Robust modeling of gum formation in Brazilian ethanol-gasoline blends based on design of experiments and artificial neural networks approaches

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ABSTRACT

This work aims to predict gum formation, based on experimental data from controlled laboratory tests. The tested conditions follow design of experiments approaches and allow to measure either unwashed or washed gum contents for different ethanol concentration, storage temperature and aging period in homologation gasoline. The response surfaces arising from the obtained mathematical models are compared with models developed with Artificial Neural Networks (ANN) available in the literature. In addition to its ability to generalize the impact of the factors, the ANN models showed the best performance. It is important to highlight that the catalytic effect of ethanol in gum formation that was observed by visualizing the available data is predicted by the ANN model, but is not statistically proven in the polynomial model. Thus, it is possible to verify a catalytic effect for low concentrations of ethanol (around 20 vol%) after aging. Without aging or after storage at low temperature (approximately 20ºC), a simple dilution effect is observed. Such conclusions provide a robust unified interpretation to justify the discrepancy between some authors from the literature.

RESUMO

Este trabalho tem como objetivo predizer a formação da goma, com base em dados experimentais obtidos em testes laboratoriais controlados. As condições testadas seguem abordagens de planejamento de experimentos e permitem medir os teores de goma não lavada ou lavada para diferentes níveis experimentais para a concentração de etanol, temperatura e período de envelhecimento em gasolina de homologação. As superfícies de resposta decorrentes dos modelos matemáticos obtidos são comparadas com modelos obtidos com Redes Neurais Artificiais (RNA) disponíveis na literatura. Além de sua capacidade de generalizar o impacto dos fatores, os modelos de RNA apresentaram o melhor desempenho,. É importante destacar que o efeito catalítico do etanol na formação da goma que foi especulado pela visualização dos dados disponíveis é previsto pelo modelo RNA, mas não está comprovado estatisticamente no modelo polinomial. Assim, é possível verificar um efeito catalítico para baixas concentrações de etanol (em torno de 20 vol%) após o envelhecimento. Sem envelhecimento ou após armazenamento a baixa temperatura (aproximadamente 20ºC), observa-se um simples efeito de diluição. Tais conclusões fornecem uma interpretação unificada e robusta para justificar a discrepância entre alguns autores da literatura.

INTRODUCTION

It is widely recognized that road transport is a significant contributor to air pollution and greenhouse gas emissions [1]. Stringent emission regulations have been implemented, leading to the reduction of such emissions. Numerous studies have been conducted to measure and predict exhaust emissions to mitigate these impacts. In addition to advancements in internal combustion engine design and operation, researchers have also explored the suitability of alternative fuels. Ethanol emerges as a promising option for mitigating emissions [2]. In Brazil, ethanol is mostly derived from sugarcane and is available as either hydrous ethanol (E100) or anhydrous ethanol blended with gasoline at volumetric percentages of 27% vol (E27) in regular gasoline and 25% vol (E25) in premium gasoline [3]. Moreover, several countries, including the United States and Brazil, allow the use of flex-fuel vehicles capable of running on a wide range of ethanol concentrations.

However, in spark-ignition engines, fossil gasoline, a complex blend of paraffinic, naphthenic, olefinic, and aromatic hydrocarbons, continues to be the primary fuel. It also contains a low concentration of oxygenates and traces of sulfur, nitrogen, and metals, which introduce instability to the fuel. During storage, the hydrocarbons react with the oxygen present in the atmosphere, resulting in the formation of a resinous, polymeric, and non-volatile substance known as gum [4-6]. The formation of gum leads to alterations in the physicochemical properties of the fuel blend, including increased fuel density, distillation temperature, aromatics, and oxygen concentration, while the concentration of olefins decreases. The deposition of gum in the injection system and combustion chamber adversely affects vehicle drivability, performance, and durability, while also contributing to increased exhaust gas emissions [5,7–10]. Washed gum, as defined by ASTM D381, refers to the heptane-insoluble portion of the evaporation residue of motor gasoline, while unwashed gum denotes the gum formed before its extraction by the solvent [11]. Brazilian regulations specify a maximum washed gum content of 5.0 mg per 100 mL of fuel for commercial gasoline and 4.0 mg per 100 mL of fuel for homologation gasoline.

The literature provides a consensus on some factors contributing to gum formation, namely increased temperature, prolonged storage time, and the presence of transition metal ions in the solution [4,5]. Several authors have conducted experimental studies to investigate the role of ethanol in gum formation within fuel blends. D'Ornellas [12] observed an enhanced gum formation in the presence of ethanol across all storage periods examined. Pereira et al. [8], on the other hand, concluded that ethanol content only had a dilution effect in the absence of aging. Pradelle et al. [6,13,14] examined various variables in gasoline-ethanol mixtures, including regular gasoline and additive-enhanced gasoline with a complex package of antioxidant, anticorrosive, dispersant, and detergent components. They found that the maximum gum formation occurred with a moderate ethanol content, but the behavior strongly depended on the temperature and duration of aging. In a European context, Jeczmionek et al. [15] investigated the unwashed, existing, and potential gum content in gasoline blends containing 15%, 20%, and 25% vol of ethanol, respectively. Their findings indicated an increase in gum content with higher ethanol concentrations.

Artificial neural networks (ANN) serve as powerful tools in this context. Neural networks consist of interconnected neurons arranged in layers, including input and output layers, as well as multiple hidden layers. These connections play a crucial role in adjusting weights and biases during network training. Once the training process is complete, the network becomes capable of predictive simulation within the range of the inputs provided in the training data [16]. Carvalho et al. [17] developed artificial neural networks and observed higher reliability than the model with a linear polynomial equation incorporating rectangular interaction terms. They constructed a dataset using original experimental data and values from the literature to conduct their analysis. Focusing on the unwashed gum, they observed errors as low as 2% for the majority of the training of the ANN approach, while for testing it was lower than 0.5 %. Analyzing washed gum results, these errors are lower for both training and testing (lower than 1.5% and 0.2%, respectively).

This study aims to investigate the characteristics of homologation gasoline with 22% vol of anhydrous ethanol using central composite design approaches (in that case, a 3^k design) and compare the obtained results with the predictive outcomes from the existing ANN model in the literature. Furthermore, these findings contribute to enhancing the understanding of gum content formation concerning factors such as ethanol concentration, temperature, aging duration, fuel type (regular and homologation), and composition (olefin and aromatic contents). By doing so, it is aimed to strengthen a comprehensive understanding that may shed light on the contrasting conclusions observed in previous literature

METHODOLOGY

MATERIAL AND METHOD

The fuel has a typical profile of Brazilian homologation gasoline, as shown in Table 1.

Table 1: Characterization of the investigated gasoline.

Property	Method	Brazilian Specification [a]	Value
Color (-)	Visual	Colorless to slightly yellow	Colorless to slightly yellow
Visual Aspect (-)	ASTM D4176	Limpid and exempt of impurities	Limpid and exempt of impurities
Ethanol content (% vol)	NBR 13992	1 (max)	< 1
Motor Octane Number, MON, with 22% vol of ethanol (-)	ASTM D2700	82.0 (min)	87.1
Research Octane Number, MON, with 22% vol of ethanol (-)	ASTM D2699	93.0 (min)	99.4
Induction period at 100°C (min), with 22% vol of ethanol	ASTM D525	- / 1000 (min)	> 1000
Density at 20°C (kg/m^3)	ASTM D4052	720.0 to 758.00	727.2
Distillation temperature Initial boiling point (ºC) 10% evaporated (°C) 50% evaporated (°C) 90% evaporated (°C) Final boiling point (°C) Distillation residue (% vol)	ASTM D86	 30.0 to 40.0 45.0 to 60.0 90.0 to 110.0 149.0 to 170.0 195.0 to.213.0 2.0 (max)	 31.9 51.1 98.5 157.7 195.7 1.3
Vapor pressure at 37,8°C (kPa), with 22% vol of ethanol	ASTM D5191	54.0 to 64.0	61.0
Washed gum content (mg/100mL)	ASTM D381	4.0 (max)	1.0
Corrosiveness to Copper, 3h at 50°C (-)	ASTM D130	1 (max)	1A
Sulfur content (mg/kg)	ASTM D7039	50 (max)	28.0
Benzene content (% vol)	ASTM 3606	1.0 (max)	0.21
Aromatic (% vol) Olefinic (% vol) Saturated (% vol)	N 2377	35 (max) 15 (max) -	30.78 9.84 59.38

The experimental procedure started with a thorough cleaning of the glassware using alcohol-free gasoline and subsequent drying in a dust-free environment. Each vessel was filled with 150 mL of fuel samples blended with varying ethanol content. To prevent fuel evaporation, the vessels were tightly sealed with caps and submerged in water baths at specified temperatures for varying durations, following the 3^k design of experiments approach. In this study, a total of 144 data points was collected, with four measurements conducted under the same conditions to ensure repeatability. The samples consisted of homologation gasoline blended with anhydrous ethanol, with ethanol concentrations ranging from 0% to 70% vol. The testing was carried out at a constant temperature of 20°C, 30°C, or 40°C over 150 days. Two central composite designs were employed, with one focusing on ethanol concentrations up to 30% vol and the other covering higher ethanol concentrations ranging from 30% to 70% vol, as shown in Figure 1 [17,18].

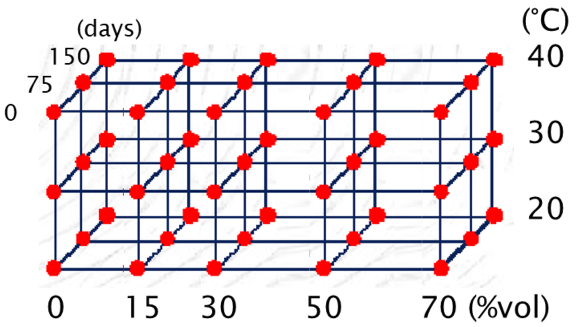


Figure 1: Design of experiments tests matrix for unwashed and washed gum data collection

NUMERICAL MODEL

The amounts of unwashed and washed gum were modeled by a second-order polynomial with interaction term of the same order, represented by the following equation:

$$Y = b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_{11}X_1^2 + b_{22}X_2^2$$

$$+b_{33}X_3^2 + b_{12}X_1X_2 + b_{13}X_1X_3 + b_{23}X_2X_3 + \varepsilon \quad (1)$$

Y is the experimental response; X_1, X_2, and X_3 are the reduced independent factors (or variables) whose impact must be assessed; b_0 is a constant term; b_1, b_2, and b_3 are the coefficients associated to the linear contribution of each factor; b_{11}, b_{22}, and b_{33} are the coefficients of the quadratic contribution of each factor; b_{12}, b_{13} and b_{23} are the coefficients of the interaction (or rectangular) terms between two factors and ε is the residue. Except for the constant term, which represents the average value of the experiments conducted at the center of the experimental domain, a positive coefficient indicates that an increase in the corresponding variable promotes the formation, while a negative coefficient indicates an inhibitory effect. Null or very low coefficient values, compared to other parameters, indicate that the effect has no or insignificant influence on the process [6].

The coefficients of the polynomial equation are determined using the least square method to minimize the sum of squared errors. To assess the model's significance statistically, analysis of variance (ANOVA), t-student tests, Pareto diagrams, coefficient of determination (R^2), and sum of squared errors (SSE) were employed. The response surface generated by this model is then compared to the results obtained from the ANN models developed in Carvalho et al. [17].

RESULTS AND DISCUSSION

ASSESSMENT OF THE MATHEMATICAL MODELS

The coefficients of each polynomial model can be found in Table 2.

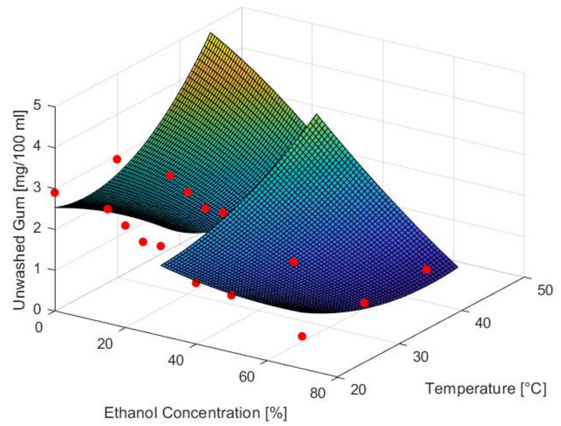
Table 2: Coefficient of the parameters for the models in term of real variables.

Range of ethanol content	0-30% vol		30-70% vol	
Factor	Unwashed gum	Washed gum	Unwashed gum	Washed gum
Mean/Interc.	**6.154012**	**2.284877**	1.819122	0.855110
(1) Ethanol (L)	0.079753	-0.025432	0.013537	0.019576
Ethanol (Q)	-0.000560	**-0.001317**	0.000304	**0.000177**
(2) Temperature(L)	**-0.301481**	0.044907	-0.007087	0.045264
Temperature(Q)	**0.006019**	-0.000852	0.002341	0.000714
(3) Duration (L)	**-0.027926**	**-0.013593**	0.000400	**0.003299**
Duration (Q)	-0.000027	0.000024	-0.000029	0.000013
1L by 2L	**-0.002870**	0.000593	**-0.002613**	**0.000251**
1L by 3L	**-0.000491**	**0.000356**	**-0.000179**	**0.000033**
2L by 3L	**0.001959**	**0.000241**	**0.000728**	**0.000066**
R^2	0.88879	0.63807	0.85571	0.83391
R^2 adjusted	0.87470	0.59219	0.83793	0.81344

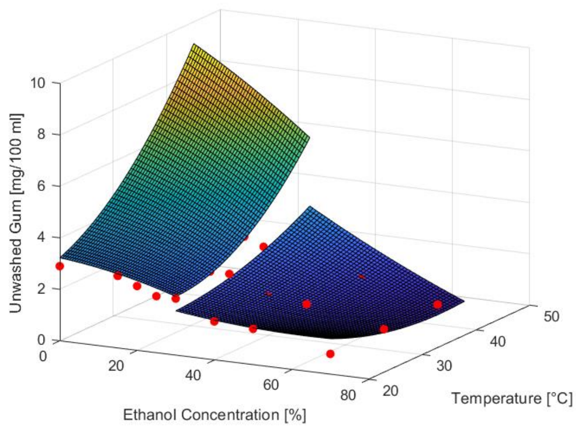
Legend: (L) – linear; (Q) – quadratic

In Table 2, significant coefficients, determined through a t-student test with a p-value equal to 5%, are indicated in red and bold. It is noteworthy that the coefficients of interest always involve the interaction terms between the parameters, highlighting the complexity of the studied phenomena. Regarding the content of washed gum, regardless of the ethanol range, the quadratic term associated with ethanol content and the linear contribution of aging duration exhibit significance. On the other hand, for unwashed gum content, the behavior differs between the ethanol dilution ranges. Up to 30% vol, the temperature exerts a linear and quadratic impact, along with the linear contribution of aging duration. For higher ethanol content, only interaction terms come into play. These findings are further confirmed by the ANOVA calculation.

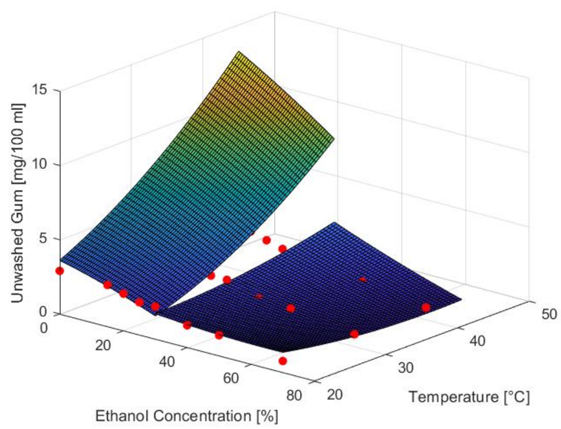
Predicted and experimental unwashed and washed gum contents for different aging durations and temperature using the polynomial models are shown in Figures 2 and 3.



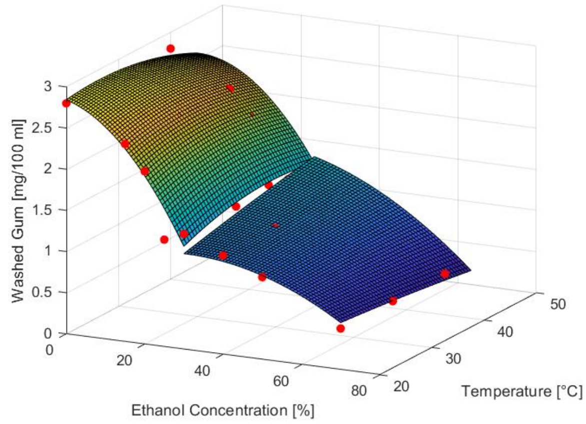
(a) Unwashed gum – 0 day



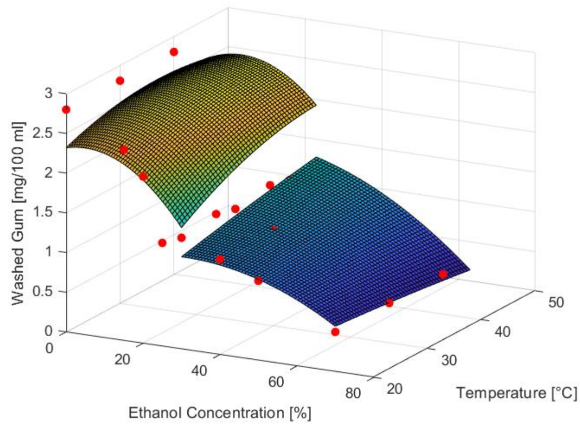
(b) Unwashed gum – 75 days



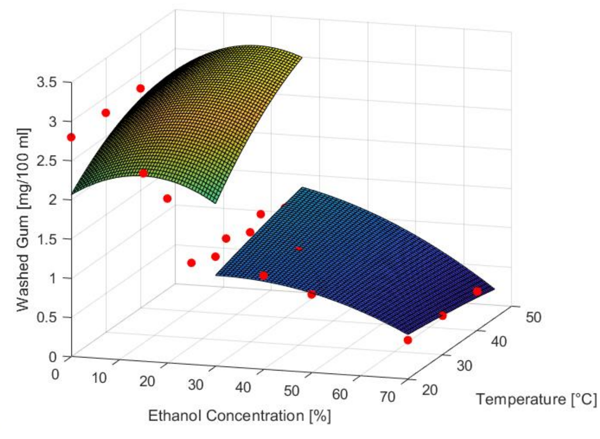
(c) Unwashed gum – 150 days



(d) Washed gum – 0 day

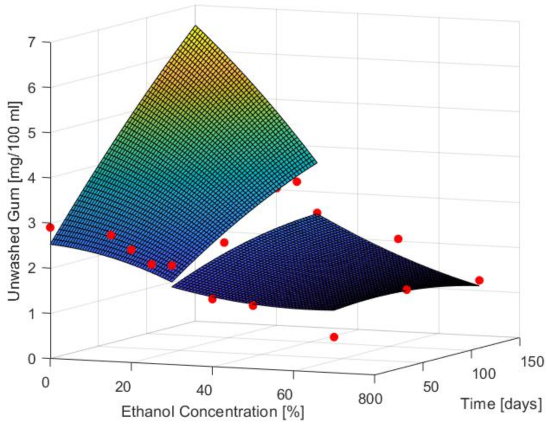


(e) Washed gum – 75 days

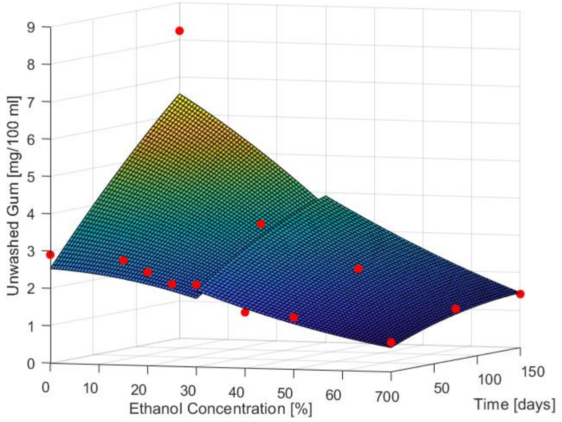


(f) Washed gum – 150 days

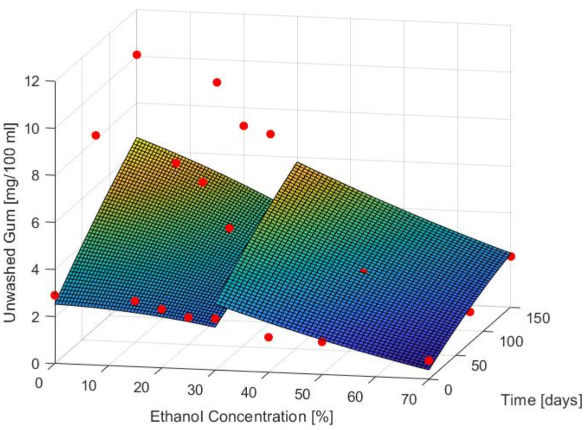
Figure 2: Predicted and experimental unwashed and washed gum contents for different aging durations using the polynomial models.



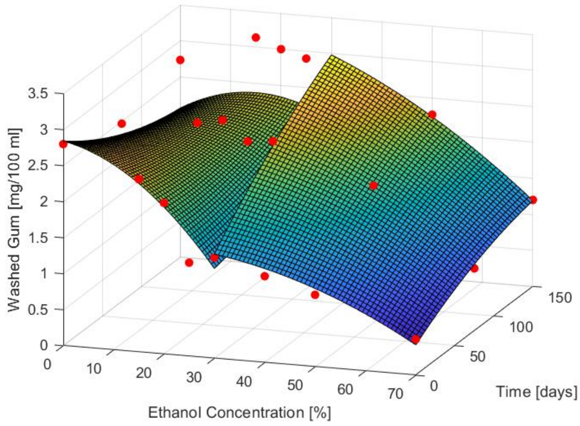
(a) Unwashed gum – 20ºC



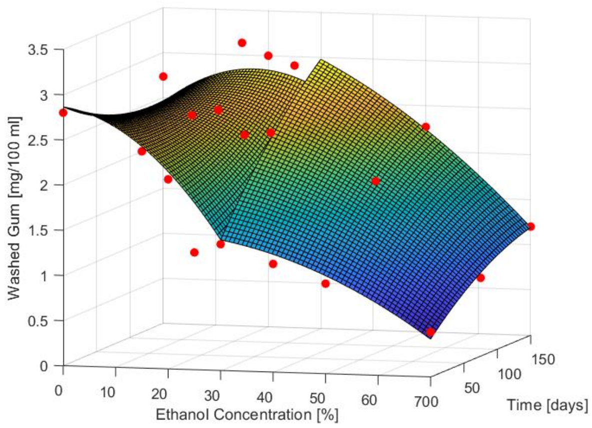
(b) Unwashed gum – 30ºC



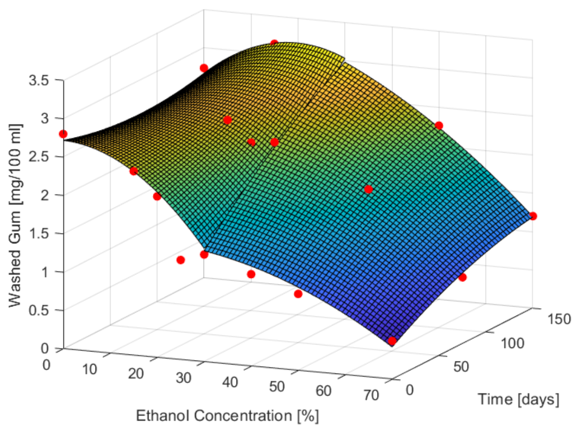
(c) Unwashed gum – 40ºC



(d) Washed gum – 20ºC



(e) Washed gum – 30ºC



(f) Washed gum – 40ºC

Figure 3: Predicted and experimental unwashed and washed gum contents for different temperatures using the polynomial models.

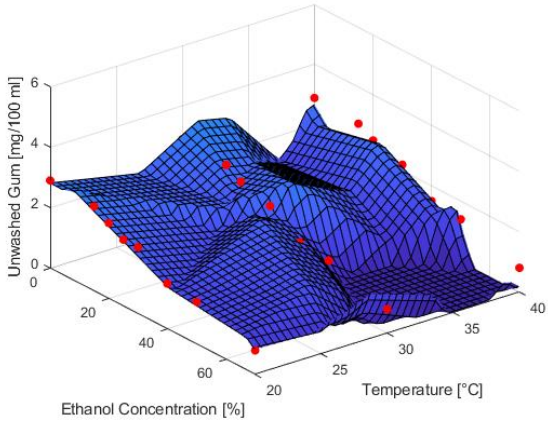
The Figure 2 enables a straightforward comparison between various aging scenarios, illustrating the contrasting behavior during winter (representing the lowest temperature) and summer (representing the highest temperature) seasons in specific regions of Brazil and worldwide, respectively. The Figure 3 provides an opportunity to assess the influence of gum formation kinetics during winter (lowest temperature) and summer (highest temperature) seasons, respectively, for a given fuel formulation, considering the impact of the time.

In the Figures 2 and 3, it can be observed that the models obtained in the range 0-30% vol and 30-70% vol of ethanol showed in most of the surfaces some significant discontinuity in modelling the behavior at 30% vol of alcohol in the blend. This behavior is a strong limitation for quantitative comparisons in the experimental domain. Despite the apparent influence of ethanol in the experimental results of unwashed gum contents, it is surprising that ethanol shows no significant effect according to these statistical analyses. However, it is important to note that the coefficient of determination for all models is relatively low, considering reference values for engineering applications. This justify the lowest reliability of the model for washed gum content in the range of 0 to 30% vol of ethanol. Nonetheless, as the homologation gasoline is more resistant to oxidation, the low level of gum production and consequently the reduced range of variation of the simulated characteristic reduce the ability of the design of experiment to distinguish noise from variable impact. Thus, while the models may be useful for qualitative assessments, it can be concluded that the design of experiment approach may not be robust enough to quantitatively evaluate the impact of ethanol and storage conditions in the oxidation of these blends. As a result, a more sophisticated predictive model, such as an Artificial Neural Network (ANN), must be employed to achieve more reliable predictions of gum content in the blends.

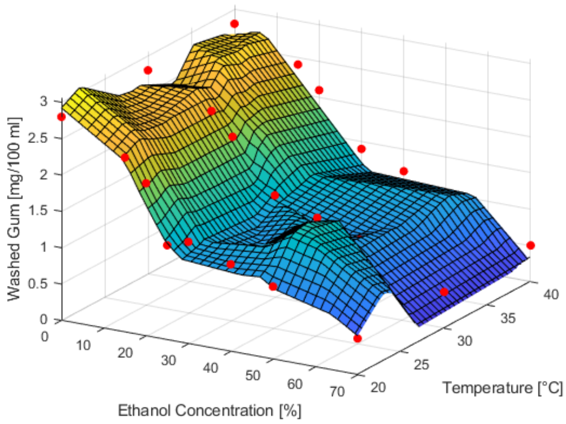
COMPARISON WITH PREVIOUS MODELS AND DISCUSSION ON GUM FORMATION'S BEHAVIOUR

Using the model developed by Carvalho et al. [17], the predicted behavior for unwashed and washed gums is depicted in Figure 4, keeping the aging time equal to 0, 75 and 150 days respectively.

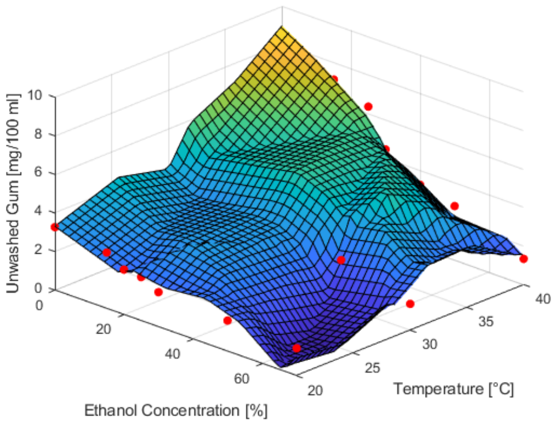
Observing Figure 4, it can be observed that the discontinuity problem at 30 vol% is solved and the range of predicted values is more well behaved than with the polynomial, with lowest residues. Nonetheless, it is evident that the model fails to accurately predict the formation of unwashed gum at high concentrations of ethanol and elevated temperatures without aging. In such cases, the model erroneously predicts a negative value for the gum content, which is known to be impossible. However, for storage periods of 75 and 150 days, a consistent dilution effect can be observed, with the gum content decreasing as the ethanol concentration increases across all temperature conditions. Despite this prominent trend, the curve exhibits some instabilities, where the gum content does not consistently decrease with the addition of ethanol. Nevertheless, across all three cases, it is evident that the addition of ethanol to the blend effectively reduces the content of unwashed gum. Notably, Figure 4 also reveals the occurrence of a local maximum in the washed gum content at lower ethanol concentrations (around 10%) and high temperatures. This behavior indicates the potential for a catalytic effect in the formation of washed gum, followed by subsequent dilution due to the addition of ethanol to the blend. In contrast, in the case of no aging, an inhibitory effect is observed at concentrations exceeding 20% of ethanol, leading to a subsequent dilution.



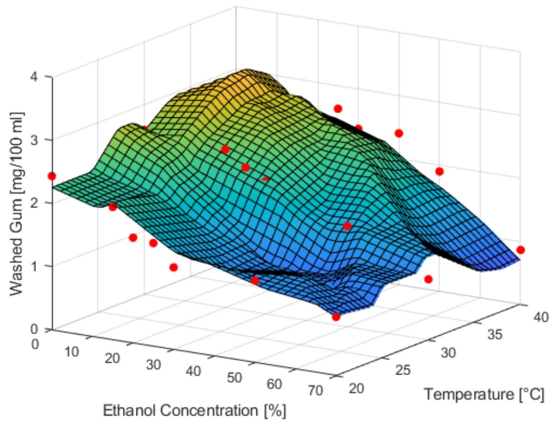
(a) Unwashed gum – 0 day



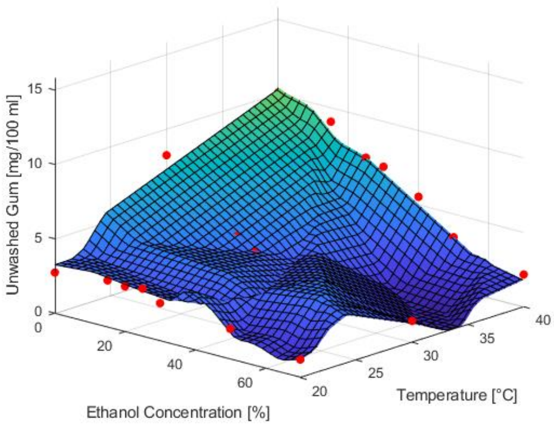
(d) Washed gum – 0 day



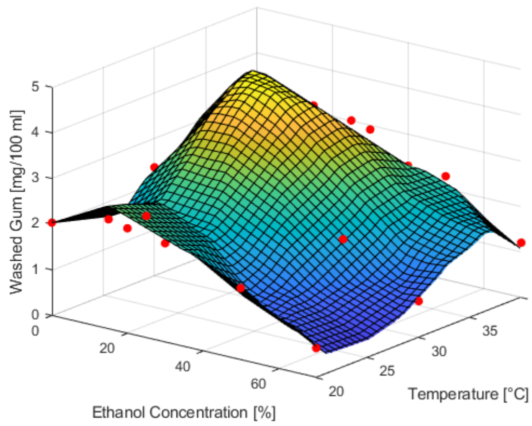
(b) Unwashed gum – 75 days



(e) Washed gum – 75 days



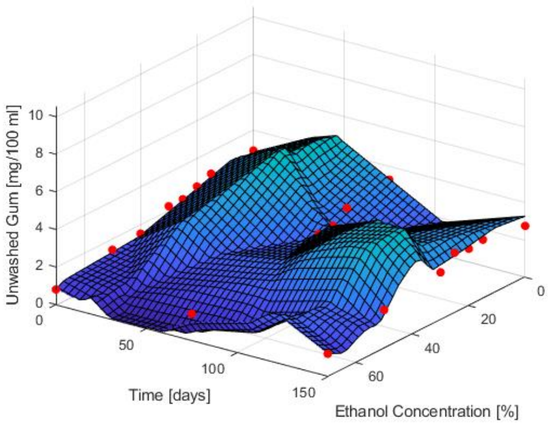
(c) Unwashed gum – 150 days



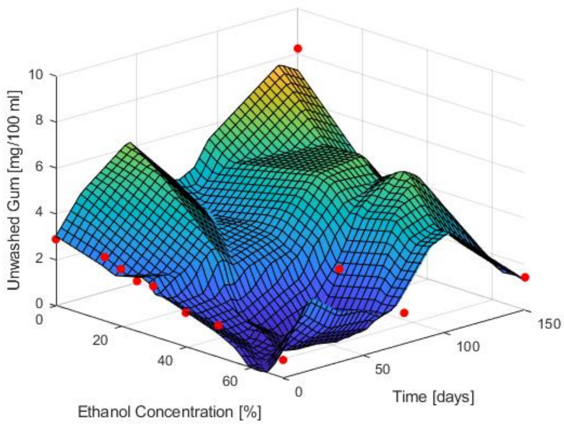
(f) Washed gum – 150 days

Figure 4: Predicted and experimental unwashed and washed gum contents for different aging durations using the ANN models.

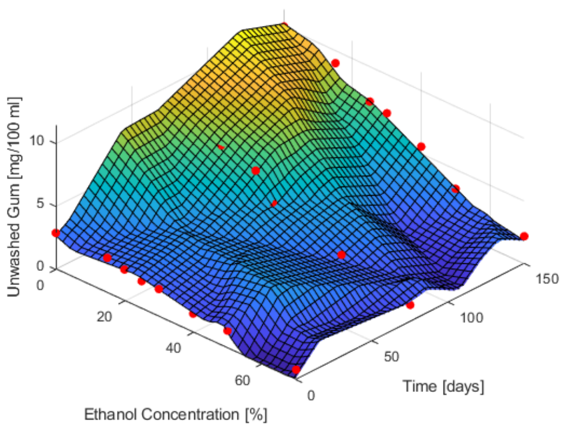
Moving on to Figure 5, it showcases the results for unwashed and washed gum content while maintaining a constant temperature (20, 30 and 40ºC, respectively).



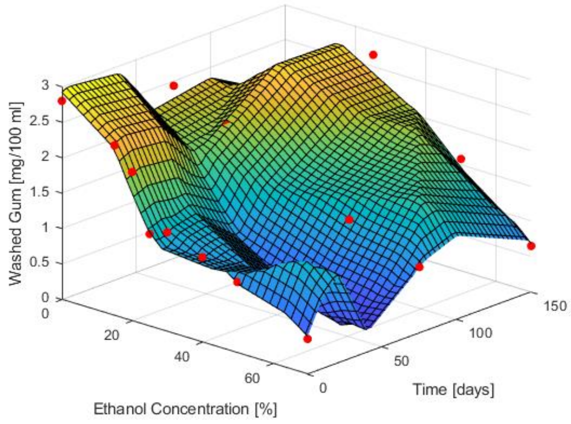
(a) Unwashed gum – 20ºC



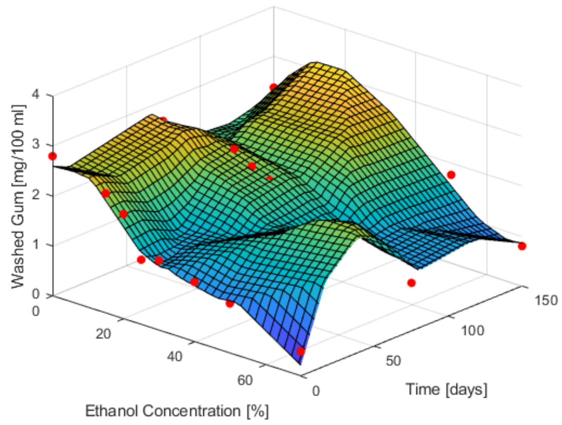
(b) Unwashed gum – 30ºC



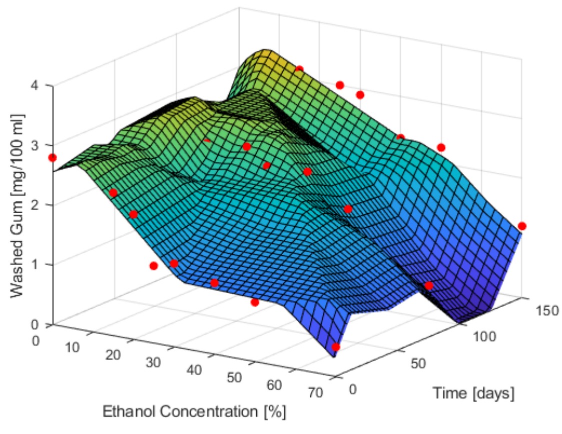
(c) Unwashed gum – 40ºC



(d) Washed gum – 20ºC



(e) Washed gum – 30ºC



(f) Washed gum – 40ºC

Figure 5: Predicted and experimental unwashed and washed gum contents for different temperatures using the ANN models.

A significant decrease in unwashed gum content can be observed as the ethanol concentration increases, consistent with findings in the literature [14]. However, the model may exhibit unexpected behaviors under certain conditions. It is not anticipated to observe a catalytic effect resulting from the addition of ethanol to the blend. Nevertheless, such behavior is evident in specific circumstances, particularly at higher aging durations (between 75 and 150 days) and temperatures of 20 and 30 °C. This highlights a limitation of the model and suggests areas for further exploration to enhance its predictive capability. Examining Figure 5, it can be observed once again that the addition of ethanol to the blend effectively reduces the content of washed gum. At both 20 and 30 °C, the model indicates the presence of a catalytic effect between 75 and 150 days, followed by a subsequent dilution. However, the same effect is not as pronounced at 40 °C, which could be attributed to factors such as ethanol evaporation or the increased activation of reactions in the gum formation mechanism, thereby mitigating its impact.

CONCLUSION

Two different approaches were compared for the prediction of the washed and unwashed gum contents in gasoline-ethanol blends: a polynomial equation derived from a central composite design of experiments (3^k) with three variables and an Artificial Neural Network (ANN) model with six variables from the literature. The two models generated from the 3^k design of experiments approach exhibited significant discontinuity in modeling the behavior at 30% ethanol volume in the blend, which poses a limitation for quantitative comparisons within the experimental domain. The analysis of the polynomial model's effect value does not provide conclusive evidence with a statistical robustness regarding the impact of ethanol. Additionally, it is important to note that the coefficient of determination for all polynomial models is relatively low when considering reference values for engineering applications. Consequently, the ANN models demonstrate higher performance due to their ability to establish more complex connections and retrieve the desired results.

Hence, the selected ANN models were able to accurately predict the experimental data for both gum formations with satisfactory reliability and at a limited computational cost. A main conclusion of this study is that the model can predict the catalytic effect of gum formation, as speculated by visualizing the available data. Regarding the impact of ethanol, it is possible to observe catalytic effects on washed gum formation at low concentrations of ethanol mixed into the gasoline after aging. However, without aging or after storage at low temperatures, ethanol has a dilution effect. These conclusions align with previous literature and provide an explanation for the observations made by other authors, who reported either catalytic or dilution effects.

As a consequence, the surfaces indicate potential regions where washed gum formation is enhanced, suggesting the possibility of conducting new experiments to confirm this behavior and further enhance the model's capabilities. However, it should be acknowledged that the model exhibits shortcomings when extrapolating into regions with no experimental data, leading to unreliable or even mistaken prediction of the blend's behavior.

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