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Prediction of flammability limits for ethanol-air blends by the Kriging regression model and response surfaces

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A R T I C L E I N F O

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Flammability limits
Ethanol
Kriging
Response Surface Methodology
Experimental data

A B S T R A C T

Flammability limits must be identified in order to assess and control handling risks of particular processes according to combustion product composition and environmental conditions. Thereby, the present paper aims to present a model that can predict flammability limits for ethanol-air blends with at different moisture concentrations by Kriging interpolation techniques. The model is based on experimental results to determine flammability limits of ethanol-air blends, and evaluate different moisture concentrations in ethanol composition. It has accurately predicted the flammability limits of ethanol-air blends at temperatures ranging between 20 °C and 210 °C, pressure values ranging from 40 kPa to 101.3 kPa, ethanol moisture concentration at 0.5% and 8%, and ethanol volume percentages from 1% to 35%. Thus, it is a valid tool to accurately and efficiently determine flammability limits of ethanol-air blends.

1. Introduction

Risks from combustion products and fuels have been posed by a wide variety of industrial processes, as well as transportation and storage operations. Fuel-air blends combust once their concentrations are within well-defined limits, i.e. lower and upper limits, also known as flammability limits (FL) or explosivity [1], which are usually determined experimentally. Since FLs are so important, many different experimental methods have been developed in order to determine them [2–5]. However, such experimental procedures are complex and time-consuming, especially when pressure and temperature conditions are rather high [6].

A large variety of empirical and semi-empirical methods are currently proved to determine flammability limits of fuel blends based on some of their characteristics. Generally speaking, these methods are adjusted so as to determine FLs. However, there are a differences when estimating upper flammability limits experimentally (UFLs) in comparison with those predicted theoretically [7].

Among the theoretical methods found in literature, some are worth mentioning, such as those based on combustion enthalpy for UFLs estimation [8,9], the stoichiometric concentration of blends [10–12], a few on vapor pressure [13], and some on the number of moles of the fuel under analysis [7]. On the other hand, Coward and Jones [14] stated that FLs should be determined at constant pressure, whose method has also been adopted by Mishra and Rahman [15]. A combustion process under constant pressure has been carried out by other researchers to determine FLs, which is consistent with the experimental determination [16].

The present study has not been focused on physical processes directly, but it is based on some characteristics of combustion processes in order to predict FLs of ethanol-air blends (previously specified ranges of pressure, temperature, ethanol content and moisture concentration) with a considerable degree of approximation. Furthermore, a statistical analysis has been conducted, through which very good results were obtained for predicting FLs of ethanol-air blends. In another manuscript, predicted FLs theoretically [17], especially UFLs regarding the physical principle of combustion, has already been approached. In this manuscript, the developed method applies the basic combustion theory and chemical equilibrium. The UFLs are determined by correlating a parameter named θ, which is the ratio of adiabatic flame temperature at stoichiometric composition to adiabatic flame temperature at UFL composition [17].

This paper is aimed at introducing a FLs prediction model for ethanol-air blends with different moisture concentrations based on

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Table 1
Parameters considered in experimental measurements.

Parameter	Range	Units
Humidity content	0.5–8.0	%
Fuel volume in the mixture	1–35	%
Pressure in the container	40–101.3	kPa
Mixture temperature	20–220	°C

Kriging interpolation techniques. The model has been developed through 342 experiments that were conducted on an experimental bench designed specifically for this research.

The method proposed herein offers advantages and disadvantages. The main disadvantage is that it only allows predicting FLs of ethanol-air blends within the aforementioned pressure ranges (mix), fuel volume, moisture concentration and temperature. However, its major advantage is that FLs have been predicted through an interpolation method with a reasonable degree of accuracy because deviations would originate essential approximations from experimental measurements.

Ethanol has been used in Brazil for 20 years as fuel for automotive vehicles, which is the only country worldwide where ethanol can be solely used as fuel in vehicles. In recent years, tests have been carried out on the use of ethanol in aircrafts by using the same kind of alcohol that is typically used in the Brazilian vehicular fleet. There are 02 types of ethanol sold in Brazil: the anhydrous ethanol with alcohol content of 99.5% in mass and hydrated ethanol with alcoholic content of 92% in mass and 96% in volume, which have been selected for the present experimental tests.

Alcohol cannot only be used in the automotive industry, but also in the aeronautical industry, thus it is initially proposed to use the same types of alcohol with the same moisture concentration as the one used by the Brazilian automotive industry. Therefore, the prediction model proposed herein allows predicting the FLs in fuels with moisture concentration ranging between high (8%) and low (0.5%) values.

The presently developed experimental bench was designed for aeronautical applications, thus the present study has been conducted with operating pressures below atmospheric pressure, e.g. by considering a typical cruising altitude of 40,000 ft (approximately 18.82 kPa). Thereby, the proposed model is suitable for predicting FLs of ethanol-air blends at temperatures and pressure conditions within the previously specified ranges. (40 kPa–101.3 kPa).

Fig. 1. Flammability apparatus.

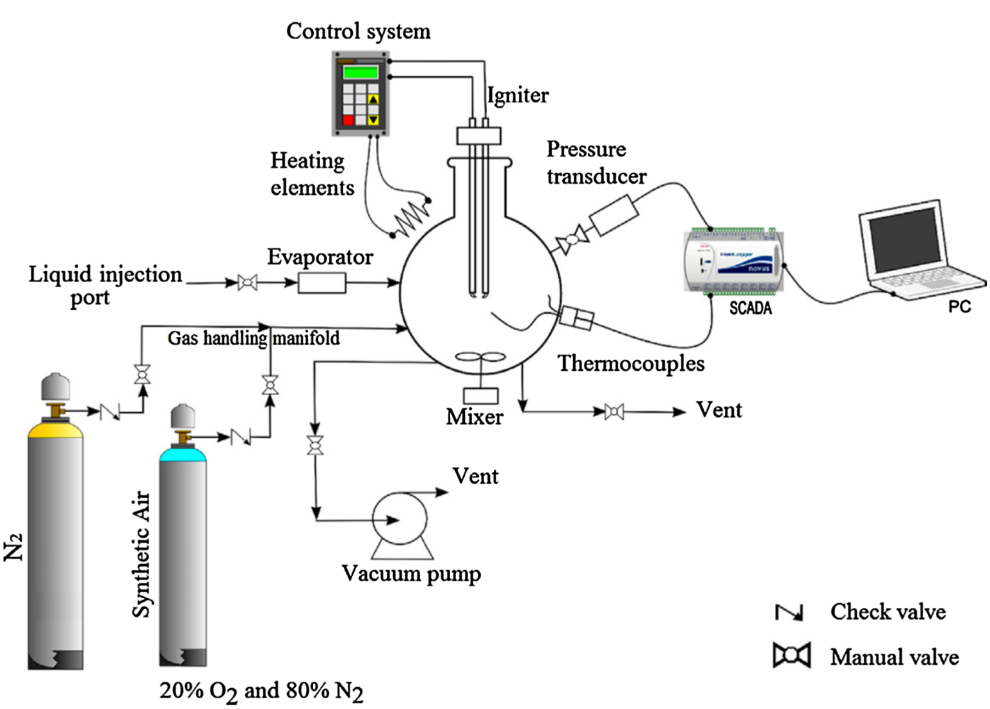


Fig. 2. Flammability apparatus (experimental bench testing).

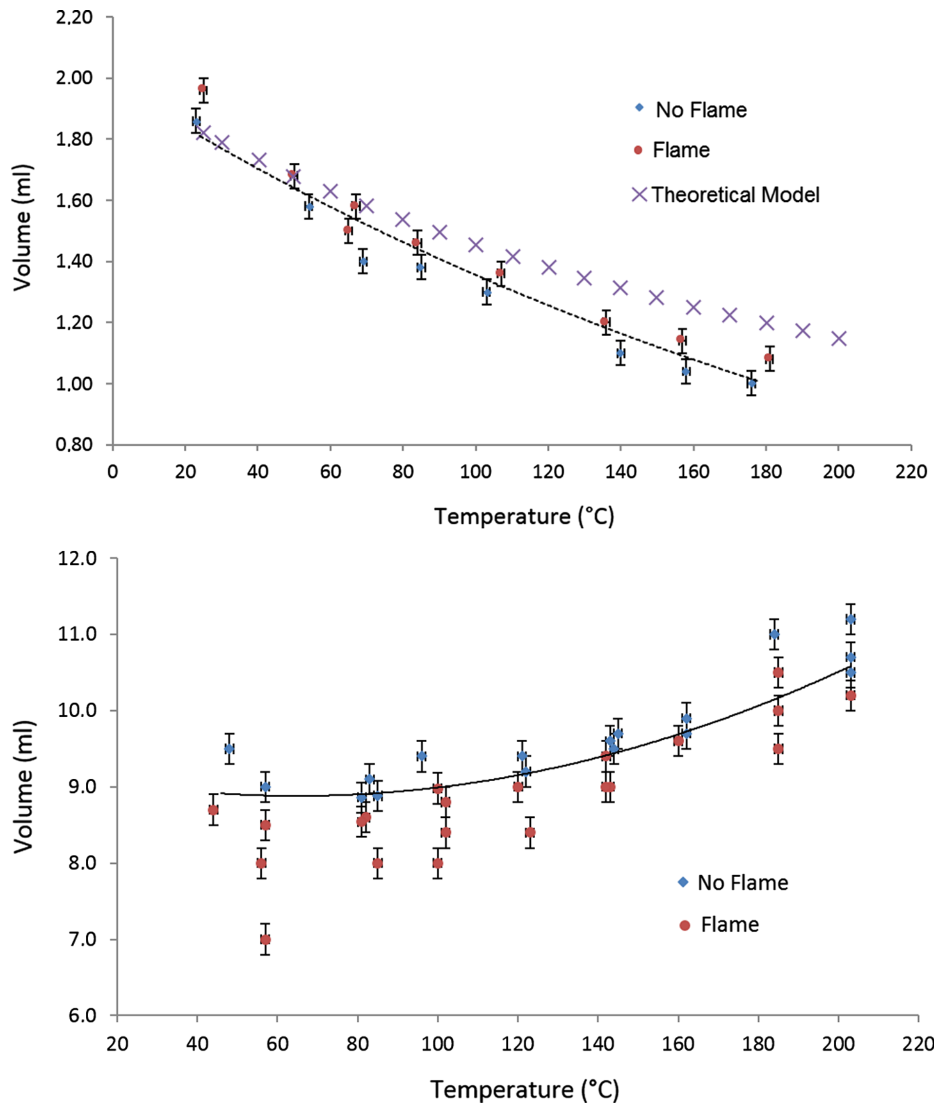


Fig. 3. Results of the tests anhydrous ethanol to 101 kPa for LFL and UFL.

Table 2
Characteristics of measurements for the lower and upper limit of anhydrous ethanol.

	Pressure	Standard deviation [kPa]	Number of experimental tests	Temperature range [°C]	Error as ethanol volume [ml]
LFL	101	1,178	17	23–181	0,04
	80	0,267	17	28–172	0,04
	60	0,340	16	23–175	0,02
	40	0,350	18	25 – 179	0,02
UFL	101	0,633	39	44–203	0,20
	80	0,739	37	40–205	0,14
	60	0,483	25	40–202	0,10
	40	0,652	22	40–203	0,06

2. Methodology: Kriging models and Response Surface Methodology

Data interpolation allows drawing maps of continuous surfaces for obtaining a discrete set of data points. However, when there are too many data points, its use is limited [18]. The Kriging method is an interpolation technique that uses an estimation procedure which provides good linear assumptions of analyzed values by choosing a weighted average of sample values with the least variance. This method quantifies spatial data structure by variograms that utilize statistical procedures, in which it is assumed that data closer to a known point has greater weight or influence over interpolation. This influence is reduced as data moves away from the observed point [19].

Another aspect that should be considered in the Kriging analysis is the tendency for isotropy or anisotropy. Anisotropy indicates whether a variable has spatial dependence towards one or more directions. If it is stronger, it can be used to determine more homogeneous areas according to the unit of measurement, which can be useful for determining experimental regions [20].

The Response Surface Methodology (RSM) is a set of mathematical and statistical techniques that are utilized to model and analyze problems of several individual or combined parameters. It was developed by Box and Wilson [21] with the aim of studying the relationship between a response variable and several explanatory factors. RSM has been widely used as an optimization technique [22,23]. The method can also be used as a prediction technique [24,25].

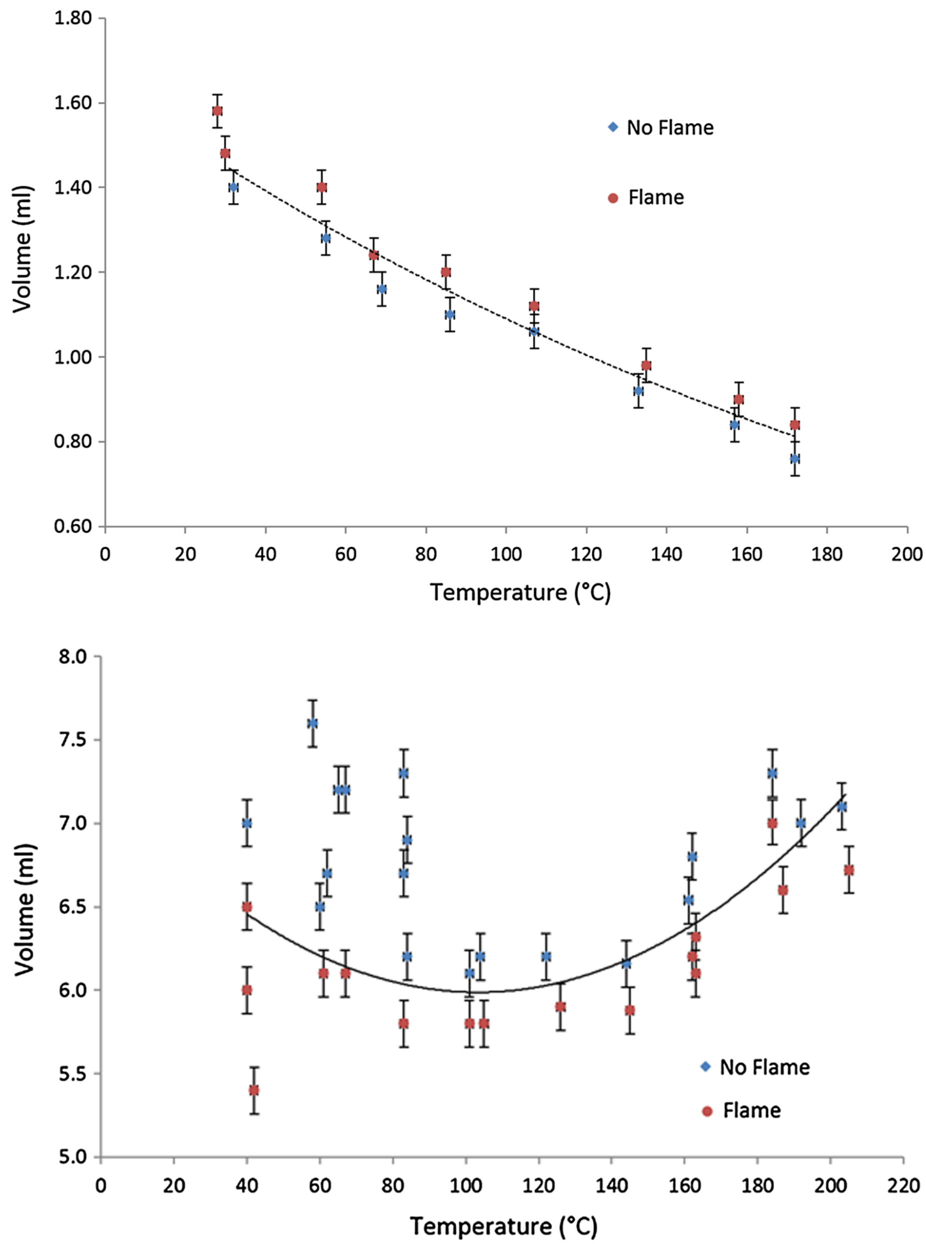


Fig. 4. Results of the tests anhydrous ethanol to 80 kPa for LFL and UFL.

3. Data and model testing

A flammability evaluation technique has been developed in which the output variable (flammability index) is a continuous variable, not a discrete variable (flammable or non-flammable). Thereby, a value of 0 is set to blends that have a Lower Flammability Limit (LFL) and 1 indicates that they have an Upper Flammability Limit (UFL). Thus, blends that have flammability index ranging between 0 and 1 are flammable, whilst those with lower flammability indexes of 0 or higher than 1 are non-flammable.

Thus, any blend can be evaluated as regards its flammability index, which can assume positive or negative values. Therefore, flammability indices closer to zero or one will be obtained by blends that are very close to their LFLs or UFLs, respectively.

An algorithm has been developed herein that not only predicts whether the blend is flammable or non-flammable, but also quantifies how close it is to upper and lower FLs. The developed prediction algorithm was based on experimental measurements.

Blends with different moisture concentrations were considered for experimental measurements, as well as percentages of fuel volume for different pressure and temperature conditions in the flask. The considered variables and their different evaluation ranges are presented in Table 1.

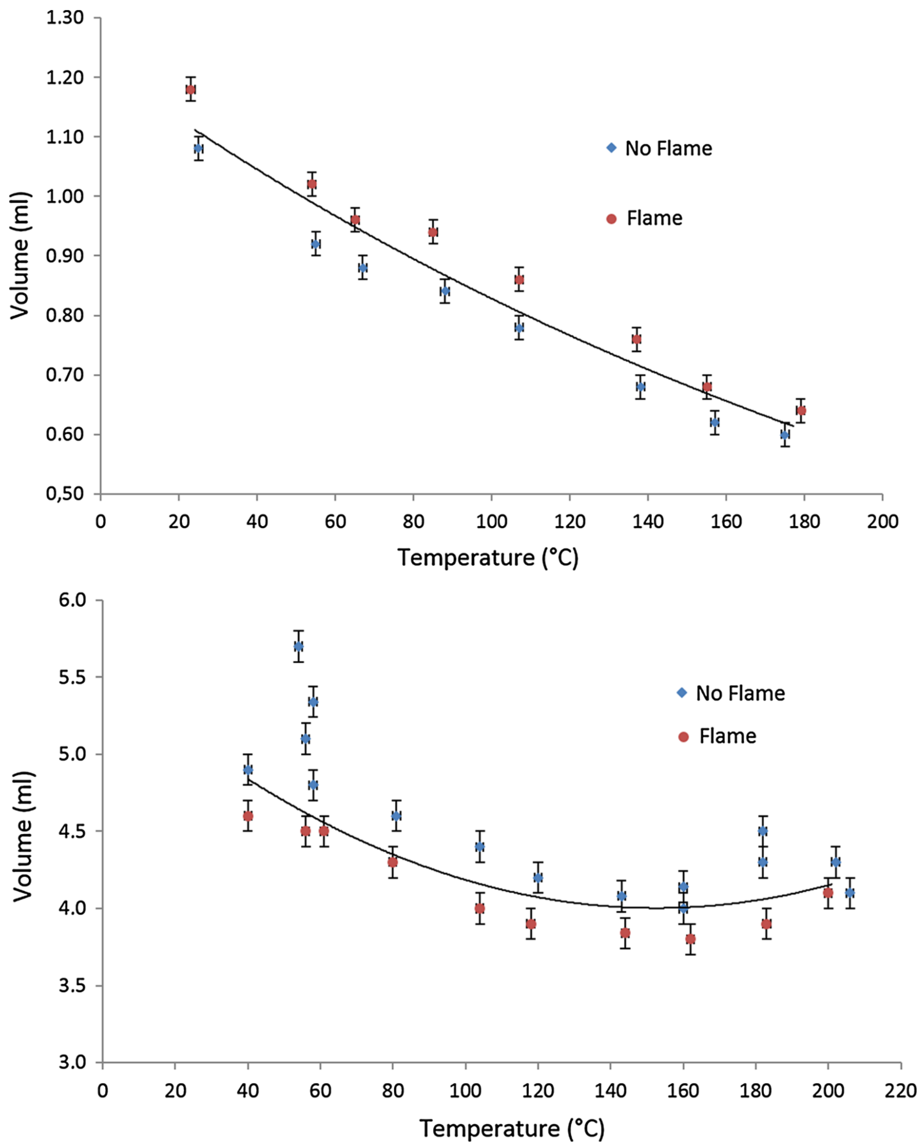


Fig. 5. Results of the tests anhydrous ethanol to 60 kPa for LFL and UFL.

4. Flammability apparatus and experimental procedure

A flammability heating chamber has been designed in accordance with American regulation ASTM E-681 [7]. The chamber was heated by using electric heating elements which are capable of increasing temperature to 300 °C. It also has a thermal insulation unit and a window for observing and recording the flame structure during each experiment. For this study, a high-speed Fujifilm® FinePix HS-10 camera was used to take pictures at high speed during each experimental test, whose details can be seen in Fig. 1. This heating chamber is an improved version of the one that had been previously designed by [12] to test flammability limits of aeronautical ethanol.

A 20-l spherical test flask has been used. It should be noted that the volume established by American Standard E-681 was increased because no specification is provided as regards its use at high temperatures and reduced pressures, which requires lower amounts of air and fuel. A drawing of the apparatus is shown in Fig. 2. The flammability apparatus was automated by using a Supervisory Control and Data Acquisition (SCADA) unit. The SCADA was connected to a computer for monitoring all variables and storing data.

The experimental procedure was in accordance with ASTM E681. For further details about the flammability apparatus and experimental procedure, it is recommended to read papers published in 2012 [7] and 2014 [26] and the master's degree thesis by [27].

5. Experimental tests results

Ethanol flammability limit curves were determined by experimental results of two different alcohols, anhydrous ethanol (99.5% ethanol and 0.5% water) and hydrated ethanol (92% ethanol and 8% water). The experiments reported herein were performed at temperatures ranging between 20 °C and 220 °C, and pressures ranging between 40 kPa and 101.3 kPa.

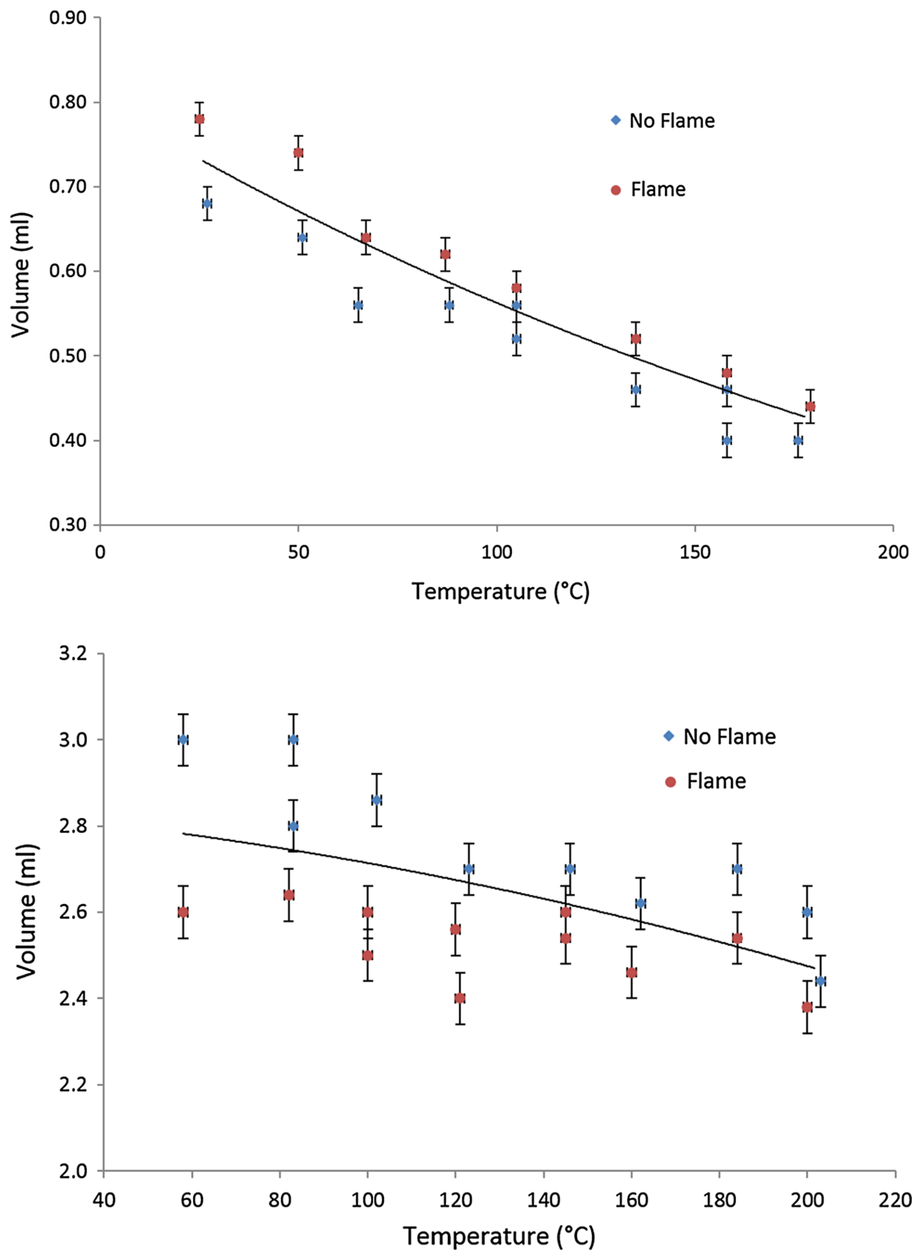


Fig. 6. Results of the tests anhydrous ethanol to 40 kPa for LFL and UFL.

5.1. *Results of anhydrous ethanol experimental tests*

Fig. 3 presents LFLs and UFLs experimental results at a pressure of 101 kPa, which matched theoretical calculations to determine flammability limits found in literature. Thus, it is verified the present experimental procedure and a correct operation of the Flammability Apparatus. Table 2 shows the details of experimental tests and Figs. 4–6 show results at reduced pressures.

5.2. *Results of hydrated ethanol experimental tests*

Fig. 7 presents the LFLs and UFLs results at a pressure of 101 kPa for hydrated ethanol, which were similar to those obtained by anhydrous ethanol. They were compared to other pieces of data from literature. Table 3 shows the test details and Figs. 8–10 show results at reduced pressures.

Minimum and maximum fuel volume values were determined and there was flame propagation in the tests flask (20.716 l). However, these volumes must be expressed as significant values for any volume. In order to do so, the methodology described by ASTM E681 has been adopted, which turns maximum and minimum volume values obtained in the heating chamber into volume percentage values that are valid for any volume as a function of temperature. The equation to convert these volumes is as follows:

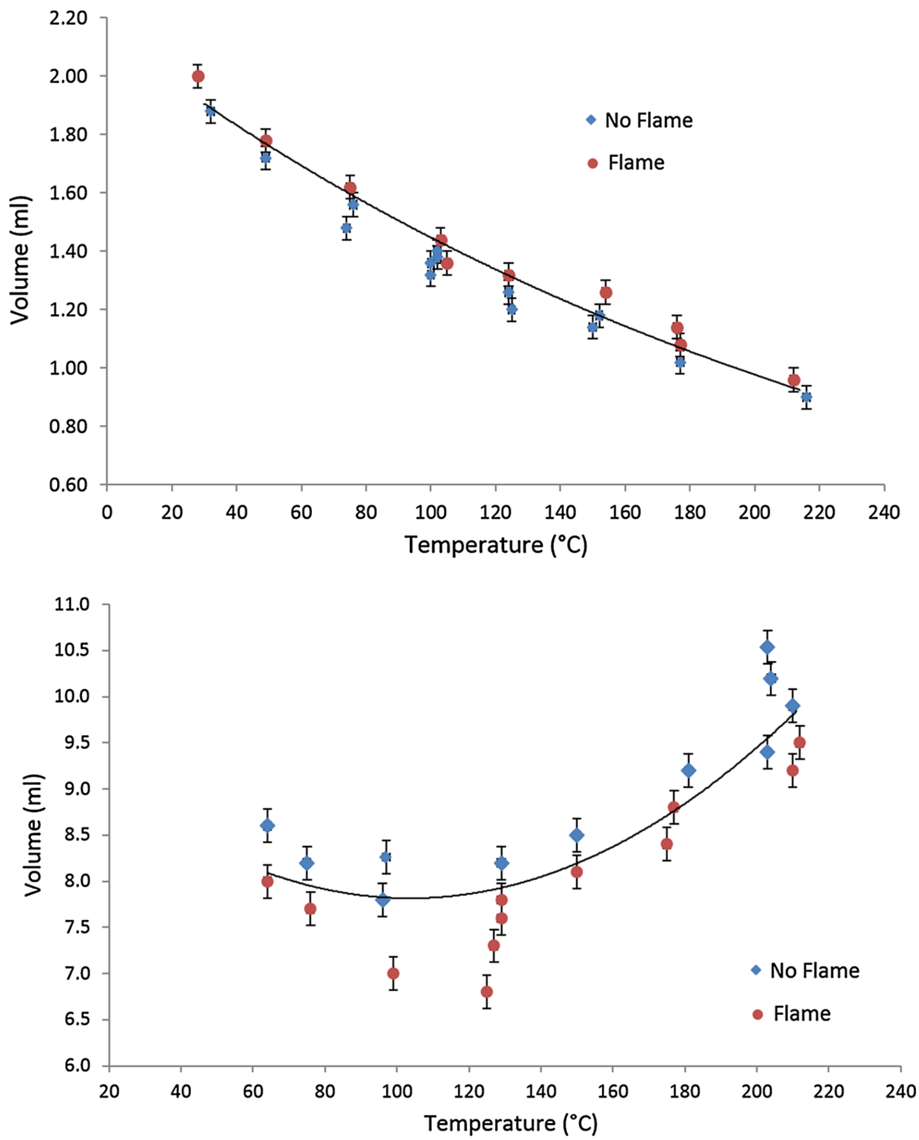


Fig. 7. Results of the tests hydrated ethanol to 101 kPa for LFL and UFL.

Table 3
Characteristics of measurements for the lower and upper limit of hydrated ethanol.

	Pressure	Standard deviation [kPa]	Number of experimental tests	Temperature range [°C]	Error as ethanol volume [ml]
LFL	101	1,1221	24	28–216	0,04
	80	1,0288	19	28–212	0,04
	60	1,8630	18	27–204	0,02
	40	1,5904	16	32 – 205	0,02
UFL	101	0,8052	23	64–212	0,18
	80	0,7844	18	51–212	0,12
	60	0,6585	14	68–216	0,10
	40	0,6675	19	65–219	0,06

$$\%V = \frac{v_f \cdot \rho_f}{MM}\frac{22{,}4}{V}\frac{P_0}{P}\frac{T}{T_0}*100 \tag{1}$$

where:

v_f: ethanol volume [cm^3]
ρ: ethanol density (anhydrous ethanol 0.7915 g/cm^3)
MM: ethanol molecular mass (46.07 g/mol)
P: Pressure [mmHg]
T: Ethanol-air blend temperatures [K]
V: Volume [L]
P_0: Standard pressure (760 mmHg)
T_0: Standard temperature [273.15 K]

From the experimentally determined flammability limits, Flammability Index valuations were attributed to each of the selected

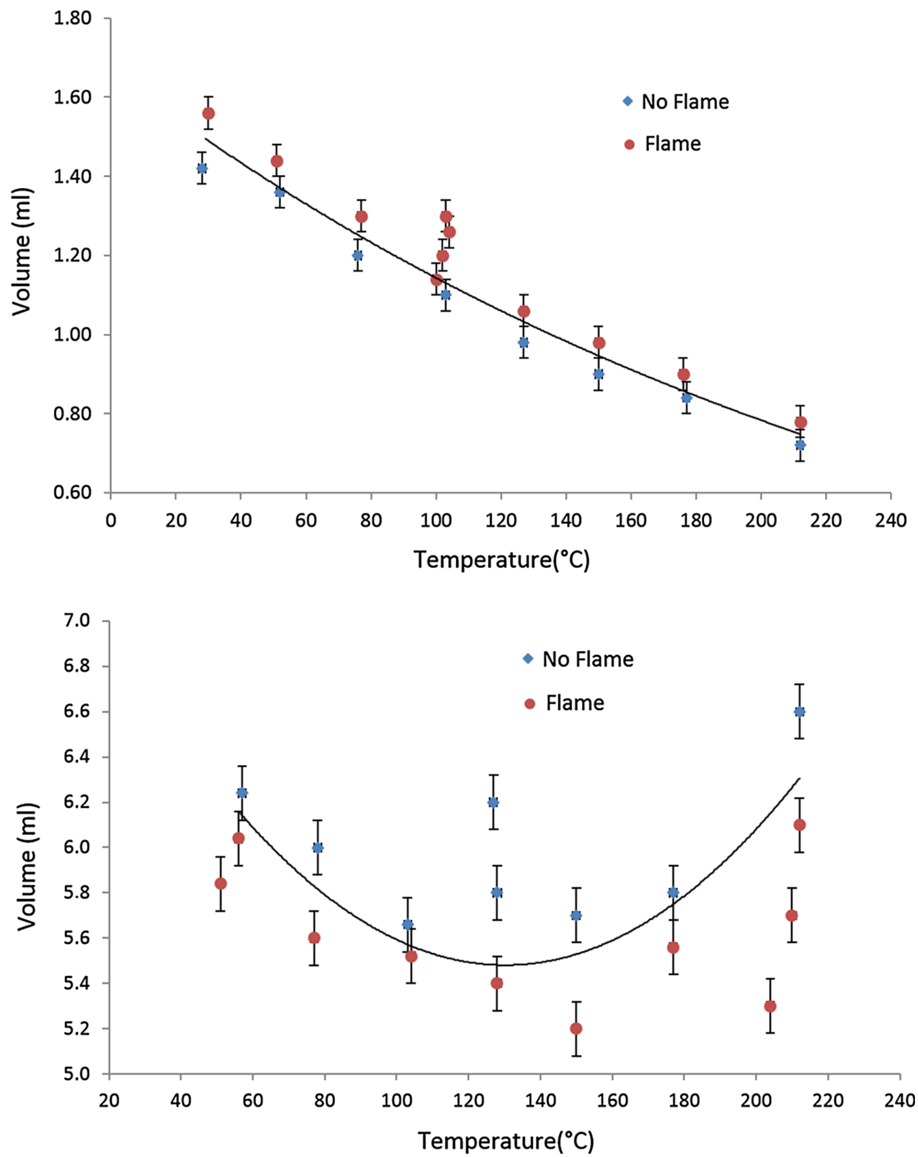


Fig. 8. Results of the tests hydrated ethanol to 80 kPa for LFL and UFL.

blends. These valuations were attributed in accordance with the following Equation:

$$IF = 1 - \left(\frac{\nu_{ul} - \nu_a}{\nu_{ul} - \nu_{ll}}\right) \tag{2}$$

where:

ν_{ul}: Percentage of fuel volume in the upper flammability limit
ν_{ll}: Percentage of fuel volume in the lower flammability limit
ν_a: Percentage of actual fuel volume.

Four input parameters were assigned to prepare the Response Surface: pressure (P), temperature (T), moisture concentration (% h), and fuel volume percentage (% V). Flammability Index was established as an output parameter.

Once flammability indexes were established for all evaluated experimental configurations, data preparation has been initiated for generating the Response Surface. Data used in the current study comprise 342 experimental tests. The tests were divided into a couple of groups: one for the response surface calculation with a total of 242 experimental tests, and another one comprising 100 tests that were used for their respective validation.

6. Discussion

Experimental data were processed through the Kriging data interpolation statistical technique by using a Gaussian variogram. The modeFRONTIER software [28] was used for data processing, which allows generating a Java code algorithm that allowed predicting flammability indexes for ethanol-air blends under specific conditions.

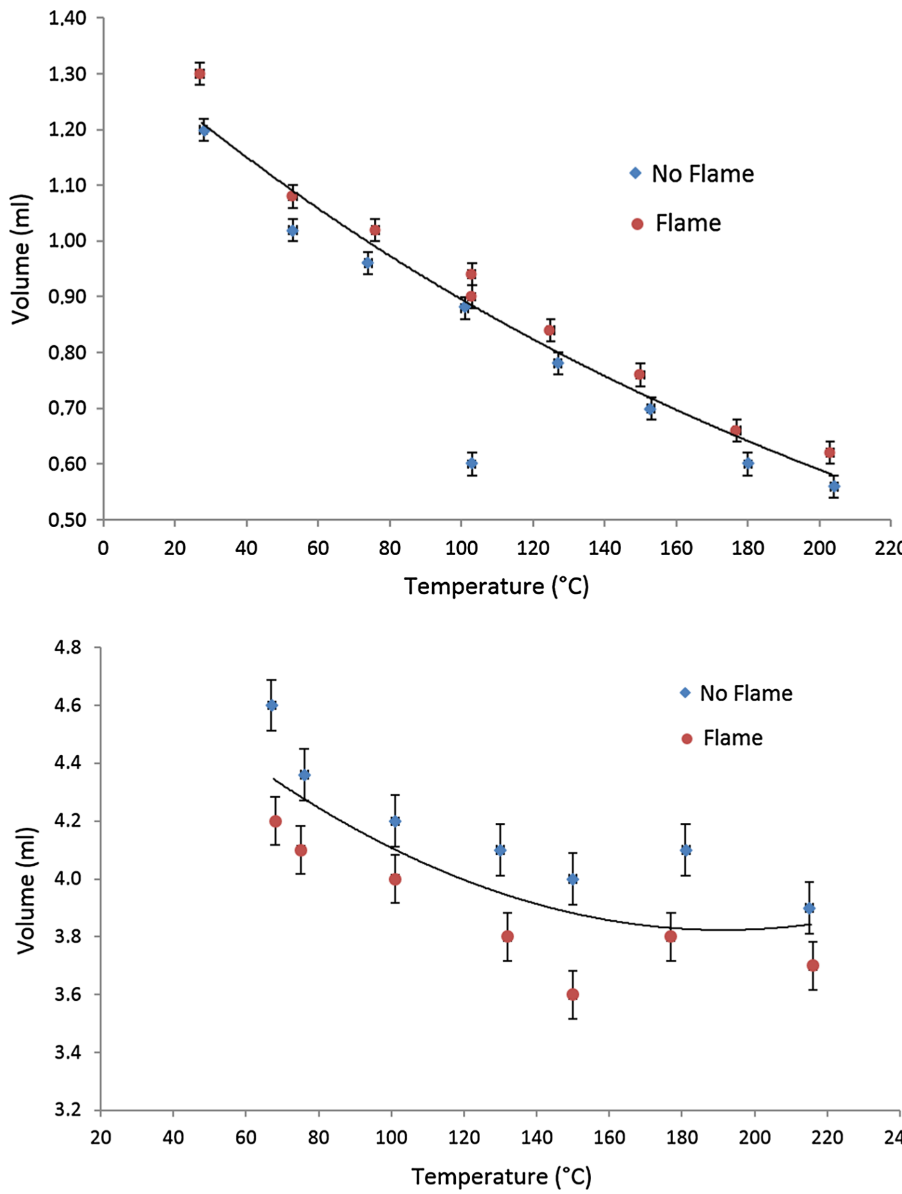
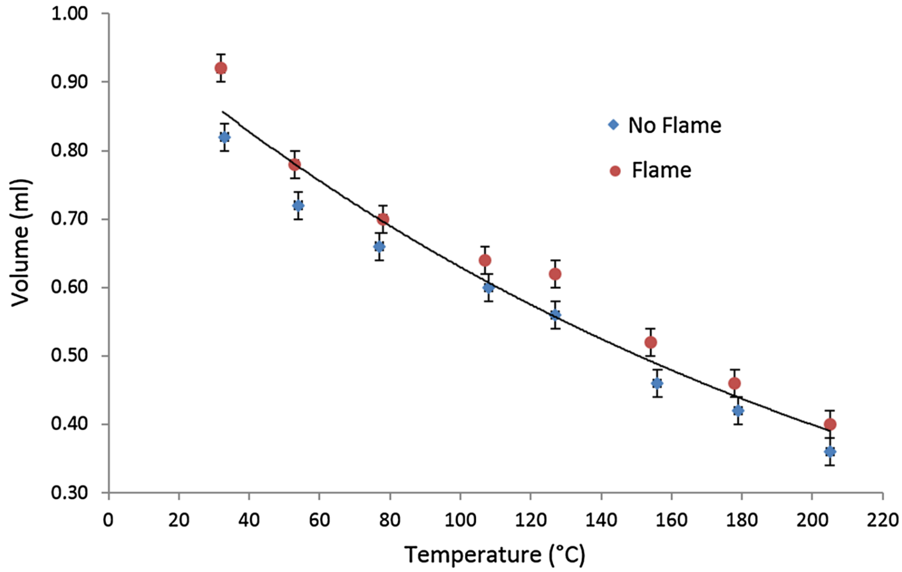


Fig. 9. Results of the tests hydrated s ethanol to 60 kPa for LFL and UFL.

Once the prediction algorithm was obtained, its validation procedure was performed. For such a purpose, 342 experimental data records were used through which a good performance of the generated algorithm was evidenced. Figs. 11–12 show the experimental results of flammability index deviations that were not considered during algorithm development. These are due to predicted flammability indexes being obtained through the prediction algorithm for upper and lower flammability limits, respectively.

From these results, it can be observed that the average of absolute deviation values in experimental data fitting as regards those obtained by the prediction algorithm is lower than 0.2%, whose standard deviation is below 0.3% for LFLs and lower than 0.5% and 0.6% for UFLs, respectively. These results suggest that the prediction algorithm is significantly accurate. It is necessary to clarify that such deviations correspond to the flammability indexes calculated by the proposed model with respect to those expected from experimentally obtained curves (Figs. 2–10). These deviations are quite low because the model is sufficiently precise. In addition, flammability indexes generally assume values between 0 and 1, whereas the data presented in Figs. 2–10 show error bars of fuel volume (ml).

LLs and ULs mean LFLs and UFLs, respectively. Similarly, a numeric expression included in these captions indicates the pressure at which the tests were performed. “A” refers to blends with anhydrous ethanol as fuel, “H” refers to those with hydrated ethanol, “w” refers to



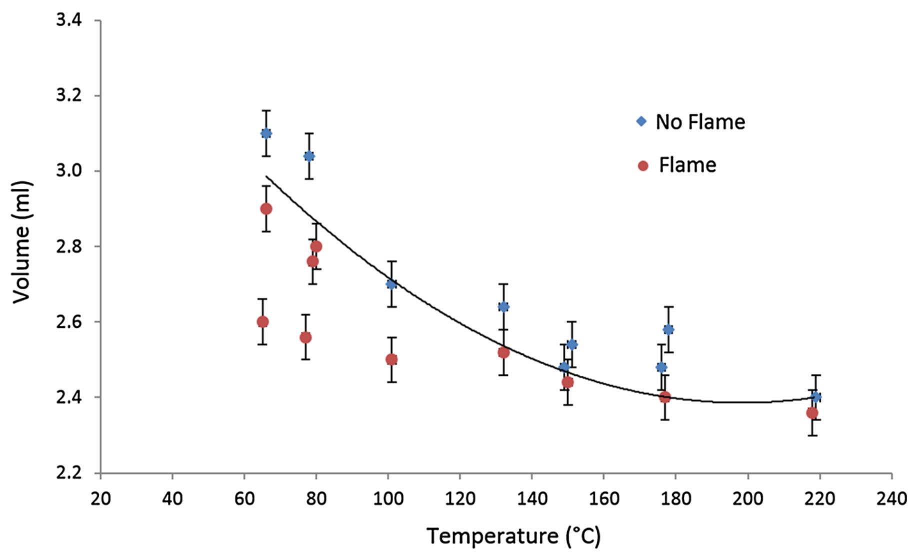


Fig. 10. Results of the tests hydrated ethanol to 40 kPa for LFL and UFL.

experimental tests in which there was no flame propagation, “p” to experimental tests that presented flame propagation, and “k” to flammability limits determined through the prediction algorithm that has been developed based on the Kriging methodology. This codification has been detailed in Fig. 13.

After validating the flammability prediction algorithm, flammability limit curves were obtained from the data provided by the algorithm, whose pressures were set at 40 kPa, 60 kPa, 80 kPa, and 101 kPa, and anhydrous and hydrated ethanol blends. These curves are shown from Figs. 14–21, along with experimental data for different pressures inside the tests flask tests. Experimental results for blends which presented flammable or non-flammable conditions are in red and blue, respectively, in the aforementioned figures. In addition, the experimental configurations that presented flammability condition close to LFLs are represented by a diamond, and those that presented a flammability condition close to UFLs are represented by a square.

According to these results, it can be concluded that the proposed algorithm can adequately predict the flammability limit of blends that exhibit variations in operating pressure and temperature, the percentage of ethanol volume in the blend, and moisture concentration in ethanol within the ranges considered herein for each analyzed parameter.

From these results, it can be observed that variations in pressure, moisture concentration in ethanol and percentage of fuel volume for ethanol-air blends do not present significant differences regarding LFLs. However, there are rather marked differences in UFLs, especially at high temperatures, which can be observed in Figs. 22 and 23 in which

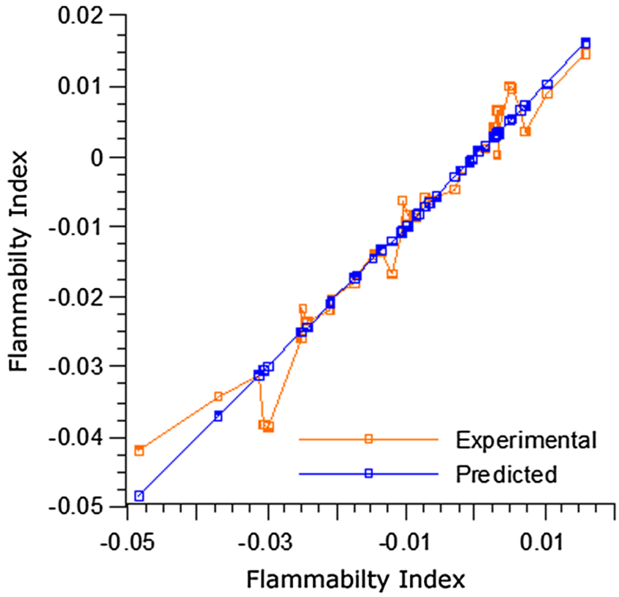


Fig. 11. Deviation of flammability values used in validation of the prediction algorithm in the lower limit.

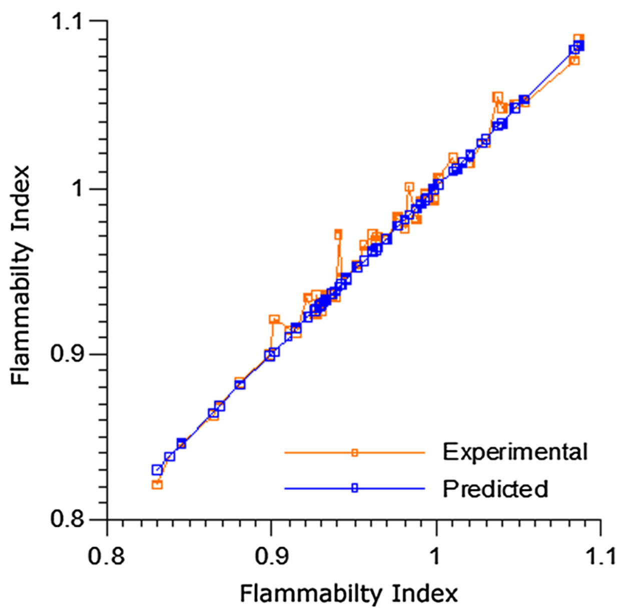


Fig. 12. Deviation of flammability values used in validation of the prediction algorithm in the upper limit.

sets of flammability limits for anhydrous and hydrated ethanol blends are shown, respectively.

Moreover, these results clearly reveal the manner through which flammability limits of ethanol-air blends are slightly reduced when moisture concentration in the fuel increases. This difference is shown in Fig. 24, where flammability limits of anhydrous ethanol-air and hydrated ethanol-air blends are compared. Moisture reduces the flammable area of ethanol-air blends, i.e. anhydrous ethanol is more flammable. The greatest differences are observed for UFLs, thus, in addition

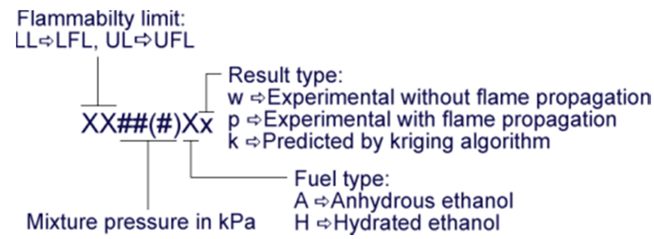


Fig. 13. Codification employed.

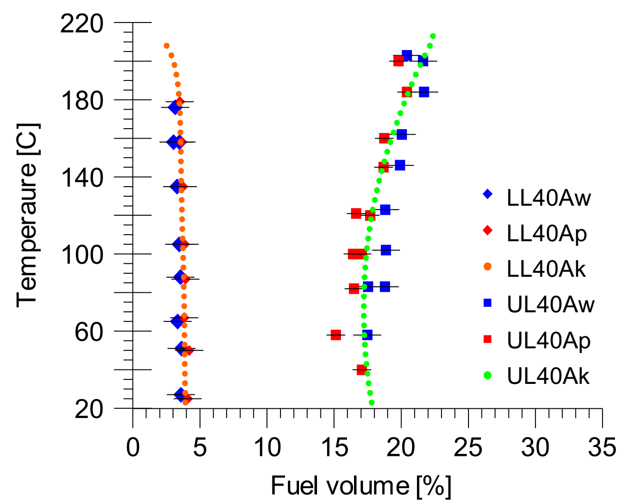


Fig. 14. Flammability limits at 40 kPa – anhydrous ethanol.

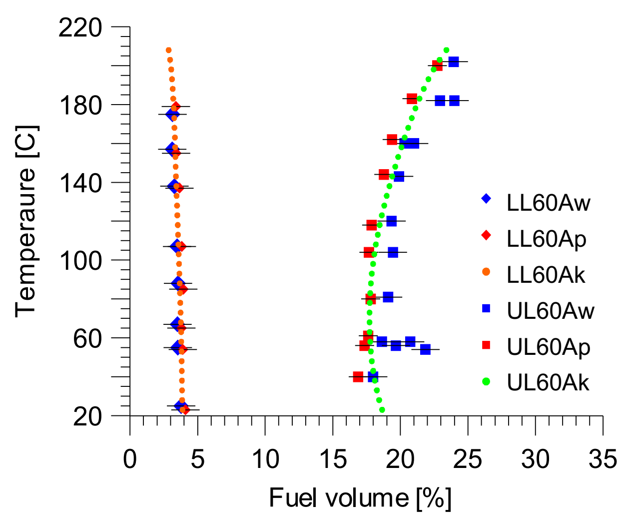


Fig. 15. Flammability limits at 60 kPa – anhydrous ethanol.

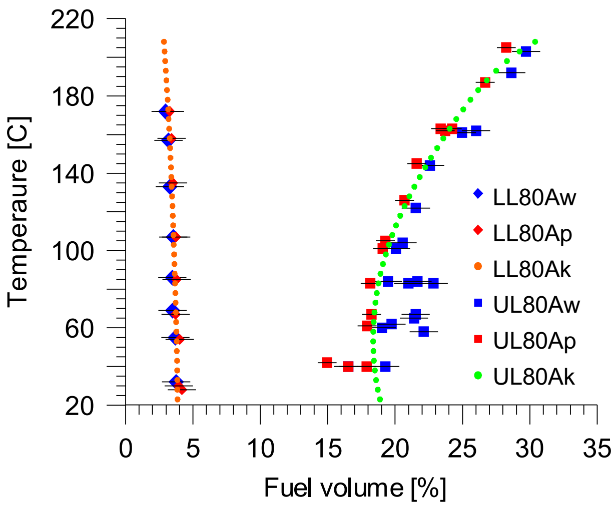


Fig. 16. Flammability limits at 80 kPa – anhydrous ethanol.

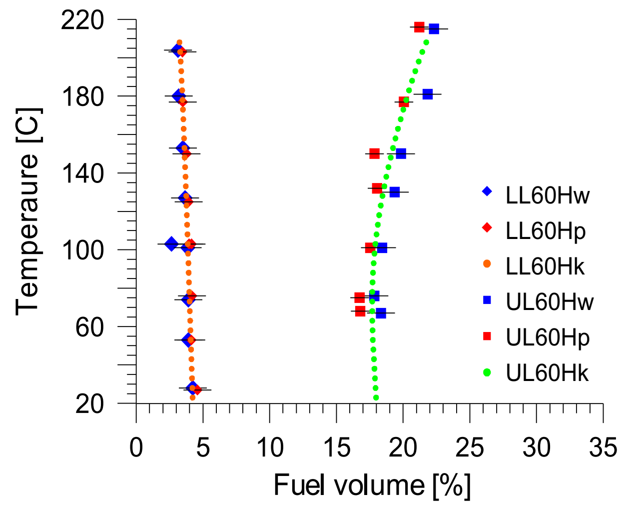


Fig. 19. Flammability limits at 60 kPa – hydrated ethanol.

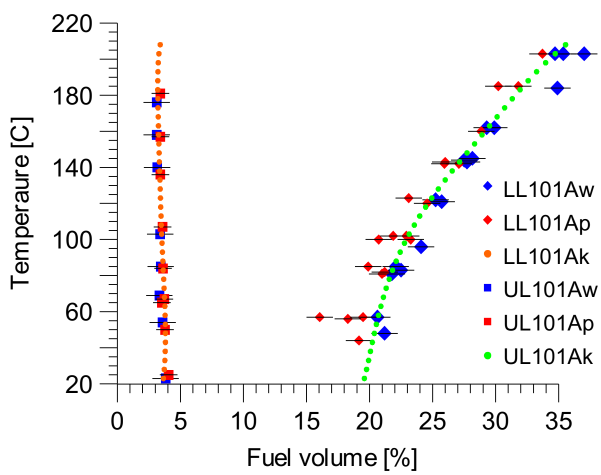


Fig. 17. Flammability limits at 101 kPa – anhydrous ethanol.

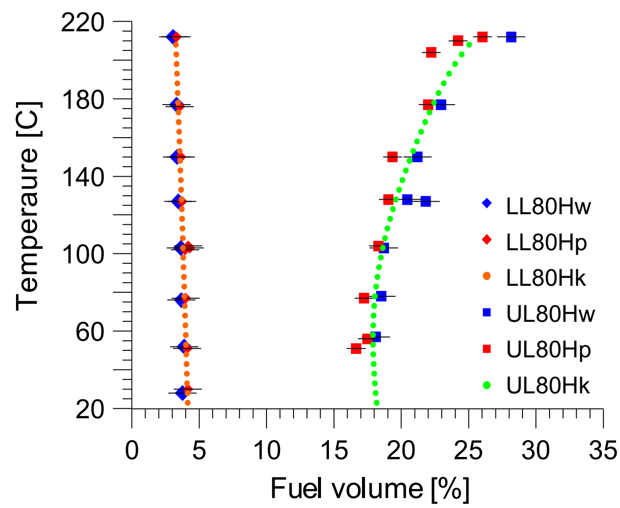


Fig. 20. Flammability limits at 80 kPa – hydrated ethanol.

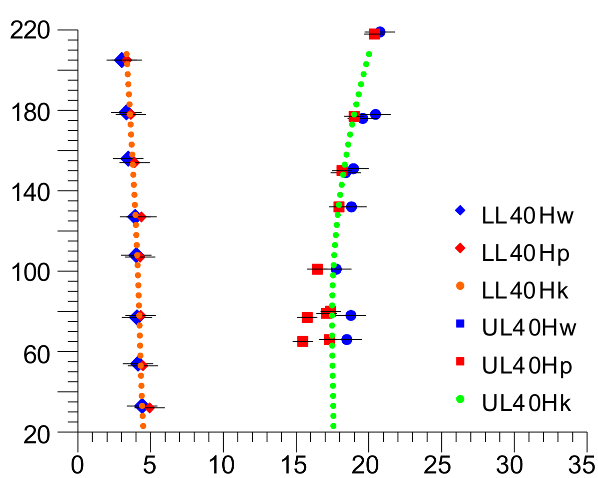


Fig. 18. Flammability limits at 40 kPa – hydrated ethanol.

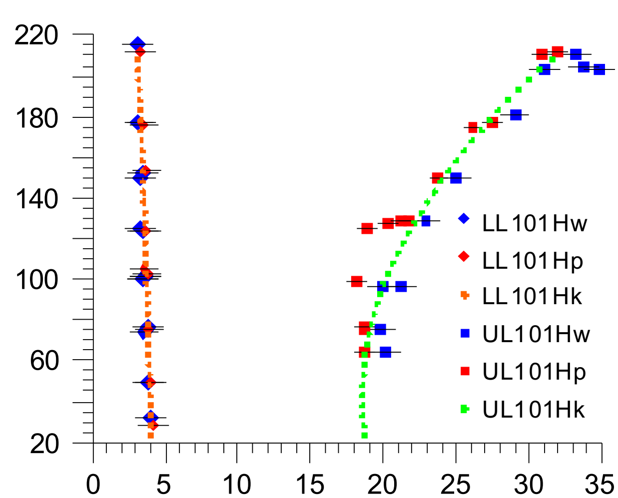


Fig. 21. Flammability limits at 101 kPa – hydrated ethanol.

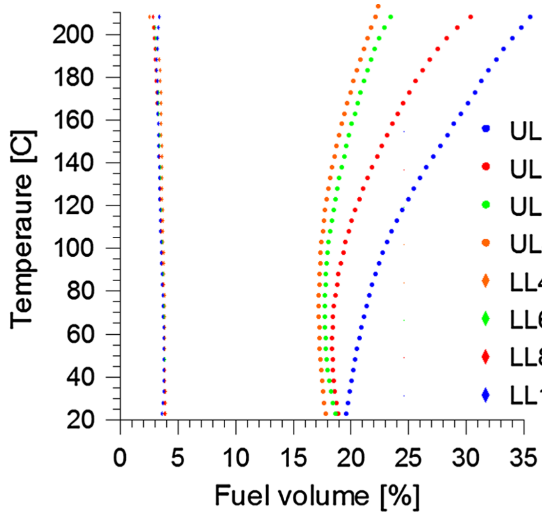


Fig. 22. Flammability limits of anhydrous ethanol-air mixtures, predicted by the developed algorithm.

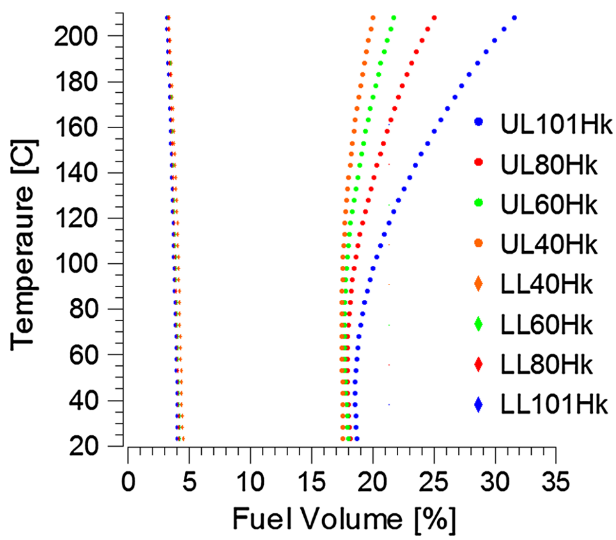


Fig. 23. Flammability limits of hydrated ethanol-air mixtures, predicted by the developed algorithm.

to being more sensitive to temperature, it is also very sensitive to water content in ethanol. This effect was expected since water vapor contained in the fuel is inert, thence narrowing the flammability zone.

In addition, the algorithm developed herein allows calculating response surfaces, in which two input parameters can be compared as a function of flammability. Figs. 25 and 26 show the response surfaces that link pressure and percentage of fuel volume to anhydrous ethanol-air and hydrated ethanol-air blends, respectively, at a temperature of 40 °C. In these response surfaces, an interval can be seen in which ethanol-air blends are flammable (region in red) and regions where they are non-flammable, either for presenting a poor blend (light blue zone) or a rich blend (dark blue zone).

Finally, It should be noted that although the curvature shape in the

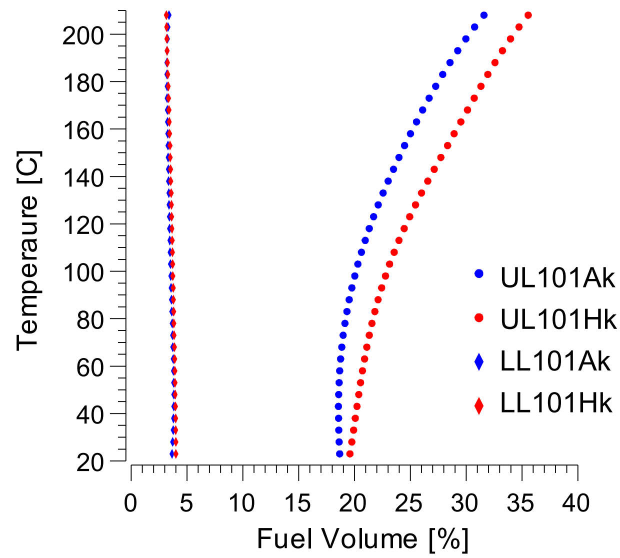


Fig. 24. Comparison of flammability limits for mixtures of anhydrous ethanol-air and hydrated ethanol-air, pressure of 101.3 kPa.

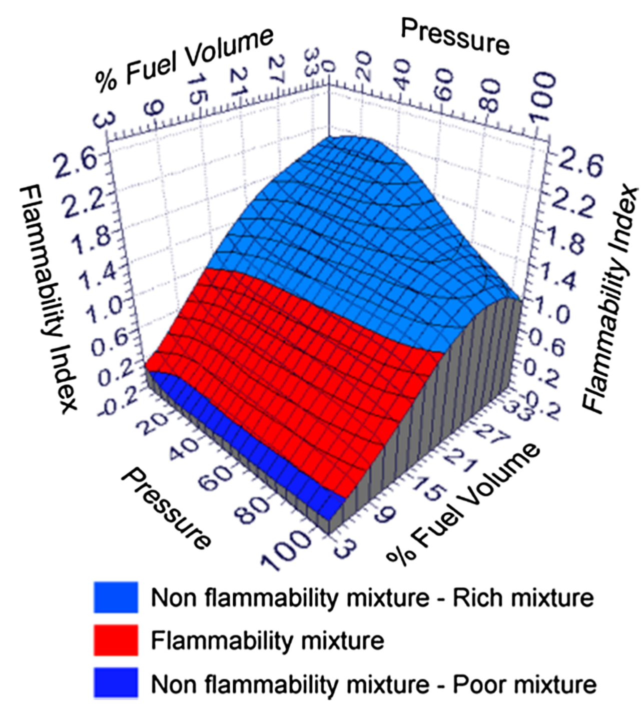


Fig. 25. Response surface to flammability index of anhydrous ethanol at 40 °C.

graphs showing Volume (ml) as a function of temperature (°C), especially in those showing upper flammability limit curves (e.g. Fig. 4), tends to decrease, and then the fuel volume increases at 100 °C with the same experimental data, but this time in a graph showing fuel volume

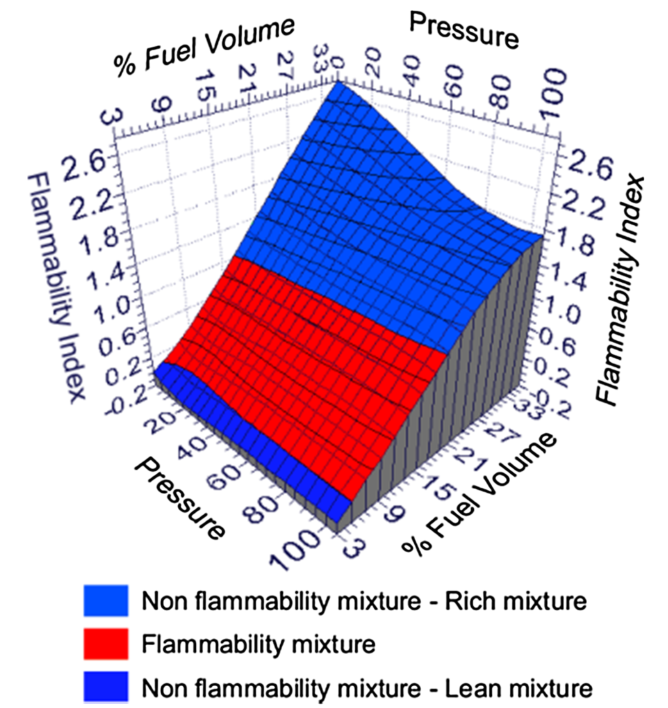


Fig. 26. Response surface to flammability of hydrated ethanol at a temperature of 40 °C.

(%) as a function of temperature (Fig. 16), the UFL curve trend is in agreement with data found in literature, once it is observed a constant increase in flammability limit as temperature rises. These graphs are fundamental since they do not take into account a fixed volume, but a generalization of any volume or space that may be occupied by a fuel. A similar behavior occurs in the rest of the figures.

7. Conclusion

This paper has proposed a technique to estimate flammability limits of ethanol-air fuel blends as a function of flammability index, which allows verifying not only whether the a blend is flammable or non-flammable, but also revealing how close it is to its flammability limits.

The presented model allows determining upper and lower flammability limits for air-ethanol blends. This prediction is based on establishing a flammability index that is obtained from pressure parameters in the test flask, air-ethanol blend temperature, fuel ratio in it and moisture concentration in ethanol. This index is established through interpolation techniques from experimental measurements. Prediction results from the model were compared with experimental measurements. The results lead to the conclusion that the developed model provides a very good approximation of required flammability limits.

The designed algorithm predicted the flammability condition of ethanol-air blends at temperatures ranging between 20 °C and 210 °C, pressure between 40 kPa and 101.3 kPa, ethanol moisture concentration at 0.5% and 8%, and ethanol volume percentages between 1% and 35%.

The algorithm is a valid tool to determine the flammability condition of ethanol-air blends due to achieving an amazing degree of accuracy and efficiency.

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