



CONTROL OF THE VARIABILITY OF THE BIOFUEL PACKAGING PROCESS THROUGH THE SIX SIGMA METHODOLOGY: A CASE STUDY

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ABSTRACT

The present work carried out the control of the variability in the biodiesel packaging through the application of the steps of the DMAIC (Define Measure, Analyze, Improve and Control) of the Six Sigma quality program. The DMAIC method brings together a set of statistical and quality tools for the study of process variability. Prior to the project, in the period evaluated between June and October of the year 2017 the average monthly variation of loss was 387.7 liters with a standard deviation of 421.9 liters. After the implementation of the improvements between January and March of the year 2018, the average monthly variation was -3.187 liters with a standard deviation of 60.95 liters. This evolution in the control and reduction of losses in the container of biodiesel was possible through the actions of improvements and involvement of the people of the billing and shipping. After all the improvements implemented, the maintenance and continuity of the controls met two important requirements of the Six Sigma quality program: customer focus and financial impact.

Keywords: Six Sigma. DMAIC. Biofuel packaging, statistical methods



1. INTRODUCTION

According to Montgomery (2004) quality is inversely proportional to variability. Quality improvement is related to reduced variability, where products and services can be produced in a stable process so that variability is small around a desired value. According to Rotondaro et al. (2011) it is common practice for company managers to view process and cost variations only in terms of averages.

This practice is risky because it can hide the root causes of variability. One family of tools widely used in developing Six Sigma programs is DMAIC. The acronym DMAIC originates from: Define, Measure, Analyze, Improve, and Control (DIP, 2003). The DMAIC method defines the steps that must be followed in executing a Six Sigma project from problem identification to permanent solution and control. In this context, this study is aimed to reduce losses due to volume variation in liters (L) of biofuels during the filling process.

Thus, the study identified some directions for improving these losses through the survey of existing data, the characterization of the filling process, the identification of the root causes of losses, the statistical analysis of data and the implementation of improvements in the filling, packaging sectors and billing which allowed for better control of the process. The results of this research may be of interest to other biodiesel plants that share the same problem. The paper is divided into five sections: 1. Introduction, 2. Biofuels Scenario in Brazil, 3. Six Sigma Methodology, 4. Description of Operation of Biodiesel Filling Process, 5. Results and 6. Conclusions.

1.1. Company involved in the study

The enterprise is a large company operating in various segments and one of them is in the production of biodiesel. The biodiesel plant started its operations around 2000 and currently its workforce has 85 employees, 55 of whom work directly in the industrial plant and the other 30 in the administrative sector.

The study was motivated by the necessity demanded by the operational management of the biodiesel plant regarding the effectiveness of the volume variability control system obtained from the mass of the scale versus the volumetric capacity of the truck, where the difference is pointed out at the invoicing moment registered in the invoice.

In this preliminary research, a high volume variation was found between the volume of the tanks and the volume obtained from the scale mass. From there it was necessary to

choose a scientific method to be used in project development; at the time there was no initiative to implement the Six Sigma program in its organizational structure at the time and it was chosen for the study using the DMAIC method.

The next step was to plan, define the research question and objectives in order to propose solutions to the organization's problem and the knowledge base. The research contextualization took place after the statistical analysis of the initial data, in which the losses and the need to reduce and control the variability in the biodiesel filling process were verified. This study generally evaluates the benefits obtained by applying the DMAIC method to the Six Sigma program for biofuel (biodiesel) shipment.

1.2. General goal

This study, as a general goal, applies the DMAIC method to control losses and analyze opportunities for improvements in the biodiesel filling process and evaluate the results of the operation.

1.3. Specific goals

To study and apply DMAIC steps to verify opportunities for improvement. It implements a management model to control the variability in the biofuel filling process. Establishes the appropriate statistical and quality tools during the application of the DMAIC steps. It uses statistical methods to compare data obtained before and after the project and points out the benefits of the study for the company and the literature.

1.4. Justification

According to Hoerl (1988) the Six Sigma method considers a set of practices with steps and statistical tools that make it possible to discover the root causes of variability and subsequently continuous process improvement. From the above, it can be seen that the Six Sigma method acts as an improvement strategy based on the reduction of process variability through the use of statistical tools and techniques for problem solving. In this case, the usefulness and justification of this research is related to the need to eliminate losses in the filling process, a common problem in biofuel companies, with significant results to reduce costs in biodiesel plants.

2. BIOFUELS SCENARIO IN BRAZIL

According to the National Petroleum Agency (ANP 2018) liquid biofuels, after being produced in ethanol and biodiesel plants, can be transported directly to distributors or

delivered to terminals for later delivery to distributors or exported. In Brazil, in the transportation and movement of fuels, the volume unit (m³ or (L)) is used as the unit of measure. Ethanol has been used in Brazil since 1920 and was established since November 1975 with the Proálcool program.

The National Biodiesel Production Program was launched on December 6, 2004 and is regulated by Law No. 11,097 of 2005. It was initially established the obligation to use 2% by volume of biodiesel mixed with diesel oil (LEITE, 2007). The demand for biofuels has been studied by several authors.

In this direction, Harris et al. (2018) developed a logistic growth curve model and identified that energy consumption in the United States until the year 2040 tends to grow more than the country's power generation, and it is necessary to continue importing fossil fuels (oil) due to increased demand forecasting. The key point well placed in the article is the need for investment, research and innovation to produce as much sustainable energy as possible to reduce carbon dioxide emissions.

In recent decades the use of oil has been discussed by many authors with implications for the environment, as a source of energy and the economy. The main reasons for the global discussion are associated with the greenhouse effect and global warming through the use of non renewable fuels. As for the economy, only time will tell what permanent effects this crisis on the international financial system will have on the energy sector and it is more difficult to predict on the environment. In this scenario, alternative sources of energy generation are highlighted, especially the ecologically sustainable ones.

They are renewable fuels derived from biological raw materials such as ethanol, biodiesel and biogas methane (VICHI; MANSOUR, 2009). In Brazil, the government created the Renovabio program, in which biofuel producers can sell certificates in the market for distributors to meet their targets and carbon emissions (RAMOS, 2018).

As of March 2018, the mandatory percentage of biodiesel in diesel oil is 10% and the evolution of the percentages of the diesel - biodiesel mixture in Brazil is described in Table 1.

Table 1: Evolution of the percentages of diesel blends in Brazil.

Year	Percentage of biodiesel
2003	optional
January / 2008	2%
Julho / 2008	3%
July / 2009	4%
January / 2010	5%
August / 2014	6%

November / 2014	7%
March / 2017	8%
March / 2018	10%

Source: ANP (2018).

Currently biodiesel production includes 14 Brazilian states, with a total production capacity of 8,488.8 million m³ per year with 55 plants authorized by the ANP. The states with the largest biodiesel production capacities are the states of Mato Grosso, Rio Grande do Sul and Goiás. The state of São Paulo has a production capacity of 619.7 million liters per year (BIODIESELBR, 2018).

Figure 1 shows the biodiesel production capacity of the Brazilian states in March 2018. Figure 2 shows the quantities in millions of liters of the volumes auctioned at biodiesel auctions from the beginning of the program until 2018. Importantly, all movements and flows of fuels and biofuels are controlled by the ANP. The purpose of the National Petroleum Agency (ANP, 2018) is to promote the preparation and publication of regulatory technical standards, contracting, supervision, quality and safety of operations in the activities of the oil, natural gas and biofuels industry. The national sourcing agents are (ANP, 2018):

- Distributors: LPG, liquid fuels, asphalt, aviation fuels and solvents.
- Resellers: LPG, automotive (gasoline, diesel, ethanol and CNG) and aviation (kerosene and aviation gasoline) fuels.
- Conveyors-Dealers-Retailers: storage, transportation and resale.
- Lubricants: collector, importer, producer and refiners.

ANTT establishes requirements and details of the correct classification of the product; the adequacy, certification and identification of packages and packages; signaling of transport units and equipment; the documentation; requirements applicable to vehicles and road transport equipment, limited quantity and special provisions (BRAZIL, 2016).

INMETRO is the body responsible for measuring the volumetric capacity of tanks that carry bulk liquids, and according to Ordinance 208 of May 6, 2016. The determined volumes and the maximum permissible errors in verifications are referred to the temperature of 20°C. The maximum permissible error in verification is plus or minus 0.25% of the nominal capacity of each materialized level in the tank or compartment.

The maximum permissible error in volume measured by the tank or compartment

cannot be used as a compensation factor in bulk liquid product transactions. The maximum permissible error in volume measured by the tank or compartment does not include the product volume variation caused by temperature variation. In the checks the position of the reference device must be adjusted according to the height of the measured void space (BRASIL, 2016 p.11).1. Six Sigma Methodology.

According to Salah et al. (2010) the success of an organization depends on how continuous improvement methodologies are implemented and managed. Therefore there are two challenges for managers of organizations, the first is the success in the implementation and then the lasting maintenance of continuous improvement. The Six Sigma methodology began in the mid-1980s at the US company Motorola to reduce quality issues and increase the company's profitability. Figure 1 shows the Biodiesel Production Capacity of the Brazilian states and Figure 2 the Biodiesel Sales Volume in Brazil.

3. SIX SIGMA METHODOLOGY

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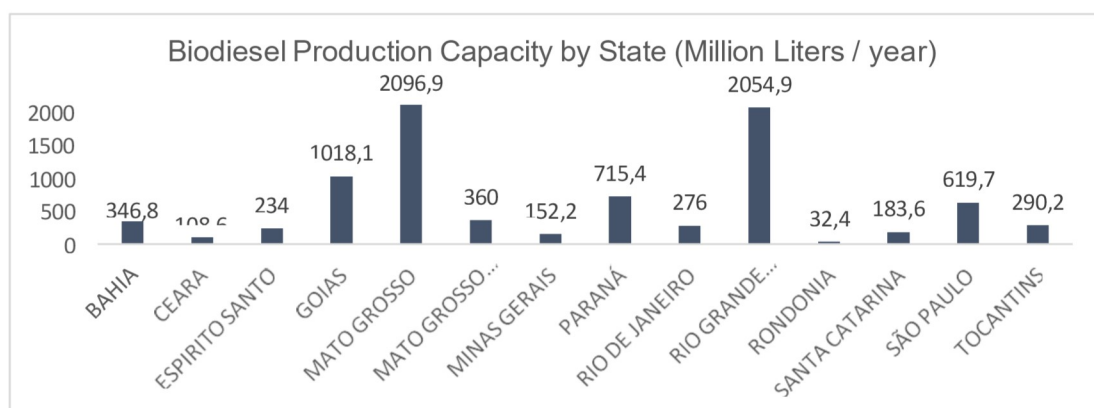


Figure 1: Biodiesel production capacity of the Brazilian states.
 Source: BiodieselBr (2018).

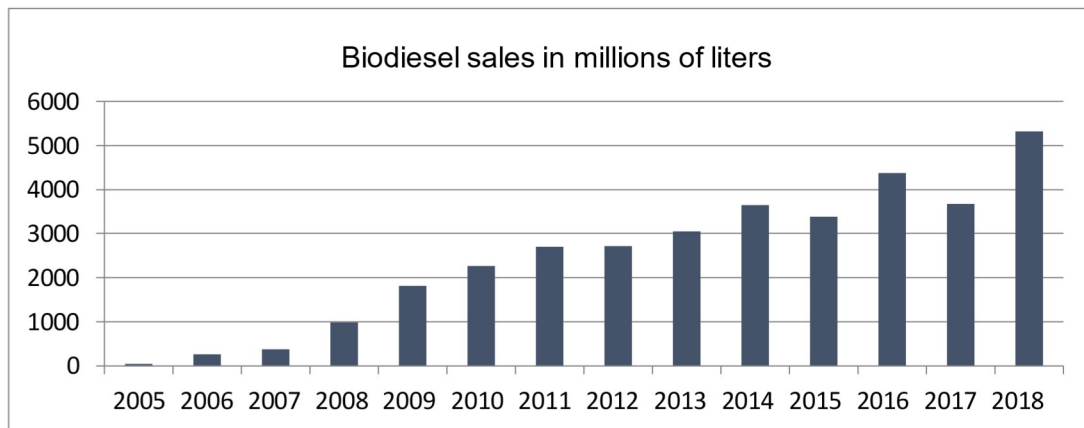


Figure 2: Biodiesel sales volume in Brazil.
 Source: ANP (2019).

At the time, Motorola's top managers admitted that the quality of its products was very poor and the company had several quality programs with little result while the world scenario was evolving in productivity and quality especially in Japanese companies. In 1987, a new technique emerged in Motorola's Communications industry to track and compare customer performance (sigma measurement) and then improve quality (six sigma program). Following the successful development of the Six Sigma (SS) program, Motorola received the Malcolm Baldrige National Quality Award in 1988.

From then on the Six Sigma methodology became known and implemented in other North American organizations. The company best known for the SS program was General Electric (GE), which started SS program activities in the early 1990s where by the year 1999, GE was already saving \$ 600 million a year. According to Hoerl's (1988) definition, the basis of Six Sigma theory is the constant search for the reduction of process variability in order to eliminate defects or failures.

According to Gross (2001), any company and sector can use the Six Sigma methodology to reduce the variability of its processes and the success of the Six Sigma program depends on the team's commitment to the project stages. At Motorola, the Six Sigma method presented itself as a quality improvement program aimed at reducing defects to 3.4 ppm (parts per million) (ANTONY, et al., 2012).

According to Truscott (2003) the application of Six Sigma techniques focuses on improving the efficiency and effectiveness of all processes and movements in all types of companies through the existing quantitative measure when compared with the desired variability seeking to satisfy customers and their effect on the financial performance of the organization.

By Six Sigma methodology in its basic definition is to have only 3.4 defects per million opportunities (DPMO) to produce a defect. Santos (2006) proposed a more complete definition of the Six Sigma methodology: Six sigma is an approach that drives the improvement of business performance and the appreciation of customer satisfaction through the strategic management approach; the application of statistical thinking at all levels of activity; the use of performance indicators; the use of a systematized methodology that delivers varied techniques to evaluate and optimize processes; and learning resulting from people's empowerment and commitment.

The literature shows that the Six Sigma methodology focuses on the statistical and strategic approach to organizations. The statistical part prioritizes processes and the knowledge of variability to propose actions in daily process management activities.

3.1. DMAIC Steps

The great challenge for business managers is to maintain lasting continuous improvement and for this purpose it was decided to opt for the DMAIC method which is the most widely used and found cycle for the application of process control based on the Six Sigma program. Among the tools most used by organizations for continuous improvement is the Deming circle, which defines as continuous improvement the endless cycle in four steps: Plan, Do, Check and Act (PDCA) (BUSHNELL, 1992).

Other continuous improvement techniques are also found in the literature such as the DMAIC and Ford's 8D methodology (MARTIN; OSTERLING, 2014). Salah et al. (2010) state that although DMAIC originated in Six Sigma, this tool can be generalized and used for continuous improvement. DMAIC steps are applied to identify root causes in the Six Sigma methodology program to reduce process failures and product defects, converging to minimize process variability (ANTONY et al., 2012). Antony et al. (2007) suggest for the application of the DMAIC methodology for service processes the following phases:

- **Define:** Define the project problem; understand the problem from the customer perspective; perform process mapping to identify the problem; identify process inputs and outputs; define project charter with project scope and boundaries, resources, deadlines, and team responsibilities; identify the project sponsor; identify all customers (internal and external);
- **Measure:** Determine current performance; measure short and long term capacity; decide what to measure; identify points for improvement;

- **Analyze:** Identify the root causes of process defects; investigate the causes of variability; understand the nature and distribution of data; determine the process variables that may be linked to the defects; quantify financial gains;
- **Improve:** Develop solutions to improve problems and prevent them from recurring; assess the impact of each potential solution using criteria decision matrix; assess the risks associated with solutions; reduce defect rate and validate improvements; reassess the impact of the potential solution;
- **Control:** Develop corrective actions to support process performance; develop and review operating procedures; identify a process owner and establish their role; implement process control plans and determine process capability; close the project and share the main lessons learned; disseminate results internally or externally; recognize team members.

4. DESCRIPTION OF OPERATION OF BIODIESEL FILLING PROCESS

In Brazil, biodiesel is sold through a public auction organized by the National Petroleum Agency-ANP. Scheduling and loading of the B100 is through FOB mode, that is, the customers designated by the distributors are responsible for the freight and the fuel distributors schedule and track the status of truck shipments. At the production plant there is a logistics professional who makes available and also accompanies the timetable so that distributors can schedule their trucks through the Petrobras website <https://www.canalcliente.com.br/> according to the minimum required quantity of pre-shipments. -defined in the plant's contract with the ANP after sale at the biodiesel auction.

The schedule and cadence of biodiesel shipments are proportional to the number of working days of the current month. The carrier, distributor and producer have access to tracking and online status of shipments. The universe of this research work comprises the shipping and billing sectors. In the expedition, the operations are performed by five employees who carry out the loading of biodiesel, glycerine, fatty acid, oil sludge and the unloading of vegetable oils and chemical inputs.

Billing is performed by two employees, first moving the system through the kilogram unit (kg) according to the net mass of the scale, then converting the mass of the balance moved in the company's system to the unit in liters (L) at 20°C through converter spreadsheet provided by the ANP. This company volume invoice (L) is sent to the Petrobrás client channel website which issues a new invoice for the company to deliver to the driver



and follow the trip. The unit used in the transportation of fuels is in liters volume (L). Biodiesel filling processes are performed according to scheduled times and availability of the truck in the company's yard.

After clearance for vehicle entry as scheduled on the Petrobras channel customer website, logistics requests the vehicle concierge to locate the carrier and invites the driver for the initial weighing; on the scale, the driver receives the boarding order and the scale ticket, and then the vehicle is directed to the loading area. The shipment order contains the volume to be loaded according to the scheduling data on the client channel website and empty fields for operators to note the temperature, density, seals and other information necessary for billing to convert biodiesel into liters (L) at 20°C and such information should be included in the invoice observation field.

When the truck arrives at loading, the operators check the vehicles and verify the validity of INMETRO-certified tank capacity calibrations, as well as the CNH and driver's MOPP proof of travel certificate. On the truck the risk labels and safety panels, the hydrostatic test validity of the extinguishers, the obligatory safety items (cone, tools, shims, extinguishers), the condition of the tires and the conservation of the vehicle are also checked the tanks; having fuels they are drained into buckets and disposed of separately at the company.

If the truck fails a checklist item, the operators notify the logistics manager and the vehicle is removed until the situation is regularized. When the vehicle is approved, the driver is asked to position the vehicle on the departure platform to start biodiesel filling. In the loading bay operators must ground the truck tank and check for remaining liquids inside each compartment to ensure non-contamination of the product; The interior of the tank must be clean, dry and odorless for loading approval and otherwise the loading is disapproved, logistics is communicated and the driver must leave the company to wash and / or dry the interior of the tank.

Whenever the vehicle retires or is delayed more than 30 minutes beyond the scheduled time it must be rescheduled on the client channel website. When the filling is approved, the loading operators check the tank to be shipped according to the batch report issued by the quality laboratory. The amount of product available is informed by the control room. To start the filling operation, the operator must place the loading arm in the upper mouth of the tank, enter the accoload the desired volume according to the shipping order

and at the end the filled volume must cover the minimum tank capacity arrow.

The driver can check the level of product loaded with the operator. During loading operators must collect 3 control samples, measure temperature and sample density and record results in shipping order, label samples and deliver one to the driver and the others are stored at the plant for a period of 30 days as a control sample.

After loading is complete, operators must measure the temperature in degrees Celsius ($^{\circ}$ C) within each tank compartment and note the value in the shipping order. Finally, all the mouths of the tank are sealed, next to the shipping order and the scale ticket are attached the report and the envelope with the emergency form that are delivered to the driver and the truck is destined for the final weighing scale. The billing receives the balance ticket and the shipping order information and this data is entered in the converter provided by the ANP.

The converter calculates the converted volume at 20° C in relation to the tank capacity of the truck and compares it with the converted volume at 20° C obtained from the net mass of the scale. This point is the most critical of the whole process and this study. Prior to this project the variability control described in this work was considered correct and allowed for this comparison to vary up to até 500 liters'. From October 18, 2017 this variation will be limited to up to '200 liters'. This means that when the variation was greater than 200 (L) the truck would have to return to the biodiesel removal filling sector and when the variation was less than (-200 (L)) the vehicle would have to return to the sector to add more biodiesel in the tank truck.

5. RESULTS

5.1. 4.1 Data selection

The development of the research in the filling process and the application of the DMAIC method were carried out jointly to the employees of the shipping and billing sectors. Periodic meetings were held with those responsible for billing and shipping teams to receive guidance on the fundamentals of the DMAIC method and together they defined the problem, collected data, mapped and analyzed the process, as well as defined and made improvements.

The idea of the project came up in October of 2017 and initially it was necessary to collect data, as exposed, performed in two stages. In phase 1, the data were collected through the archives of documents from June to October of 2017. This information was

obtained from the documents filed in the company's invoicing, which contained the printed process for each biodiesel shipment. (invoice, converter spreadsheet, balance ticket).

Scale mass, density at 20 ° C, trailer volume, date of shipment were entered into a spreadsheet for further analysis. In the spreadsheet, the volume of the trailer arrow (tank capacity) at 20 ° C and the volume of the mass of the scale at 20 ° C were calculated and compared these two quantities by the difference (Volume converted from the 20 ° C scale to the Volume converted from the 20 ° C arrow) resulting in the change in loaded volume at 20 ° C.

Phase 2 began on October 18, 2017, when there was the first opportunity for improvement, where the billing team was defined as responsible for recording data in a spreadsheet for monitoring variations. At this time, the first goal was defined by a loss variation trailer in liters (L) of biodiesel with allowed variation of ± 200 (L).

In this case, if the volume verified by the billing was more than 200 (L) the vehicle should return to the filling sector for the excess removal and if the volume was less than 200 (L) the truck should return to the shipping sector to add biodiesel.

5.2. Statistical analysis

The data (graphs and statistical analyzes) were obtained and analyzed using Minitab[®] software, version 16. Figure 3 corresponds to the histogram of the data from June to December 2017. In this period there are two data clusters (mix of two distributions) with mean variation of 299.4 (L) and standard deviation of 388.1 (L). From the time series graph from June to December in 2017, also presented in Figure 3, two data clusters were observed as exposed, that is, a mixture of two distributions.

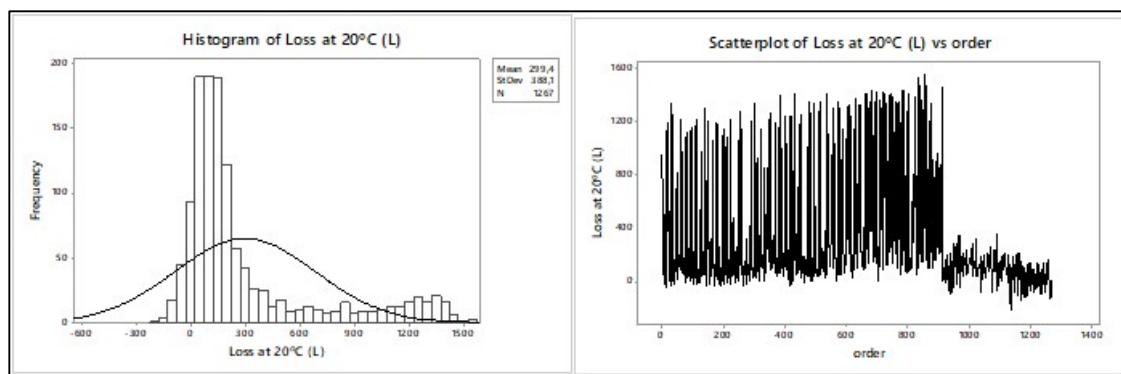


Figure 3: Loss data histogram and time series graph.

Source: Author (2019).

From a descriptive analysis of the data, the identification of the presence of two

data clusters with lower loss averages for the months November and December 2017 was considered as an input for analysis of the “improve” step of the DMAIC method. This result is confirmed in Figure 4 (box-plots and 95% confidence interval graphs for monthly result averages). Figure 4 shows three data clusters with lower loss averages for the months of November and December. From November of 2017 onwards, the “improvement” phase began and the reprocessing index (%) and average monthly loss variation (L) indicators were created.

The reprocessing index refers to the trailers that return to the sector to add or remove product with variation up to 200 (L). With the data from June to December of 2017, a multiple linear regression analysis model was applied with the loss response and the independent variables trailer temperature, trailer volume, mass scale, density at 20° C, correction factor. at 20° C and month.

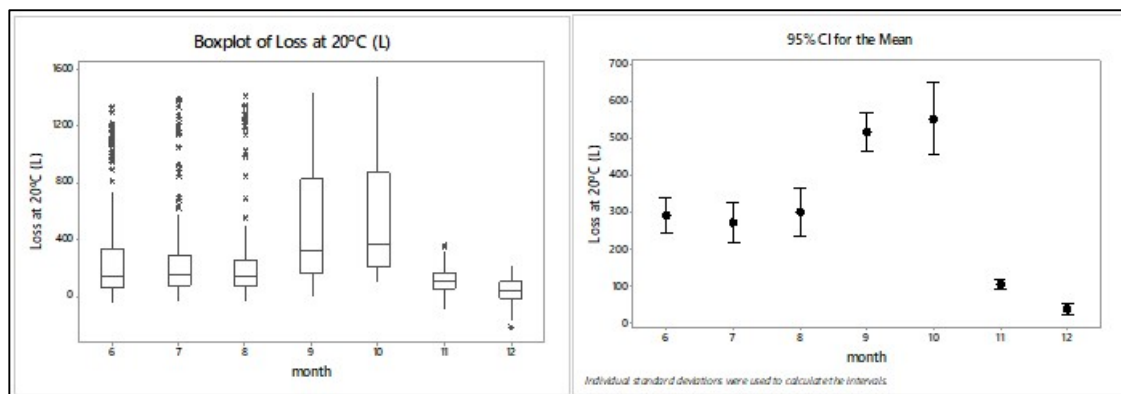


Figure 4: Box-plots and 95% confidence intervals for monthly result means.

Source: Author (2019).

Using Minitab® software, version 16, the least squares estimators (LSE) of the multiple linear regression coefficients as well as the p-values used to test whether correction factors are significant (non-zero regression parameters) are presented in the table. Table 2. The determination coefficient value for this model is given by $R^2 = 99.70$ which indicates an excellent fit. This regression model explains 99.7% of the variability of losses from June to December 2017. From the results of Table 2, it is observed that the significant covariates (p-value < 0.05) in response (loss) are trailer volume (negative estimated value, that is, larger volume implies less loss); balance mass (positive estimated value, that is, higher balance mass implies greater loss) and density at 20% (negative estimated value, that is, higher density 20% implies less loss). From graphs of the residuals of the fitted model (not presented in this article for space saving) it was verified that the necessary assumptions (normality and constant variance of the residuals) were verified.

Table 2: LSE of the regression coefficients (SE: standard-error; T: Student statistics).

Term	Coef	SE	T	p-value
Constant	69171	15715	4.40	< 0.001
Temp. truck	16.5	12.1	1.37	0.172
Volume of truck (L)	-0.98523	0.00141	-698.29	< 0.001
Weighing mass (kg)	1.14705	0.00164	700.03	< 0.001
Density at 20°C (kg/L)	-53008	576	-92.04	< 0.001
Correction factor at 20°C	-23961	15843	-1.51	0.131
month	-0.201	0.319	-0.63	0.529

Source: Autor (2019)

As seen in Figure 3, there are two data clusters. These data were divided in order to improve the analysis and comparison after the beginning of the actions taken through the study with the DMAIC steps and understanding through the statistical tools (EMQ of the regression parameters presented in Tables 3 and 4).

Table 3. LSE of the regression coefficients (period of june to october of 2017; R2 = 99.80).

Term	Coef	SE	T	p-value
Constant	-239550	140252	-1.71	0.088
temp 1	253	108	2.35	0.019
volume 1	-0.98547	0.00156	-630.84	< 0.001
mass balance	1.14759	0.00181	632.39	< 0.001
density 20-1	-62093	4319	-14.38	< 0.001
Correction Factor 1	287956	141844	2.03	0.043
month 1	-0.122	0.496	-0.25	0.806

Source: Author (2019).

From the results of Table 3 (period from June to October 2017), it is observed that the significant covariates (p-values < 0.05) in the response (loss) are: temperature; trailer volume; balance mass; density at 20% and correction factor 1. From the results of Table 4 (period from November to December 2017), it is observed that the significant covariates (p-values < 0.05) in the response (loss) are: trailer volume; balance mass; density at 20% and correction factor 2).

Table 4: LSE of the regression coefficients (period of november to december of 2017; R2 = 97.40).

Term	Coef	SE	T	p-value
Constant	80686	13291	6.07	< 0.001
temp 2	9.0	10.1	0.89	0.373
volume 2	-0.97204	0.00976	-99.64	< 0.001
mass balance 2	1.1308	0.0113	99.93	< 0.001
density 20-2	-52875	834	-63.40	< 0.001
Correction factor 2	-35386	13227	-2.68	0.008
month 2	-3.35	2.06	-1.63	0.104

Source: Author (2019).

Finally, control charts were constructed for both periods (see Figure 5) from which it can be observed that there were many points outside the control range and high variation from June to October 2017 (first panel). This was significantly improved from November to

December 2017 (second panel).

For the months of November and December of 2017, a comparison of control charts was also made in relation to the capacity of the trailers. For this, we analyzed the volumes of trailers smaller than and equal to 36 thousand (L), trucks with capacity between 36 thousand (L) and 48 thousand (L) (Figure 6) and for trucks with capacity greater than 48 thousand (L) (Figure 7).

This analysis indicated that the highest average volume loss in biodiesel filling occurs in trailers with capacity greater than 48 thousand (L), since the average loss was 102.3 (L) while the lowest average was for trailers with capacities up to 36 thousand (L) which presented an average loss of 5.7 (L).

A logistic regression analysis was also performed for data related to the months November and December 2017 with binary response, $Y = 1$ (positive value for loss) and $Y = 0$ (negative value for loss) and covariates considered separately, temperature, volume, scale mass, density at 20%, correction factor 2 and month. Using Minitab version 16 software, maximum likelihood estimators (EMV) were obtained for the regression parameters for each of the logistic regression models.

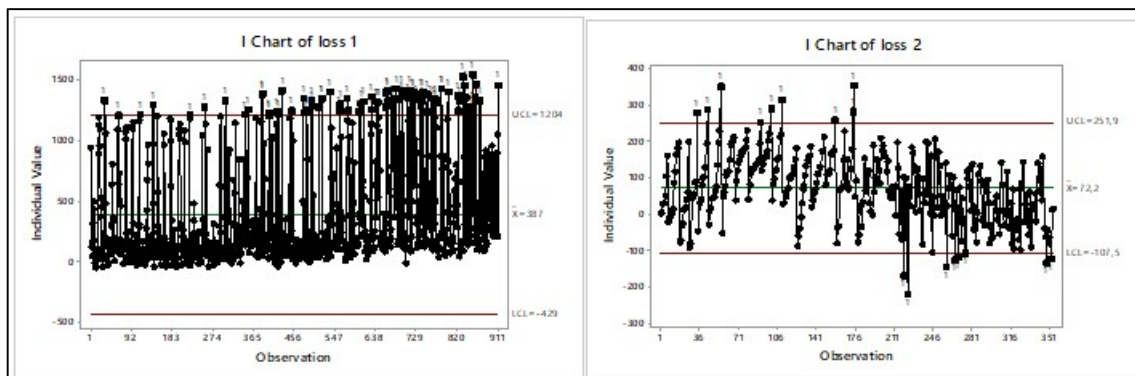


Figure 5: Control charts (June to October 2017 and November to December 2017).

Source: Author (2019).

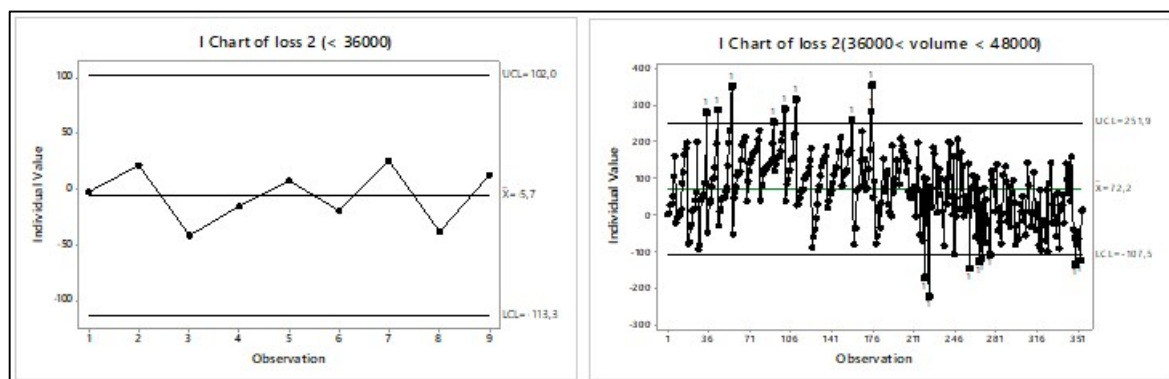


Figure 6: Loss control charts (L) for trailers less than or equal to 36,000 (L) and for trailers

between 36,000 (L) to 48,000 (L)
 Source: Author (2019).

From the results obtained, it was observed that the following covariates show significant effects on the probability of losses (p -value < 0.05): temperature, density at 20%, correction factor and month. Considering binary logistic regression models assuming two covariates together affecting the binary response $Y = 1$ (positive value for loss) and $Y = 0$ (negative value for loss) among the four covariates that showed significance in the models assuming only one covariate, temperature, density. At 20%, correction factor and month we have in Table 5, the maximum likelihood estimators (MLE) and the p -values for the regression parameters of the six logistic regression models considered.

From the results of Table 5, logistic regression models with two covariates temperature/density at 20%, temperature/month, density at 20%/correction factor and correction factor/month have significant effects (p -value < 0.05) of the two covariates in binary response $Y = 1$ (positive value for loss) and $Y = 0$ (negative value for loss).

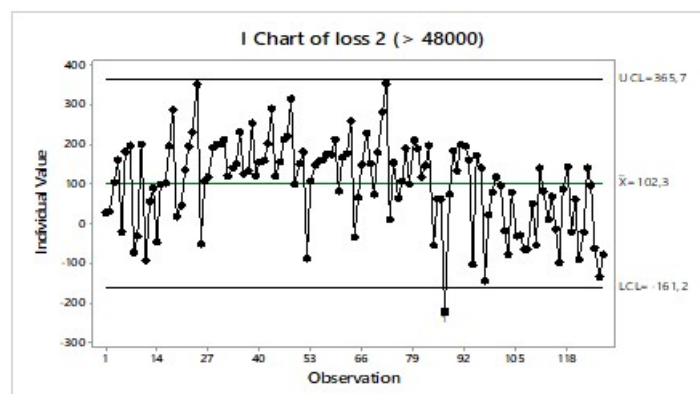


Figure 7: Loss control charts (L) for trucks with capacity $> 48,000$ (L).
 Source: Author (2019).

Table 5: MLE and p -value for the regression parameters of logistic regression models considering two covariates simultaneously in each model.

Covariate	p-value
Covariates temperature and density	
Temperature	0,003
Density	0,003
Covariates temperature and correction factor	
temperature	0,662
Correction factor	0,814
Covariates temperature and month	
temperature	0,002
month	$< 0,001$
Covariate density and correction factor	
Density	0,004

Correction factor	0,005
Covariates density and month	
Density	0,856
Month	<0,001
Covariates correction factor and month	
Correction factor	0,003
month	<0,001

Thus, operators began to observe the trend of variability that resulted in lower average monthly variability with values closer to zero as observed in Table 6. Table 6 shows the data from January to March 2018.

Table 6. Uploads data from January to March 2018.

Data / Month (2018)	January	February	March
Average monthly change (L)	-1	-12	1
Standard Deviation	59	64	59
Number of uploads	281	269	261

Source: Author (2019).

With the results obtained in the study, it was established that from January of 2018 onwards, the target of the average monthly loss variation (L) indicator is 50 (L). Another improvement was to decrease the calibration periodicity of the accoload equipment from 06 to 03 months. Table 7 shows an estimate of the company's average gain from the reduction in biodiesel volume loss from January to March 2018.

The result shown in Table 7 demonstrates the impact on the operating cost of the filling process operation due to the variability (+) in favor of the customer and exceeding the limit required by the ANP prior to the Six Sigma continuous improvement program.

Figure 8 shows surface contours of responses to the predicted probabilities considering the four regression models with the two significant covariates.

From the contours of Figure 8, it is possible to verify the largest and smallest probabilities for losses from each pair of covariates combined.

In the last phase of the DMAIC, a cash management panel was prepared in the loading sector illustrated in Figure 9.

In this panel operators are responsible for noting daily the average volume variation (L) of the previous day's loads; This data is available on the network and entered daily by the billing team.

Table 7: Company average volume gain (L) estimate from January to March 2018

Data	Data / Month (2018)	January		February		March	
		Before	After	Before	After	Before	After
(1)	Montly variability average (L)	387.7	-1	387.7	-12	387.7	1
(2)	Standard Deviation	421.9	59	421.9	64	421.9	59
(3)	Adicional volume (+ for client)	809.6	58	809.6	52	809.6	60
(1) – (2)	Less volume (-for client). In both cases(Before-After) the variability is inside the ANP limit	- 34.2	- 60	- 34.2	- 76	- 34.2	- 58
(4)	Number of Uploads	281	281	269	269	261	261
(3) × (4)	Volume related to loss in volume (L) of the company	227.498	16.298	227.498	14.612	227.498	16.860
(3) × (4) Before – After	Reduction of loss in volume (L)	211.200		212.816		210.638	
$\frac{(3) \times (4)(before-after)}{(3) \times (4)(depois)} \times 100$	Reduction of loss in %	1,298.9%		1,456.9%		1,249.3%	

Source: Author (2019)

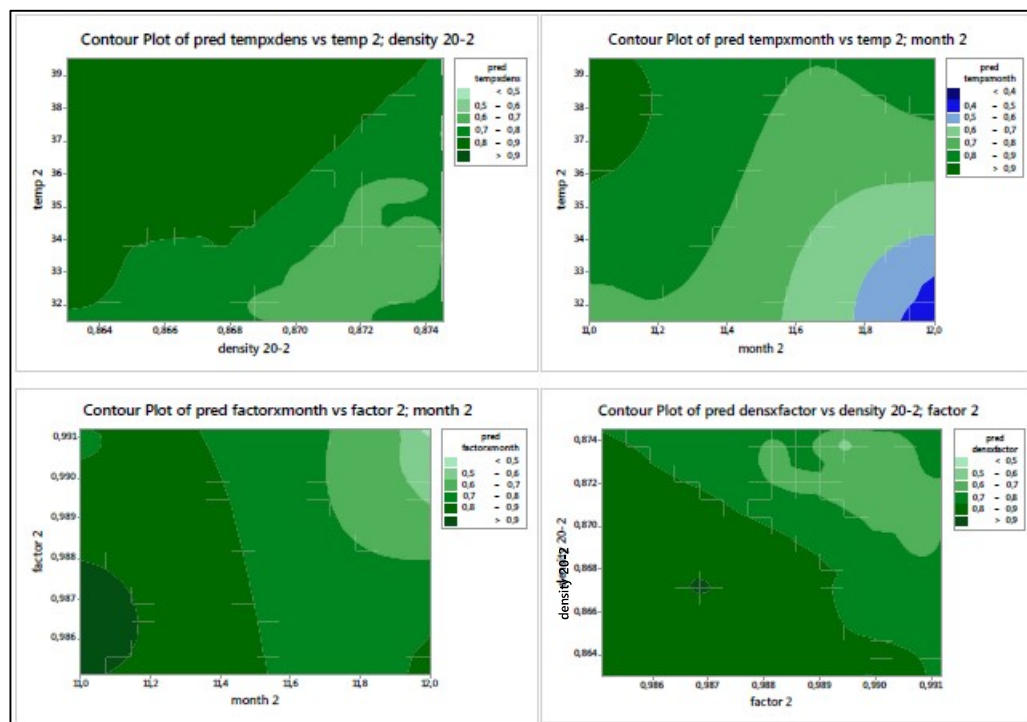


Figure 8: Surface contours of responses to predicted probabilities

Source: Author (2019)



Figure 9: Cash management implemented in the loading room
 Source: Author (2019)

Figure 10 shows the histogram for this period with 911 observations, showing good normality, average loss value close to zero, with a value of -3.187 (L), standard deviation of 60.95 (L). During this period it is possible to observe the last stage of the DMAIC, the “control” phase. Figure 10 also presents the time series for losses from January to March 2018, from which only one data cluster confirms the control of loss variability in the filling process.

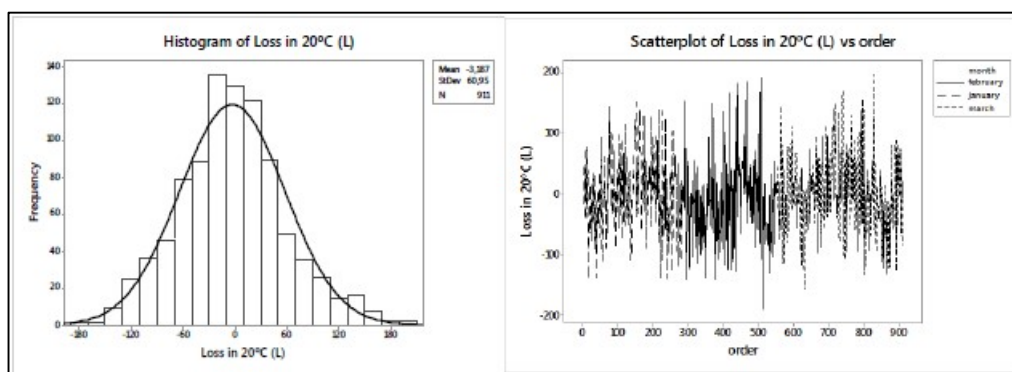


Figure 10: Loss histogram (L) from January to March 2018 and time series from January to March 2018

For the year 2018, quality control plots of loss variation were also constructed (Figure 11), which shows few points outside the control limits and negative average loss variation (l) in the value of -3.2 (L). For the months of January to March 2018, control volume charts were also constructed: trailers less than or equal to 36000 (L); trailers between 36000 and 48000 (L) and trailers larger than 48000 (L) (see Figure 11). From these graphs, better control is observed for trailers equal to or less than 36000 (L). For trailers between 36000 (L) and 48000 (L) and greater than 48000 (L) there are few points outside the control range and no value above 200 (L) positive or negative variation since vehicles with this variation they returned to reprocess. This confirms the vast improvement in loss variability.

6. OVERALL CONCLUSIONS

The objectives of this study were achieved since it was possible to identify opportunities for improvements in the biodiesel filling process through the application of DMAIC. After all the improvements implemented, maintenance and continuity of controls met two important requirements of the Six Sigma quality program: customer focus and financial impact. Analyzing customer acceptance it was found that there were no more complaints from distributors after the project started regarding the control of shipped volume. The improvement project made it possible for the billing and shipping teams to learn and integrate in relation to the filling processes, making them more efficient.

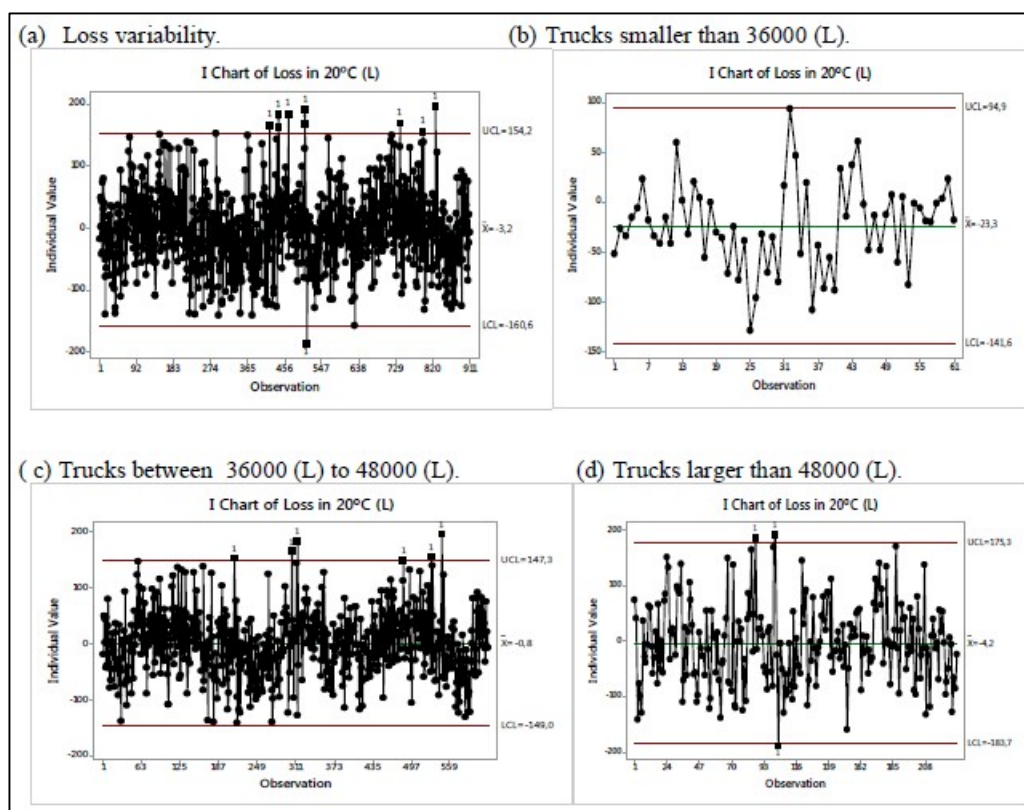


Figure 11: Control graph for loss variation at 20°C and for trucks with different volumes
 Fonte: Author (2019)

These results may be of great interest to companies in the industry. Figure 11 presents the Control Graph for loss variation at 20°C and for trucks with different volumes. The team understood the importance of controls for the knowledge gained during the DMAIC method steps, and statistics and data analysis motivated the team to increasingly improve performance. Shipping and billing employees felt that there was an increase in knowledge about the filling processes and gained insight into the entire process contributing to the continuity of improvements. The study contributed to the creation of indicators in the filling sector providing financial profitability for the company, professional improvement of the

team and efficiency of the filling process.

The results of the study are also confirmed by the usual process capability measurements and graphs as observed in Figure 12 considering specification limits of -200 to 200 (L) of variation, being analyzed from the beginning of the project, that is, corresponding to the three periods: June 2017 to October 17, 2017; October 18 to December 31, 2017 and January 2018 to March 2018).

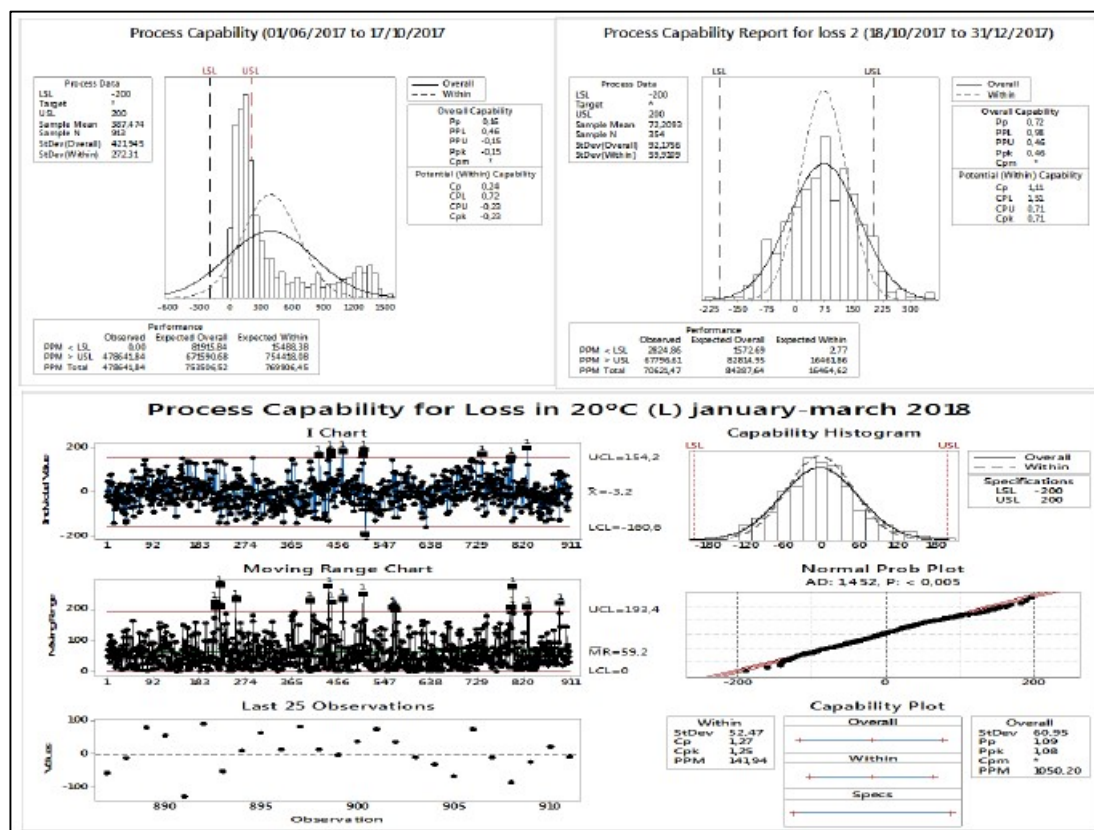


Figure 12: Usual process capability measurements and graphs in the three steps considered
 Source: Author (2019)

From January 2018 to March 2018 it is observed that the process is under full control within specification limits. In addition to reducing the volume losses (L) of the filling process with a direct impact on the operation cost, the effectiveness of the control system as a consequence of the readjustment of the dispatching equipment's measurement procedures, as well as the Training and awareness of employees regarding the actions that must be taken continuously to perpetuate the loss minimization, as mentioned in this paper.

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