catalyst. Total aldehyde conversions in the cold phase are between 25% for E100 and 57% for E22, and steady state results are 68% for E22 and 75% for E100.

As previously mentioned, there is no consensus in the literature about formaldehyde emissions, but an increase in ethanol content promoted the formation of not only acetaldehyde, as expected, but also formaldehyde. In addition, before the catalyst, a linear decrease in the formaldehyde/acetaldehyde ratio with ethanol content is observed, either during the cold phase or steady state, as indicated by Figure 8. The differences observed

between the profiles before the catalyst emphasize that a higher temperature during the fuel combustion favors formaldehyde formation. After the catalyst, the behavior changes. The formaldehyde/acetaldehyde ratio also shows a linearly decreasing trend with the increase of the ethanol content. However, after the cold phase, this ratio is approximately 0.13 and is virtually independent of the ethanol content, indicating an equilibrium behavior.

In an attempt to achieve a better understanding of the formaldehyde formation, Figure 9 presents the emissions for the initial 195 s of the test before the catalyst, making it possible to observe the relationship of unburned ethanol with emissions of methane (CH_4), ethylene (C_2H_4), methanol and formaldehyde.
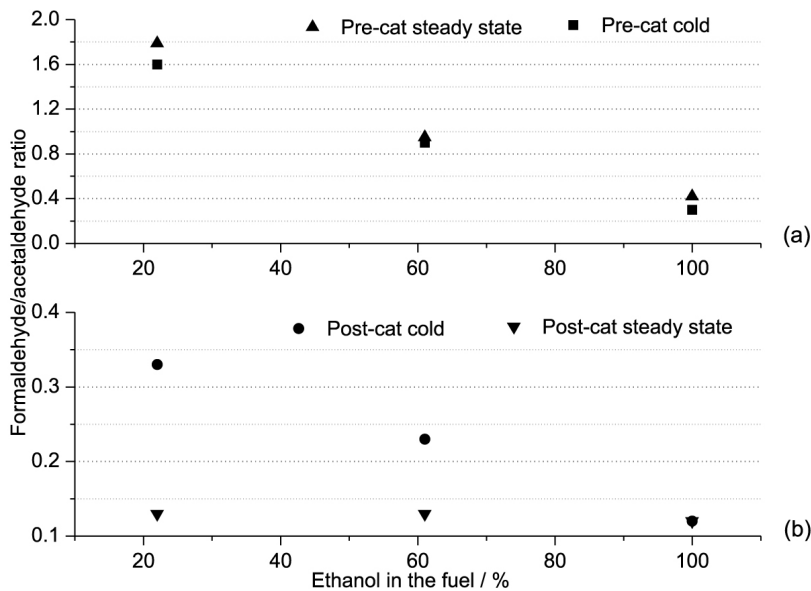

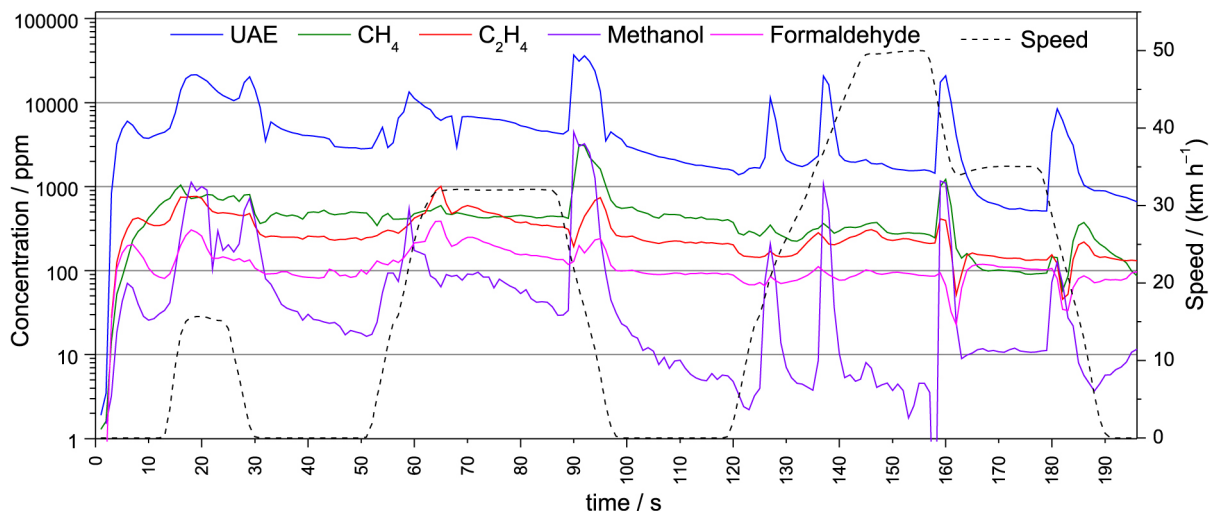**Figure 8**. Formaldehyde/acetaldehyde ratio (a) before and (b) after the TWC, measured by FTIR.

Figure 9. Emissions of formaldehyde, methanol, CH_4, C_2H_4 and unburned alcohol emissions (UAE).
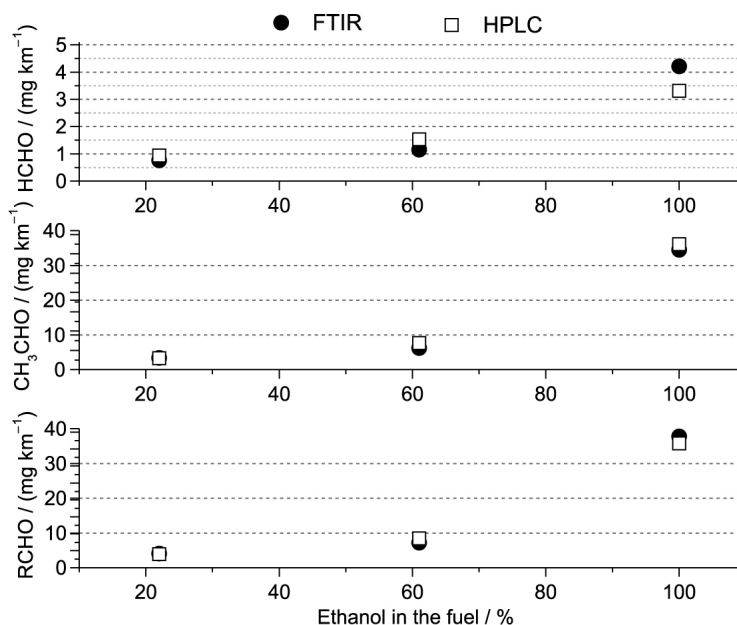
Figure 10. Formaldehyde, acetaldehyde and total aldehydes measured by FTIR and HPLC.

Some reports in the literature[44,55] state that formaldehyde is formed from reactions involving methane and compounds with two carbons. There are also studies indicating that formaldehyde occurs as an intermediate species in hydrocarbon combustion, being formed early in the combustion at low temperatures.[56]

Considering the formation of formaldehyde via CH_4, once the necessary energy to activate the CH_4 molecule during the fuel combustion has been achieved, a series of reactions involving $CH_3\bullet$ radical with itself and with other radicals, such as oxygen species, may occur. Reaction of the $CH_3\bullet$ radical with an oxygen species promotes the formation of methanol, which can be partially oxidized to formaldehyde.[55] Thus, the aldehydes present in the exhaust gases may form as intermediate species by the post-combustion oxidation of methanol.

Finally, it is equally important to evaluate the raw emission values after the catalyst. In the cold phase, formaldehyde emissions were 1.6, 2.5 and 9.4 mg km^{-1} using E22, E61 and E100, respectively, while for the same range, emissions of acetaldehyde were 6.3, 16.1 and 135.7 mg km^{-1}. In the steady state, the formaldehyde values were at 0.4, 0.5 and 1.6 mg km^{-1}, and acetaldehyde values were 2.8, 4.3 and 14.3 mg km^{-1}. Currently, for light vehicles, the legislation states that the total aldehydes (RCHO) should be below 20 mg km^{-1} (L6 phase of PROCONVE). Comparing this value with the values obtained, we conclude that the motorcycle would meet the limits of total aldehyde emissions for the steady state phase. However, in the cold phase, the emission from E100 exceeds the threshold value.

The average values obtained with the FTIR technique were compared with those obtained by HPLC using the analysis of variance (Figure 10). The post-catalyst formaldehyde emissions were found to be statistically equivalent. Acetaldehyde emissions show significant differences for E61 and E100. The average emission value by FTIR for the total aldehydes was 14% lower than with the use of HPLC for the E61 because the FTIR samples raw emissions in the exhaust, and the HPLC methodology collects the sample after the dilution process. The total aldehyde emissions for E100, measured by HPLC, presented values 8% lower than the values obtained by FTIR, and the effect is constant for both test cases (pre- and post-catalyst). Different values between FTIR and HPLC techniques can be attributed to corrections made in sampling/dilution procedures between the two techniques.

Conclusions

The methodology used for collecting and analyzing aldehydes in motorcycle emissions is technically feasible, and the collection process is effective and shows good repeatability between tests.

The use of FTIR to identify and quantify formaldehyde and acetaldehyde provided additional information for understanding the formation and emission processes. The results demonstrate the usefulness of this technique in the investigation of vehicular non-criteria emissions and provided a comparison with the traditional HPLC methodology, where the results obtained for the use of gasoline and ethanol were considered statistically equivalent.

Regarding criteria pollutants, the catalyst performance showed lower values than the values obtained in automobiles, which have efficiency values greater than 90% for these compounds, because there is less control of the air/fuel ratio on motorcycles. The question of cold phase emissions should also be assessed because the conversions were too low and lower than 10% in some cases.

With regard to non-criteria pollutants, an increase in ethanol content in the fuel promoted an increase in aldehyde emissions. The formaldehyde/acetaldehyde ratio linearly decreased with the ethanol content in the fuel when measured before the catalyst in both the cold phase and the steady state and in the cold phase when measured after the catalyst. The same analysis after a catalyst in the steady state showed that the ratio remained stable and did not vary with ethanol content, suggesting an equilibrium behavior.

Under steady state conditions, the catalyst was found to be efficient for formaldehyde conversion, regardless of the fuel used. However, for the acetaldehyde under the same conditions, the catalyst performance was lower, probably because of a significant increase in acetaldehyde emissions and the presence of unconverted ethanol in the exhaust gases, which can be related to the formation of acetaldehyde in the catalyst.

In the cold phase, the combustion process proved to be incomplete, with the presence of unconverted ethanol at significant levels and methane, ethylene, methanol, and aldehydes, among others, in the exhaust gases analyzed before the catalyst. Under these conditions, the catalyst is cold, hindering the conversion of the pollutants. However, at low temperatures, the catalyst can promote reactions such as the ethanol dehydrogenation, and at 200 °C and above, it can form acetaldehyde, as found in the emissions before and after the catalyst up to 350 s.

The results obtained accent the need to reduce the time needed to reach the steady state. The approximate value obtained in this study is approximately 6 min, and this amount of time is very high, considering that the paths in a small or medium city can be short, and the catalyst is underutilized, resulting in high emissions of aldehydes and other pollutants. One possible solution would be to place the catalyst near the engine, as in systems with close-coupled technology and as used in light vehicles to reduce the time to start the catalytic reactions. This option should be accompanied by a study of the thermal stability of the catalyst components.

Another important aspect to emphasize is the composition of the catalyst, which must be optimized for motorcycles to avoid undesirable reactions or the generation of non-criteria pollutants.

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