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DETECTION AND ANALYSIS OF ADULTERATED FUELS IN INTERNAL COMBUSTION ENGINES: DEVELOPMENT OF A PROTOTYPE

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Abstract. In Brazil, even with frequent inspections, cases of adulteration in anhydrous ethyl alcohol fuel (AEAF) through the addition of water above the limits allowed by law (4,9% v/v) are very common. Therefore, the objective of the study was to develop a device capable of identifying these adulterations and, for that, the sensibility of five physicochemical parameters was tested (electrical conductivity, density, viscosity, refraction index, and pH). Accordingly to the referential, lower (3,2% v/v), equal (4,9% v/v) and higher (5,2% v/v) volumetric percentages of water were mixed with the AEAF. Posteriorly, a detector device able to identify adulteration in fuels was developed. A programming code was written for the operation of an electro-resistive sensor and, after a successful simulation in the software Tinkercad, it was transferred to an Arduino UNO R3 board. The results of electrical conductivity of AEAF showed the following values: 180 $\mu\text{S/m}$ (3,2% v/v); 348 $\mu\text{S/m}$ (4,9% v/v) and 380 $\mu\text{S/m}$ (5,2% v/v). The other parameters tested did not demonstrate significant sensibility. These results were applied in the development of the prototype with an electro-resistive sensor that can be integrated into vehicles as an onboard system that identifies adulteration in fuels based on electrical conductivity measurements.

Keywords: Fuel, adulterated, detection device, electrical conductivity.

1. INTRODUCTION

Many types of fuels are commercialized around the world, with gasoline and ethanol being the main sources of energy used in light vehicles. In Brazil, although the National Agency for Petroleum, Natural Gas and Biofuels (ANP) often inspects the gas stations, there are still cases of adulteration in fuel.

During the period of assessment, it was noticed that the hydrated ethanol was the fuel with the most irregularities. Among the non-conformities detected by the inspection, two stood out the most: the specific weight at 20°C/alcohol content was under the technical specification in 52.1% of the samples analyzed and the electrical conductivity content was above the allowed by law in 16.7% of the samples (ANP, 2020).

Figure 1 (ANP, 2020) illustrates the percentual distribution of the irregularities verified on the hydrated ethanol samples that were not in conformity with the specification.

Moreover, it is notable that when the fuels are submitted to adulterating substances, the incomplete combustion of the fuel occurs, resulting in the production of more toxic gases that are harmful to the environment and human health. This process does not occur when the fuel is unchanged.

Furthermore, in the literature that are references to projects that developed researches focussing on the detection of adulterated fuels, such as: "Development of Sensors and Electronic Systems with WEB Interface for the Detection of Pollutant Gases and Adulteration in Fuels" (Becari and Ramirez, 2010); "Detection of Adulteration in Gas and Prevision of the Physical-Chemical Parameters from Distillation Curves Associated With Chemometric Tools" (Mendes, 2012); "Project and Development of a System for Detection of the Volumetric Fraction of Water Contained in Ethanol" (Luceiro, 2017). Although that are many studies about fuels adulteration, none of them presented a portable prototype nor adapted it to vehicles, ensuring that the authorities are triggered as fast as possible. Which means that all the projects in literature are based in laboratory tests that uses ideal conditions to perform the measurements in fuel.

In this context, the realization of the present study is justified by the need to expand the inspection of the gas stations since, even with the ANP inspection, there are still cases of proprietaries that use illicit practices to lucrative benefits. The operation of the sensor in analysis has as goal identify adulterations through the relevant physicochemical parameters related to the adulteration of the Anhydrous Ethyl Alcohol Fuel (AEAF), performed by the addition of water above the limits allowed by law, to sell it as Hydrated Ethyl Alcohol Fuel (HEAF).

So, the prototype of the device proposed in this project could be an alternative to detect adulterated fuels since it's a new technology never implemented in practice. The innovative factors are:

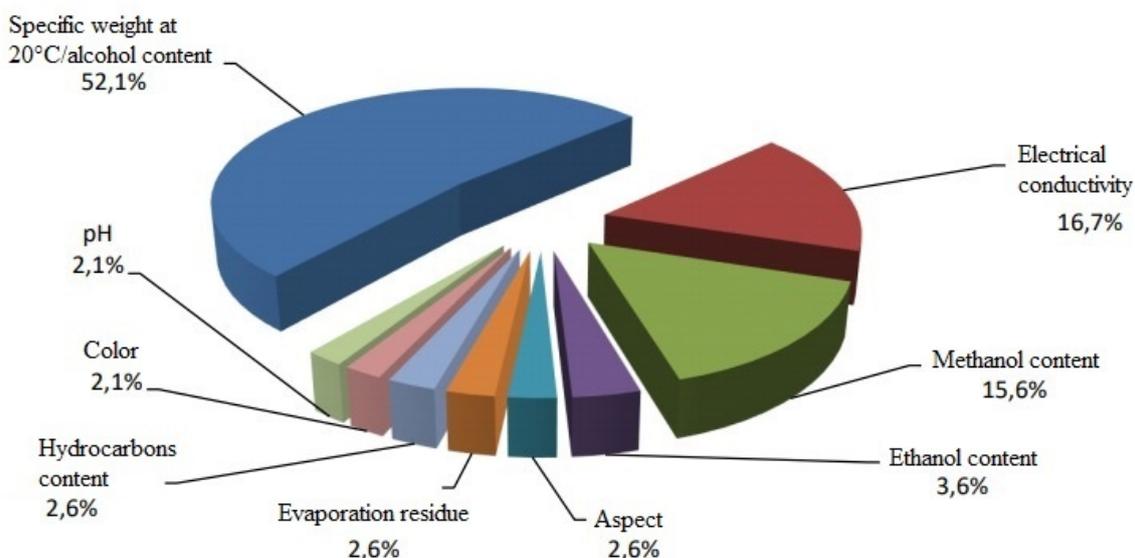


Figure 1. Percentual distribution of the irregularities verified in the hydrated ethanol

- Its a portable device (still in the development phase) that measures the electrical conductivity of fuel samples and notifies, through electric signals, quickly and simply, if the fuel is in good conditions of use;
- This device will be adapted in the vehicle tank entrance and interconnected to the car panel to notify the driver if the fuel has good or bad quality. During the filling, a sample of the fuel will fall into a secondary reservoir containing an electrical conductivity sensor.

2. Objectives

2.1 General objective

The research has as goal the evaluation of five physicochemical parameters: electrical conductivity, density, viscosity, refractive index, and pH; accordingly with their sensibility to identify adulteration in Anhydrous Ethyl Alcohol Fuel (AEAF), commonly performed by the addition of water. Using the results obtained from this study, present a methodology of development for the operation of a sensor that identifies fraud in fuel.

2.2 Specifics objectives

- Verify the influence of the physicochemical parameters in the detection of adulterations, such as electrical conductivity, density, viscosity, refractive index, and pH;
- Determine the parameter that will be detected by the sensor;
- Write the sensor's programming code;
- Perform computational simulations in the Tinkercad software to validate the code;
- Build the prototype.

3. Theoretical referential

3.1 Electrical conductivity

Electrical conductivity can be defined as the ability of a material to conduct electric current through ions present in a solution (ABNT, 2016). A characteristic of the solutions is their higher or lower capacity to conduct electric current. The Brazilian standard, governed by the Brazilian Association of Technical Standards (ABNT), that verifies the maximum value for the electrical conductivity of HEAF is ABNT NBR 10547 de 2016. This standard is followed by the Technical Regulation ANP n° 3/2011. Thus, the maximum limit for the electrical conductivity of the HEAF at 25°C is 350 $\mu\text{S}/\text{m}$.

Electrical conductivity and electrical resistance are the numerical representation of the malleability of a material and its ability to allow the passage of an electric flow through its extent. Thus, they characterize the material's propensity to transport electric charges. The ability to transport electricity is directly related to the number of free electrons in the

material or substance (Fujiwara, 2015). Therefore, it can be affirmed that a material or electrolytic aqueous solutions can be characterized according to their electrical conductivity.

The electrical resistivity (ρ) [$\Omega \cdot m$] (Eq. (1)) and the electrical conductivity (σ) [S/m] (Eq. (2)) are properties that depend on the geometrical characteristics of the material or solution. They are identified by:

$$\rho = \frac{R \cdot a}{l} \quad [\Omega \cdot m] \quad (1)$$

$$\sigma = \frac{1}{\rho} = \frac{l}{R \cdot a} \quad [S/m] \quad (2)$$

Where R is the resistance, in ohms [Ω], a is the area, in square meters [m^2] of the transversal section of the material, and l is its length [m].

In electrolytic aqueous solutions, the electric current conduction occurs due to the transport of electrons performed by ions that move when in contact with an electrostatic field. (Barrow, 1982). The migration speed of the ions present in the electrolytic solution is limited by the solvent resistance to the movement of the particles from one electrode to another with opposite charge (Ohlweiler, 1981).

From the theoretical foundations here presented, the sensor proposed in this research project has electrodes that are under influence of an electric potential difference applied between its terminals. In this way, is possible to understand that the migration speed of ions has a linear correlation with the electric potential difference, and also it is known that the electrolyte solutions follow Ohm's Law: the electric current is directly proportional to the electric potential difference and inversely proportional to the electrical resistance of the surroundings.

Therefore, knowing that the conductivity of a substance can vary accordingly with its composition, in this research the study target was to analyze the HEAF electrical conductivity due to the increase of the water percentage in its composition, to notice if this parameter is capable of identifying this sort of adulteration.

3.2 Sensors

A sensor is a system that receives a signal or a physical stimulus and usually replies with an electric signal that is sent to a controller (Fraden, 2010). Therefore, the stimulus is understood as the number, property or condition detected and converted in an electrical signal (Sousa *et al.*, 2012).

Sensors are separated into Active Sensors, defined as those who do not generate voltage, and Passive Sensors, defined as those who generate an electrical signal after receiving a stimulus (Mendonça, 2008).

Sensors rely on signal conditioning for the device of data acquisition perform the measurement in an effective and precise way. The main signal conditioning technologies provide distinct improvements not only regarding the performance but also regarding the accuracy of the system responsible for data acquisition (NI, 2021).

In this context, the methodology for the development of a sensor capable to detect adulterated fuels is based on the detection of an external stimulus (electrical conductivity of the fuel) to provide an electrical signal through a passive sensor. A few examples of passive sensors are: thermocouple, pH sensor, piezoelectric sensor, and also electrical conductivity sensor.

3.3 Arduino

Arduino is a platform of electronic prototyping, created by Massimo Banzi and David Cuartielles in 2005, to allow the development of the control of interactive systems, at low cost and affordable (AP, 2021).

Currently, there are on the market many companies focused on the manufacturing of "free hardware", including its own platform, that enables the development of many tasks with signal processing and data acquisition. The Arduino stands out in this market. Signal processing already is a necessity not only in the measurement of physical quantities for study purposes but also in the performance and operation of the process (El-Heri, 2017).

Through the Arduino, it is possible to send and receive information from practically any other electronic system. Thus, is possible to develop, for example, a sensor data capturing system, as temperature, lighting control, conductivity, process and send this data to a remote system, or show them on a screen. So, for didactic purposes, the use of this type of plaque was proposed in this research project. Among its advantages, it is possible to cite: low cost, functionality, practicality, and the possibility to consult many didactic materials on the web, that are easy and free.

4. Methodology

In this research methodology, many tests were performed in the Technology Laboratories of the Superior Institute of Engineering of Porto – ISEP (Portugal) and the Minas Gerais Federal Institute (Brazil). For this aim, samples of AEAF were adulterated by the addition of water, to verify which of the physicochemical parameters would be able to identify these adulterations.

4.1 Density

Density is the ratio between the material weight and the volume occupied by it. That means that density is a specific property of each material and can be calculated for liquids, solids, or gases (Fogaça, 2015). The measurement unit commonly used to represent the density of liquids is $[g/cm^3]$. Nevertheless, according to the International System (IS), the measurement unit used for density is $[kg/m^3]$.

In the context of this academic project, density refers to the quantity of pure gas per liter of fuel inserted in the car. Usually, the density showed in the service station indicates the weight of fuel present in one liter of that gas. That is why this parameter was considered relevant during the development of this project.

In this way, the first parameter analyzed was the density of the samples. Firstly, 500 mL of each AEAF sample was transferred to a measuring cylinder. By doing this, the densimeter floated freely, without touching the container bottom or walls. After that, the densimeter (graduated in $0.800 - 0.900 g/cm^3$), cleaned and degreased, was submerged into the product samples and it was released with a rotating movement, with the purpose to make the densimeter quickly reach the equilibrium and float freely (without touching the measuring cylinder walls). Thus, the density reading was done on the surface of the liquid.

4.2 Viscosity

Viscosity is the measurement of the internal resistance of a fluid (gas or liquid) to the movement or the fluidity. In other words, refers to the resistance offered by the liquid when a layer moves in relation to an underlying layer (Çengel, 2013). Therefore, the higher the viscosity, the higher the resistance to movement, and the lower is the flow capacity of the fluid.

The viscosity can be explained by the cohesion forces of the molecules of the fluid. In this project, the fuel represents the fluid. When trying to move a layer of water over another, for example, is necessary to surpass the resistance force induced by the attraction between the molecules of the two layers (Çengel, 2013). In this way, this parameter is relevant for the project because it enables to identify if the fuel is out of the conditions allowed by law. In other words, viscosity is a characteristic that can alert about the quality of fuels.

So, the second parameter analyzed was viscosity. Firstly, the samples were transferred to a Cannon Fenske viscosimeter through a tube with the assistance of a funnel. Then, the viscosimeter with the fuel was submerged in a thermostatic bath and waited for the temperature to stabilize in $20^{\circ}C$. Next, suction was applied in the tube using a pipette bulb until the liquid that was in the primary bulb of the tube reached the secondary bulb. After that, the pipette bulb was removed and the flow time between the marks was measured.

4.3 pH

The hydrogenic potential (pH) is an index that indicates the acidity, neutrality, or basicity of a medium (Souza, 2015). This index is also applied in the context of fuels, being a target of control by the ANP. The Technical Regulation n^o3/2011 specifies that the pH of the HEAF must be in a range of 6 – 8, which means that it can't be too basic nor too acid.

The value of pH is related to the concentration of hydrogen ions (H^+) in a solution. Lower values of the pH index indicate that there are more ions H^+ and fewer ions OH^- in the solution (Souza, 2015). On a scale of values, the substance can be considered acid (pH 0 – 6), neutral (pH around 7), or basic (pH 8 – 12).

Knowing that the pH of a substance can vary accordingly to its composition, in this project the variation of the pH of the HEAF due to the increase of water in its composition was analyzed, to evaluate if this parameter is capable of identifying this sort of adulterations.

Therefore, the third parameter analyzed was the pH. For that, the method described in the Brazilian standard NBR 10891 (pH determination, potentiometric method). Thus, to measure the pH of the HEAF samples with different water percentages the pH-meter was used – brand Metrohm, in the Technology Laboratory of ISEP. So, to analyze the samples the electrode was inserted into the solution. During the immersion was necessary to homogenize the fuel solution and, by that, the electrode was placed in a rotating movement, without touching the container walls. Lastly, it waited until the pH-meter reached stability in a fixed value and the results were collected.

4.4 Refraction index

Refraction is the optical phenomenon characterized by the alteration of the speed of light due to a change in the propagation medium. The absolute refraction index (n) is a dimensionless value that represents the ratio between the speed of light in the vacuum (c) – $3,0 \cdot 10^8 m/s$ – and the velocity of light in any environment (v) (SILVA, 2015). Furthermore, the refraction index is commonly used to determine the concentration of ethanol in water so that the existence of a maximum point is capable of limitate this application (Galvão *et al.*, 2015). So it is expected that the increase in the water volume percentage also increases the refraction index of the HEAF mixture with water.

Therefore, the objective of the refraction index test was to evaluate if the refractometry is a method capable of predict irregularities in the HEAF. This test also had the objective to verify if the refraction index of the HEAF changes (increase) due to the increase in the water volume percentage.

Thus, the fourth parameter analyzed was the refraction index. The methodology used in this test consisted in perform the reading of the refraction index of the mixtures containing different percentages of water with an ABBE Refractometer (brand Atago). Maneuvering the handle, with the help of a Pasteur pipette, a drop of each sample previously homogenized was placed to form a thin film in the prism surface. With the measurement command, the line that separates the hemispheres was adjusted with the cross of the screen itself. Therefore, the reading of the refraction index values was performed with four decimal digits, with the last one being determined by estimation.

4.5 Electrical conductivity

The electrical conductivity is the capacity of the material to conduct electric current through the ions present in the solution (ABNT, 2016). One of the characteristics of solutions is their higher or lower capacity to conduct electric current. Both ethanol (C_2H_5OH) and water (H_2O) are substances formed by covalent bonds between the atoms, so they don't have ions in the pure state. The ethanol O-H bond is polarized, but there will not be a sufficient number of ions to modify the electrical conductivity of the solution when mixed with deionized water (Martins *et al.*, 2013).

The concept of deionized water is related to the absence of the other components that aren't water. Thus, the conductivity is practically null, which indicates that this type of water is an electrical insulator. In the sort of fraud that this project evaluates, the use of untreated water is ordinary, which leads to conductivity values much higher than those specified by law for the HEAF. So, it can be affirmed that the target of analysis to identify adulterations in the HEAF are impurities present in the ethanol or untreated water.

In this way, the fifth parameter analyzed was the electrical conductivity of the AEAF adulterated samples. To measure electrical conductivity, with different percentages of water, the conductivity meter *COND 51* of the brand *XS Instruments* was used, from the Technology Laboratory of ISEP. A sensor that measures electrical conductivity with a resolution of $0.01 \mu S/m$ ($1 \mu S/m$) was connected to the conductivity meter. The temperature in which the test was performed was $25^\circ C$. So, to analyze the samples, the conductivity meter was inserted in the solutions containing different percentages of water added. Then, the samples were slightly agitated to maintain the value of electrical conductivity uniform, allowing the results obtention.

The ANP Technical Regulation n° 3/2011 specifies the maximum limit of $350 \mu S/m$ for the conductivity of the Hydrated Ethyl Alcohol Fuel (HEAF) at $25^\circ C$. The maximum concentration of water allowed by law in the mix is 4.9% (v/v). The value of electrical conductivity of the sample with 3.2% (v/v) was below the maximum limit, while the sample with 5.2% (v/v) showed a value above this limit, which leads to the belief that this parameter is efficient and accurate to detect this sort of adulteration.

To confirm if this parameter is really relevant in this context, the electrical conductivity of a HEAF sample with 4.9% of water was measured. This sample had a conductivity value of $348 \mu S/m$, which is very close to the maximum allowed by law ($350 \mu S/m$). So, it can be concluded that this parameter has a very high potential to detect this sort of adulteration with accuracy.

4.6 Development of the sensor

After being verified that the electrical conductivity was the parameter that would be identified by the sensor of this project, the methodology of this research pursued to develop a prototype that was able to integrate a simple electronic prototyping platform (Arduino) to an electrical conductivity sensor. In this way, to promote the development of the control of an interactive system, it sought to elaborate a programming code that allowed the capture of data, its processing, and its sent to a display screen.

The programming logic involved in the device consists in turn on a green LED if the electrical conductivity value is equal to or below the maximum allowed by law for the mixture of ethanol and water ($350 \mu S/m$). However, if the reading value exceeds the maximum value, a red LED will light up.

4.7 Simulation

For simulation and validation of the programming code purposes, to verify if the circuit will actually work and avoid unnecessary expenses, computational simulations were performed (Fig. 2) using the software *Tinkercad*. This software basically consists of a free and online platform where is possible to create and program electro-electronics circuits in a didactic way and test the functionality of its components.

Figure 3 (HE, 2021) illustrates the connection diagram of the sensor with the Arduino. Through this image, it is possible to notice the geometric arrangement of the components that integrate the circuit. It is worth mentioning that, to ensure the measurement accuracy, the addition of a temperature sensor to measure the temperature and reach its automatic

compensation is highly recommended.

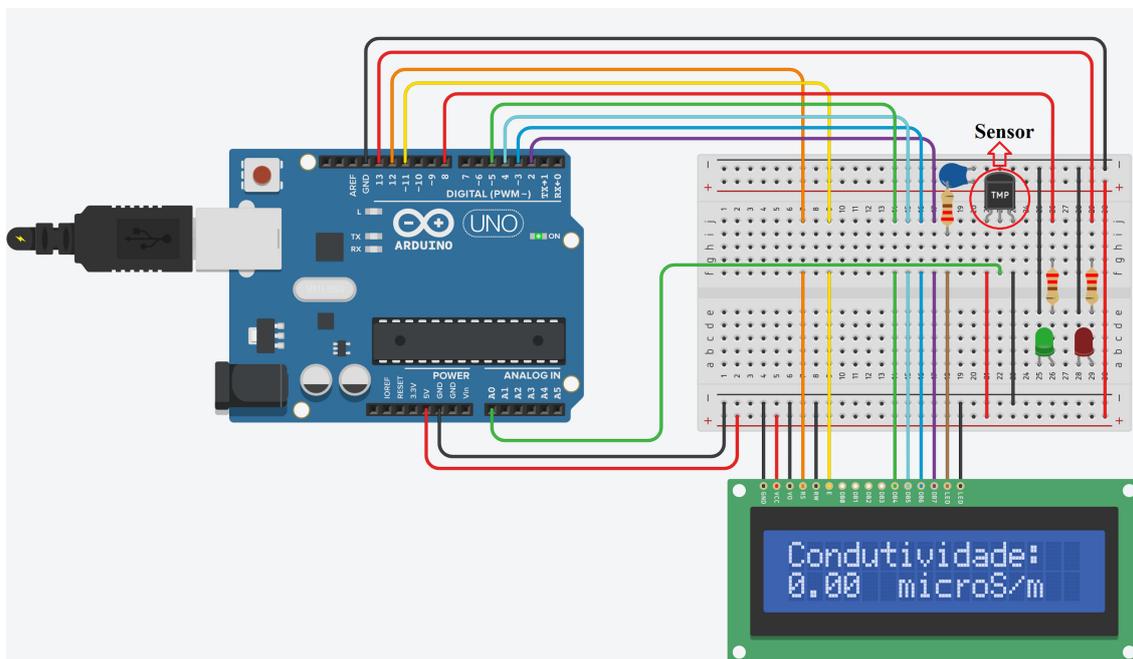


Figure 2. Simulation of the electronic circuit of the sensor in the software *Tinkercad*

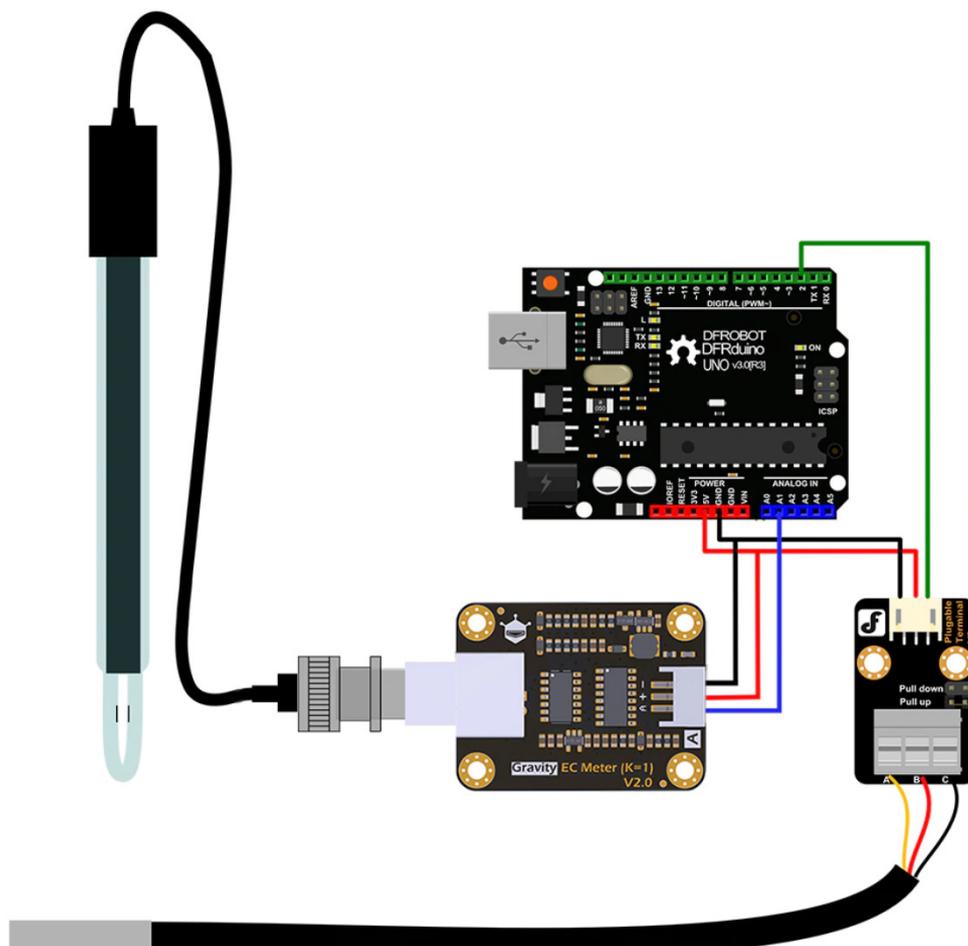


Figure 3. Diagram of the connection between the electrical conductivity sensor and the Arduino

5. Results and discussions

5.1 Density

The results obtained from the topic 4.1 were: the ANP Technical Regulation nº 3/2011 specifies that the maximum limit of the hydrated ethyl alcohol fuel (HEAF) density at 20°C should be in a range of 0.8076 – 0.8110 g/cm³. The maximum concentration of water allowed by law in the mix is 4.9% (v/v). The sample with 5.2% (v/v) presented density equal to 0.8110 g/cm³, which is the maximum value allowed by law. Therefore, it was notable that density is a parameter whose limit value has a small margin of tolerance. Hence, it showed to be inappropriate to identify adulteration in this type of fuel as an onboard system integrated into a vehicle since its variation is very small, requiring a measurement device with a very high degree of accuracy.

5.2 Viscosity

The results obtained from the topic 4.2 were: accordingly to the technical specifications of the product, where the physicochemical properties of the HEAF are specified, issued by the leading distribution company of this fuel (Petrobras), it is believed that the HEAF viscosity at 20°C is 1.2 cP. The maximum concentration of water allowed by law in the mix is 4.9% (v/v). However, the sample with 5.2% (v/v) also presented the viscosity provided by Petrobras within the conformities. Therefore, this parameter is not capable to identify adulterations. Beyond that, since this parameter is not standard referred, it should not be used to obtain conclusions about fuel adulteration.

5.3 pH

The results obtained from the topic 4.3 were: although the pH is a fundamental parameter to the fuel the quality control during its production, it is not a method appropriated to identify adulterations in the AEAF through water addition, since even increasing the percentage values of water, the pH of the samples was still inside the value range provided by law (pH 6 – 8). Therefore, this parameter also proved to be inadequate.

5.4 Refraction index

The results of the test described in the topic 4.4 showed an average refraction index of 1.3637 and a standard deviation equal to 0.0003815. These numbers evidenciate that the non-conformity of the HEAF samples can be identified when the refraction index of this fuel is above the maximum limit of 1.3641. If the refraction index is outside of this confidence limit, it indicates that the sample has some kind of alteration in its composition. On the other hand, although these results had been satisfactory to identify such irregularities, it would not be possible to adapt a refractometry system to a vehicle onboard system, therefore, these results only are effective in environments with controlled conditions.

5.5 Electrical conductivity

Regarding the adulterations suffered by the Anhydrous Ethyl Alcohol Fuel (AEAF) through the addition of water above the maximum value allowed by law (4.9% v/v), it was possible to verify that the most relevant parameter was the electrical conductivity. Since the ANP Technical Regulation nº 3/2011 specifies the maximum limit of 350 µS/m for the conductivity of the AEAF at 25°C; samples with 3.2% (v/v), 4.9% (v/v) e 5.2% (v/v) of water added in their compositions were analyzed. The results were quite positives, being: 180 µS/m (3.2% v/v); 348 µS/m (4.9% v/v) e 380 µS/m (5.2% v/v).

Thus, there is a great possibility to develop a practical device capable of quantifying the electrical conductivity and integrate it into a vehicle onboard system. Therefore, between all the parameters analyzed in this research, this was the one selected to present a supposed methodology for the development of a sensor that identifies adulterated fuels. Figure 4 portrays the prototype of the device that identifies frauds in fuels developed in this research.

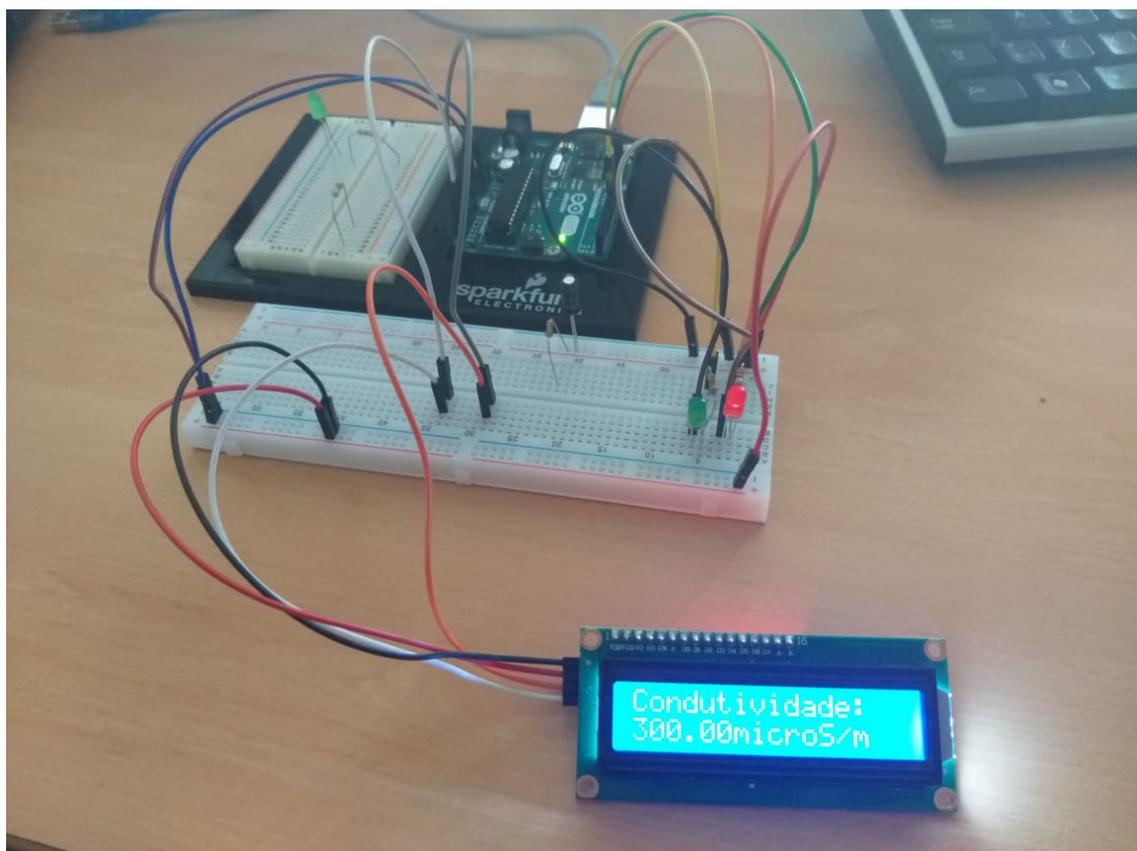


Figure 4. Final prototype developed

6. Conclusion

Therefore, it was concluded that, among all the parameters analyzed, electrical conductivity was the most appropriate to be used in the methodology of development of a sensor that detects adulteration in fuel (adulterated AEAF) since this parameter is capable to indicate, with precision, the capacity of the fuel conduct electrical current through ions (impurities) in its composition. Therefore, is feasible to construct a device that can be integrated into vehicles as an onboard system that identifies adulteration in fuels based on electrical conductivity measurements. The other parameters tested did not demonstrate significant sensibility.

These results were applied in the development of the prototype with an electro-resistive sensor. The programming of the electronic circuit, developed in this research for the sensor, was simulated in the computational software Tinkercad, to test the programming logic involved. The results of this simulation were positives, showing expected values of measurements for the conductivity and indicating, through a red LED, when the fuel is adulterated.

Lastly, the programming was transferred to an Arduino UNO R3 board and the components were assembled in the breadboard. Thus, after the realization of several tests, it was possible to build the prototype of the sensor. Therefore, the research developed contributes with literature and the practical application in the identification of fraud in fuels.

7. ACKNOWLEDGEMENTS

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