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EXPERIMENTAL DESIGN TO STUDY CORROSIVITY OF DIESEL-BIODIESEL MIXTURES ON MICROALLOYED STEEL

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ABSTRACT

Material selection is a crucial part of industrial process and the analysis of the effects of operational variables on the corrosion plays an important role. The present study was undertaken to evaluate the corrosion of a microalloyed steel in contact whit diesel-biodiesel mixtures. A design of experiments, the analysis of variance (ANOVA) and surface response methodology (SRM) were used in order to evaluate the corrosivity of the fuel mixtures in contact with steel. The effect of biodiesel concentration, addition of multifunctional additive and immersion time were the evaluated factors to research the corrosion behavior of microalloyed API X70 steel. The response used in the exploitation of the design was the corrosion rate; it was assessed through gravimetric measurements on samples. The results of the gravimetric tests showed that the immersion time has the most significant effect on corrosion resistance, following of additive and biodiesel concentration. ANOVA and SRM revealed a decrease in the corrosion rate with immersion time, effect that suggests the formation of a protective layer on the metallic surface. It can also be concluded that the use of lignin promoted the reduction of corrosive processes, especially when mixed with biodiesel; this result was corroborated by microscopy analysis.

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INTRODUCTION

Metallic materials are among the most widely used class of materials in the industrial sector. Much of this application is due to the search for materials that have a specific combination of properties, such as mechanical strength, toughness and corrosion resistance. Microalloyed steels exhibit some of these characteristics, promoting wide use in the oil, naval, civil construction and transport industries. Ferrous alloys are the most used materials for the manufacture of land vehicles and this scenario should not change in the coming years, especially considering the acceptable benefit–cost ratio. Ferrous alloys known as HSLA (High-strength low-alloy) have properties such as high mechanical strength and tenacity. These alloys consist of low carbon steels or carbon-manganese steels, to which small amounts (generally less than 0.1%) of aluminum, vanadium, titanium or niobium are added. These materials can also present copper, silicon, molybdenum, nickel or chromium as alloying elements, which generate high resistance after heat treatment (Patel et al, 2005). Another characteristic of microalloyed steels is the greater ductility or degree of deformation that they support until the moment of fracture. This quality expands the possibility of a further reduction of the structural mass in vehicles, allowing the reduction of fuel consumption, which is particularly interesting for applications in diesel vehicles and diesel blends. Several papers in the literature show the application of experimental design to the study of the interaction of metals and alloys in contact with diesel and blends with biodiesel. As well as research that statistically assesses how contaminants (impurities) contained in these fuels influence corrosivity, oxidation stability, thermal stability and storage time, changing the final quality of the product and the material in contact. Jain and Sharma (2012), report results of the study of the effect of metallic contaminants on the oxidation, thermal stability and storage of Jatropha curcas biodiesel using the response surface methodology (RSM). The study aimed to obtain correlations to predict the ideal concentration of antioxidants to provide stability to fuel oxidation. Biodiesel samples with different concentrations of pyrogallol in contact with transition metals (Fe, Ni, Mn, Co and Cu) were studied. The induction period (IP) and thermogravimetric analysis (TGA) were used to evaluate the thermal behavior of the biodiesel samples. A comparison between the experimental values and those predicted by the correlation shows that all data points are within ±10% deviation lines from the experimental results. From the experiments, it was found that if the metal concentration was zero, 200 ppm, of pyrogallol are enough to make the biodiesel stable for 6 months. If the metal (Fe) concentration was 2 ppm or more, 800 ppm pyrogallol are enough to make the biodiesel stable for 5.5 months. The factors influencing corrosion in diesel-service underground storage tanks was investigated by researchers of the EPA (U.S. Environmental Protection Agency) in 2016. The objective of the research was to understand the potential risks to human health and the environment caused by the problem of corrosion in tanks that store diesel fuel, considering that this occurs both in steel tanks and in most internal metal components. ANOVA was conducted to determine if the amount of acids correlated with the presence of corrosion in metallic materials, together with other variables such as water content, biodiesel, sulfur, alcohol, metals and other physical chemical parameters were monitored. The study did not identify any statistically significant predictors, however it appears that tanks with cleaner, drier fuel may be less likely to be associated with serious corrosion problems. According to statistical analysis, particle and water contents in fuel were the closest to being statistically significant predictors for metal corrosion (U.S. EPA, 2016).

Dwivedi (2018), used the Box-Behnken factorial design to optimize the storage period of karanja oil biodiesel in contact with various metals and with the addition of different antioxidants for a period of 6 months. The best result for the stability to biodiesel oxidation was 8.34, 8.40 and 8.20 h for the interaction with iron (Fe), aluminum (Al) and zinc (Zn) respectively. Under these conditions, after 6 months, metal concentrations of 1.10, 0.85 and 1.45 ppm were observed in biodiesel, respectively. The authors demonstrated the need to use an antioxidant to prevent the fuel from deteriorating. Komariah (2017), observed the behavior of materials used in palm biodiesel storage tanks (glass, high density polyethylene and stainless steel). Fuel properties were monitored for 12 weeks. The tanks were placed in a confined indoor space with a temperature range of 27–34 °C. The response variables were density, viscosity and water content. During the storage period, the average density of B20 changed slightly in all tanks, while the viscosity tended to increase drastically, especially in polymeric tanks. Based on physicochemical analyses, the steel storage tank is making minimal changes in fuel properties.
High corrosivity in samples with 20% biodiesel in diesel blends of 5000 ppm sulfur on microalloyed API X70 steel was observed by Oliveira (2017). Recently, Tavares (2020), evaluated the corrosion resistance of an API X70 microalloyed steel in contact with rapeseed biodiesel and obtained the linear regression model through an analysis of variance (ANOVA). To investigate the influence of acidity on the corrosion resistance of steel, samples of pure biodiesel and biodiesel with 1, 3 and 10% oleic acid were added. The corrosion resistance of steels was determined using measures of mass loss, corrosion rate and conductivity. The results suggest that canola biodiesel creates a protective layer called "biofilm" under the microalloyed steel after the first weeks of contact, causing corrosion to decrease and a reduction in acidity to be observed. After 30 days of contact, a significant increase in corrosion rates can be observed, probably caused by the rupture of the biofilm. In another study, the authors managed to reduce the corrosive effect of a mixture of high sulfur and soybean biodiesel on microalloyed API X70 steel with the additive produced from sugarcane bagasse (Tapanes, 2020).

Considering these statistical studies, it is observed that there are still uncertainties about the behavior of biodiesel and biodiesel/diesel mixtures in contact with metallic materials; the need for scientifically planned evaluations is evident. Evaluations that statistically show the performance of these fuels, their corrosivity and stability in a fuel-metal system, which will enable the definition of the necessary operating conditions for the viable use of microalloyed steels in the industry. In this context, this article aims to apply statistical techniques to evaluate the interaction of microalloyed API X70 steel with diesel/biodiesel mixtures of different composition. The study made it possible to verify and statistically prove the applicability of the material in the oil industry.

MATERIALS AND METHODS

Experimental Procedure : The experiment was set up in the laboratory where API X70 steel specimens were used. The steel used has not yet undergone the lamination process, which provides the necessary mechanical properties, even so, the absence of this treatment does not interfere with the assessment of corrosion resistance. Procedures for specimens' preparation and the weight loss tests were performed following ASTM G1 and ASTM G31 methods. The prepared specimens were stored in desiccators to avoid atmospheric corrosion. Table 1 shows the chemical composition of mild steel sample used in this study. The fuel mixtures were prepared with diesel, soybean biodiesel and Calcium Lignosulfonate. The diesel was obtained from the local fuel station, this fuel meets the Marine diesel oil A (DMA) specifications described in ANP Resolution Nº 52 de 29/12/2010. The soybean biodiesel was produced in the Laboratory through basic transesterification reaction (Tapanes, 2020). Calcium Lignosulfonate grade one, used as a multifunctional additive in this study, was obtained from the market, it is an amorphous yellow powder, extracted from of wood. To evaluate the corrosivity of the fuel mixtures on the steel, immersion tests were performed to calculate the weight loss and the corrosion rate. Initially the steel specimens were weighed and after were suspended with the aid of a thread in the beakers each containing fuels mixture; and the beakers were kept stationary to avoid displacement effect. The exposure periods were a total of 1440 hours, with measurements taken at specifics time intervals (24, 48, 72, 720 e 1440 horas). In each measurement, the specimens were taken out from the media, and the corrosive films were removed and samples were cleaned properly before drying. The dried samples were weighed carefully in a balance until a stable weight was achieved. The average corrosion rates of steel in the environment under study were calculated using weight losses of specimens over time. The losses were obtained by the difference between the weight before and after immersion. The measurements were taken at the define time intervals and from these results, the corrosion rate was determined according to ASTM G31. In each measurement of weight loss of specimens, the acid number of fuel mixtures were measured too, according to the ASTM D664 standard test. After each specimen reaches 1440h of immersion in the fuel mixture, images of the material surface were obtained by optical microscopy and the corroded areas were quantified by image analysis software *ImageJ*. It was set the same scale for all images. The microstructural observation of the microalloyed steel used in this work was carried out through optical microscopy using an OLYMPUS BX 51M optical microscope, connected to a computer containing the MSQ® program, microstructural image analyzer.

Factorial Design: To implementation of the experimental design a full factorial designs was chosen. Based on the available literatures and previous corrosion experiments three factors were used as the independent variables. The factors were Biodiesel concentration (B); Additive concentration (A) in fuel mixtures and contact time between fuel mixture and microalloyed steel, represented by Immersion Time (T) and monitored in periods of 24, 48, 72, 720 and 1440 h. The responses used in the exploitation of the design were the determination of the corrosion rate (CR), results of the gravimetric experiments and the acidity number of fuel mixtures (AN) determined according to the ASTM D664 standard test. The design information about each variable and corresponding levels are shown in Table 2.
Multiple regression tests were used to develop a mathematical model, which allows simulating the behavior of factors on the response variable. A second-degree polynomial model was established for the response. In order to obtain the model, 20 experiments were carried out, each one of them was perform at random to reduce systematic errors. The statistical data treatment was accomplished using the analyses of variance (ANOVA) to determine the significance of factors and Response Surface Methodology (RSM) for optimization.

RESULTS AND DISCUSSION

Statistical analyses: Response measured for the CR of the different mixture were represented in Table 3. Mathematical model was developed to acquire an understanding of the effect of the studied variables and their interactions on the corrosion rate of microalloy steel. ANOVA, illustrated in Table 4 is used to decide which model parameters affect significantly the experimental outputs. This table provides the percentage of variance explained by the mathematical model in comparison to the variance contained within the experimental results. The probability for ANOVA (Significance of F) was 0.00024, smaller than 0.05, confirming the validity of the suggested model. Komariah (2019), evaluated the corrosion of three different steels in tanks for B100 and B20 blends from different diesel samples. The authors found that the occurrence of corrosion was related to some changes in the physical properties of the fuel stored after 135 days of storage. Among the properties with the greatest variation, acidity, sulfur and water content gained prominence. Table 4 illustrate the estimated regression coefficients and P-values for each independent variable. The level of confidence was defined was 95%, that represented the degree of the statistical reliability of the model and it allows determining the uncertainty or risk degree, which is the difference between 1 and the confidence level (5% in this case). For every independent variable, if the P-value is lower or equal to the risk degree (0.05) there is a significant correlation between the dependent variable (in this case the CR) and the independent one, while P-value higher than 0.05, shows that the dependent variable is not correlated to the independent one. A marginal effect (p value = 0.07) was observed between biodiesel concentration and immersion time, interaction BT. The estimated regression coefficients in the model show that the main and interaction effects have a noteworthy influence on CR. The p—values of B, A and T are below the accepted value of 0.05, them the three main effects are significant, Table 4. The R-Sq and adjusted R-Sq (adj) values for the CR regression model are 82.85% and 74.94%, respectively. These coefficients show high levels (above 70%). Concerning adjusted R^2, the model explains 74.94 % of the variation in the data, which implies that the model has good predictability.

The model equation for the CR of steel is shown in Equation 1.

$$CR = 0.02517 - 0.00044 * B - 0.01075 * A - 0.00002 * T + 0.00001 * A * T \pm 0.0044$$

As can be seen, the equation includes an intercept, three linear terms and one interaction term. The parameters B, A and T are the functions of biodiesel concentration, additive concentration and immersion time, respectively. The only interaction term recognized as significant by the software is the interaction of AT. BA interaction was marginal effect and BT interaction was insignificant, therefore removed from the equation. Residual analysis was performed to check for the assumptions of ANOVA and validate the regression model. These assumptions are: error terms are normally and independently distributed with a mean of zero and a constant variance. The results of Residual analysis were shown in Figure 1.

Table 1. Detailed composition analysis of steel API X70 sample used in the experiment

Elements	C	Si	Mn	Cr	Mo	P	Cu	Ni	Fe
Wt.% compositions	0,06	0,26	1,58	0,22	0,11	0,02	0,02	0,02	Balance

Table 2. Variables employed in the experiments

Factor		Factor levels	Values
B, %m/m	*Biodiesel concentration**	2	0, 20
A, %m/m	*Additive concentration**	2	0, 1
T, hours	*Immersion time*	7	24, 48, 72, 720, 1440

* *in fuel mixtures*

Table 3. Experimental matrix used with response vectors.

Experiment	1	2	3	4	5	6	7	8	9	10
CR	0,0069	0,0136	0,0039	0,0035	0,0027	0,0182	0,0147	0,0164	0,0032	0,0025
Experiment	11	12	13	14	15	16	17	18	19	20
CR	0,0141	0,0153	0,0136	0,0031	0,001	0,0303	0,0279	0,0192	0,0036	0,002

Table 4. ANOVA of factorial design for the CR

Source of variation	Regression
S	0,0044
R-sq	82,85%
R-sq (adj)	74,94%
Degree of freedom	6
Significance of F	0,0002432

Term	Intercept	B	A	T	BA	BT	AT
Coeficients	0,025	-0,0004	-0,011	-1,86E-05	0,0001	3,51E-07	7,76E-06
Value P	1,24E-7	0,0174	0,0056	4,29E-05	0,5711	0,0708	0,0487

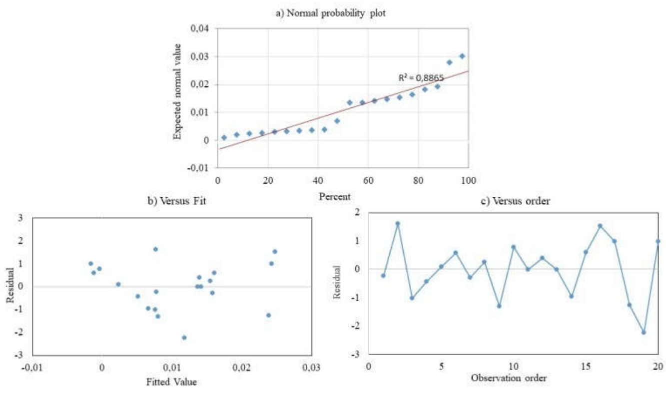

Figure 1. Residual plots for CRs of steel showing the accuracy of the model used

The normality assumption was checked by plotting Normal P-P Plot of Residuals as shown in Figure 1a. There is nothing unusual with this plot as the residual is seen to be fairly distributed along the mean line and there is no possible outlier that reveals any non-normality in the distribution. An observation is an outlier if the standardized residual related to that observation is less than -3 or greater than 3, according to Montgomery (2005). In addition, the residual plot in the time sequence (Figure 1b) is satisfactory as there is no positive correlation indicating violation of the independence assumption. Finally, the residual versus adjusted value plot of Figure 1c configure an unstructured pattern demonstrating that the constant variance assumption is answered. Figure 2 shows the Pareto chart of the effects of the controlling variables on the response variable. By the Pareto chart were plots the effects in the decreasing order of their absolute values. The reference line on the chart indicates which effects are significant, in this study was used Lenth's method to draw the reference line. In Figure 2 is possible observed that the three main effects (B, A and T) are statistically significant, besides interaction AT and BT. These effects confirm the results obtained in the ANOVA table (Table 1b). In addition, it is seen that the largest effect is immersion time (T) because it extends the farthest. The effect of interaction Biodiesel concentration and Additive concentration (BA) is the smallest because it extends the least.

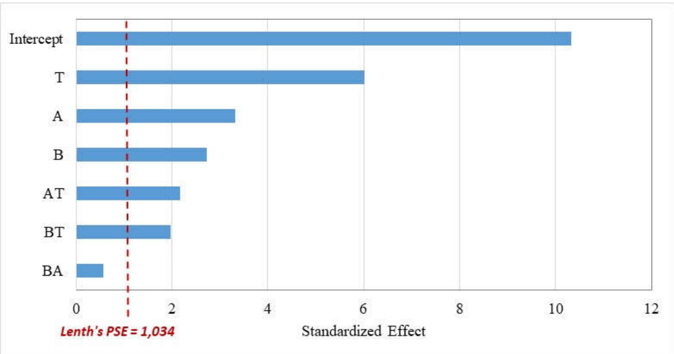

Figure 2. Standardized Pareto Chart for CR

Response Surface Analysis: Figures 3a and 3b show the evolution of the CR as a function of the biodiesel concentration, additive concentration and the immersion time. The graphs shown corroborate the significant influence of these factors on the response. In Figures 3a and 3b it is possible to observe the initial rate of steel corrosion decreases with the immersion time.

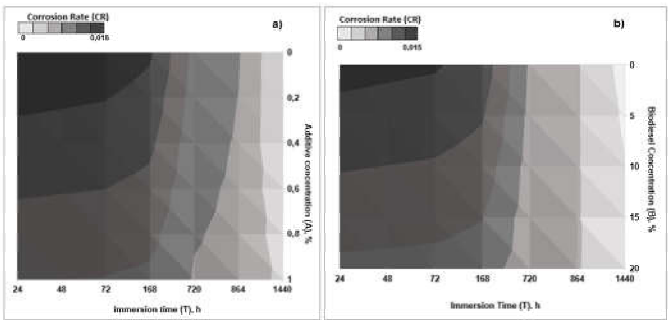

Figure 3. Variation of CR with a) additive concentration and the immersion time, fixing biodiesel concentration at 20%; b) biodiesel concentration and the immersion time, fixing additive concentration at 1%

This result suggests the development of a protective surface film (corrosion products) that promote a reduction in the weight loss of the steel and, consequently, in the decrease in CR. Some authors have reported evidence of this effect in microalloyed steels. Onyeji (2017), Vera (2015) and Davis (2000) also attributed the decrease in the CR to the formation of corrosion products on the surface of the specimens during the loss-in-mass tests of API steels in seawater. Studies perform with steels and alloys reported that the light rare earth elements (LREEs) Cerium and Lanthanum that compose these steels, have an effect on the corrosion properties, promoting the formation of passive films of corrosion products (Tang, 2019). However, only a few studies have investigated the effect of microbonding with these elements on corrosion behavior. Yoo (2010), evaluated the effects of LREEs (Ce, La) in super duplex stainless steels on the corrosion of the material in aqueous solutions through electrochemical tests and surface analysis. The results showed the formation of a passive film enriched with chromium. Figure 3a shows response surface of the AxT on CR, it is observed the additive concentration increase reduces the CR, a logical result considering that the additive's function is to reduce the formation of compounds that decrease stability oxidation. Similar results were obtained by Santos (2017). Factorial design made it possible to analyze the influence of factors on response variable. Considering the values presented in the response surface analysis, it can suggest that the use of 1% of the additive concentration (A) minimizes the CR.

Figure 3b presents that during the weight loss tests there is a reduction in the CR with the increase in the biodiesel concentration in the mixture. Similar results were observed by Tavares (2020), confirming the formation of a film that reduces corrosion. It is important to point out that this study did not consider variations in sulfur and water content in fuel mixtures. Some authors report the influence of these factors on the CR. Oliveira (2017), analyzed the sulfur concentration present in marine diesel, in the order of 5000 ppm, the authors noticed that the sulfur content is a great aggravating factor for the metal deterioration, the higher the biodiesel content added, due to the oxidative instability of biodiesel over time. Similar results were obtained by Tapanes (2020) and Komariah (2019). Komariah (2019), evaluated the corrosion of three different steels in tanks for B100 and B20 mixtures from different diesel samples. The authors found that the occurrence of corrosion was related to some changes in the physical properties of the fuel stored after 135 days of storage. Among the properties with the greatest variation, acidity, sulfur and water content gained prominence.

Microscopy analysis: Figure 4 shows the micrographs of the specimen obtained through optical microscopy, right after the removal of the evaluated fuels at 1440h. All micrographs presented were taken at 200x magnification. According to Table 3, the specimens with immersion time of 1440h were the experiments 5, 10, 15 and 20. Experiments 5 and 10 are of specimens that were in contact with mixtures with 20% biodiesel. Experiments 5 and 15 are of specimens that have been in contact with additive mixtures. Therefore, experiment 20 represents the specimen that was 1440 h in contact only with fossil diesel.

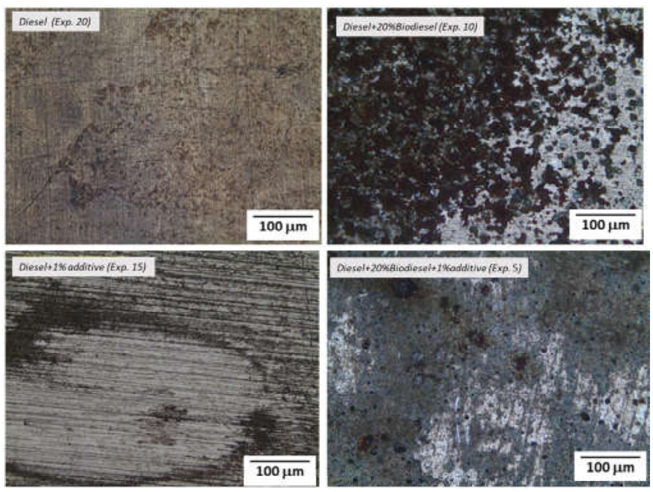

Figure 4. Optical images of microalloyed steel specimens after 1440 h immersed in fuels mixture

Through optical microscopy analysis it was possible to verify, after 1440h, the addition of biodiesel to diesel promotes an increase in corrosiveness in the fuel mixture, and this increase provides greater corrosion of the specimens. It is observed in experiments 5 and 10 the specimens present two types of corrosive processes on the surface (pitting corrosion and generalized corrosion). The results suggest that after 1440 h, fuel mixtures with 20% of biodiesel content may break or reduce the protective layer formed on the surface, causing an increase in the CR value. On the specimens immersed only in fossil diesel (with and without additive) it was observed with greater intensity generalized corrosion, although small pits can be observed on the metallic surface. The use of lignin as an additive (concentration of 1%) promoted the reduction of corrosive processes, especially when blended with biodiesel. There is clearly a reduction in the size and number of pits on the surface of the sample. The specimens characterized in Figure 4 were sanded to remove localized corrosion and better visualization of pits. Figure 5 illustrates the micrographs after sanding.

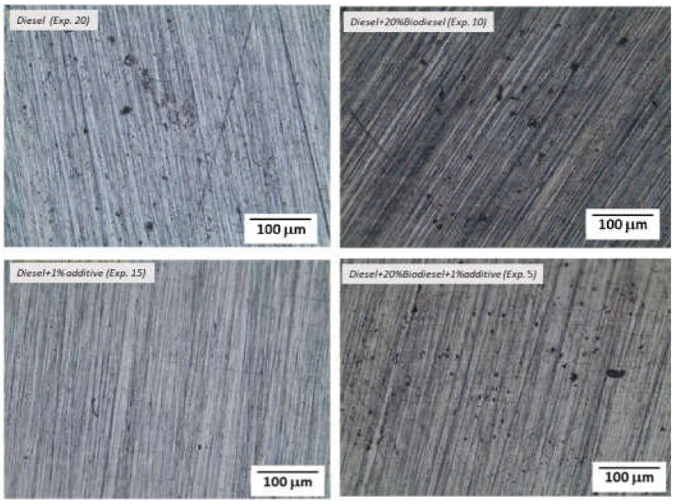

Figure 5. Optical images of microalloyed steel specimens after 1440 h immersed in fuels mixture and subsequent sanding

After removing the oxide formed, product of generalized corrosion, was evident that pitting corrosion becomes pronounced with addition of 20% biodiesel to diesel. Also was observed that the addition of lignin acts as an inhibitor of the corrosive process of specimens. However, after removing the oxide, it was confirmed that lignin was more significant in reducing generalized corrosion than pitting.

CONCLUSIONS

In this research, design of experiment was used to evaluate the interaction of diesel-biodiesel mixtures with microalloyed steel.

- The ANOVA of the CR demonstrated that the second-order regression model is the best fit, with the most statistically significant parameters being immersion time (T) and additive concentration (A). In a lesser degree of statistical significance is the Biodiesel concentration (B) and the AT interaction.
- The full factorial design of the response CR showed that there is a regression model with acceptable fit (R2adjusted= 74.94% and significance F=0.0002432) and adequacy of the model was supported by residual analysis
- SRM revealed a decrease in the CR with immersion time. Effect that suggests the formation of a protective layer on the metallic surface.
- The study demonstrates the effective action of the lignin-based additive on fuel corrosivity.

With the microscopy analysis of microalloyed steel samples after 1440h of contact with diesel-based fuels, we can conclude that:

- After 1440 h, biodiesel concentration of 20% in diesel promotes an increase of corrosion, accentuating both generalized corrosion and pitting.
- The lignin-based additive presented an inhibiting effect on corrosive processes biodiesel, being more efficient in reducing generalized corrosion than pitting corrosion.

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