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Construction of Control Charts to Help in the Stability and Reliability of Results in an Accredited Water Quality Control Laboratory

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Abstract: Overall, laboratory water quality analysis must have stability in their results, especially in laboratories accredited by ISO 17025. Accredited parameters should be strictly reliable. Using control charts to ascertain divergences between results is thus very useful. The present work applied a methodology of analysis of results through control charts to accurately monitor the results for a wastewater treatment plant. The parameters analyzed were pH, BOD_5, COD, total suspended solids, and total phosphorus. The stability of the results was analyzed from the control charts and 30 analyses performed in the last 12 months. From the results, it was possible to observe whether the results are stable, according to the rehabilitation factor that cannot exceed WN = 1.00 and the efficiency of removal of pollutants that remained above 70% for all parameters. The method of determining the technological reliability and stability of the treatment station using control charts is an efficient tool for detecting any instability in the results. These results help to monitor the results of the analyses more clearly and thus enable a rapid response to possible disturbances and maintain the quality of the analysis control, as well as determining the accreditation entities.

Keywords: water quality; control charts; reliability; wastewater treatment

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1. Introduction

Quality management in accredited laboratories consists of a set of actions based on the international regulatory standard ISO 17025. It is seen as fundamental to maintaining the suitability of its services and products. Organizations are increasingly looking for improvement in their procedures and have tools contributing to achieving their goals. Using statistical controls, continuous improvement, training, and participation reduce the process's variability and consequently increases quality and productivity. Some tools help ensure quality control. Some examples are check sheets, histograms, and control charts [1,2]. For Samohyl [3], quality is ensured by minimizing variations in the characteristics of products and procedures.

Control charts or charts are one of the main statistical tools used to control and monitor processes. They signal the presence of accidental causes and special causes in a process [4]. According to Corrêa [5], every process has variation, and a natural or common cause is within the control limits. In contrast, so-called special causes need more attention because they indicate values outside the control limits and therefore need rapid correction. Using control charts to manage routine analyses allows easy detection of negative trends in analytical work, enabling quick corrective action, reducing out-of-specification results, and consequently avoiding non-compliance [6]. They can be applied in several areas and are used worldwide. A study carried out by Razif [7] proves that the use of control charts contributed to faster detection of anomalies in a daily cycle of analysis and showed similarities in the characteristics of the water quality data of the Surabaya river in Indonesia between 2014 and 2015 for several parameters such as BOD_5, COD, and TSS.

In an analogous study in Brazil using control charts in conjunction with other statistical tools, it was possible to identify the problem in the domestic sewage treatment plant. The treatment did not produce an effluent with characteristics that meet the specifications or release standards of the environmental legislation, indicating the need for restructuring and correction of the efficiency of the process [8].

The International Standard ISO: 7870-1, 2019 addresses the objectives regarding control charts, among the main ones being: indicating if the process is stable, comparing information from samples that represent the current state of the process against the control limits that reflect this variability; estimating the magnitude of the variability inherent to the process; and aiming to determine if the variability of the process has remained stable or if there are oscillations. In a study by Liz and Piotr (2022) [9], they concluded that control charts could be an effective tool for assessing the operation of a sewage treatment plant, allowing the detection of any disturbances during the sewage treatment process in the tested facility. Thus, they enable operators to take appropriate action to remove them quickly and ensure the natural reservoir's water quality. The Shewhart-type control chart is the most used and has broad applicability. In building this model, a preliminary period of subsequent sample analysis is needed to determine the control limits [10]. After this analysis period, statistical treatment is applied to obtain the control charts.

According to Zan et al. [11], control charts are used to monitor whether the process is controlled or not. They have long been used for quality monitoring in the manufacturing process. If only random causes affect the operation, it is considered that the production process is natural or normal [12]. At this time, the control chart tends to fluctuate randomly in the symmetrical coordinate system. The traditional control chart easily detects the abnormity beyond the boundary. Still, it is challenging to identify the abnormity range that usually requires human judgment and is easily affected by various factors.

The problem of water pollution in urban areas is essential and current due to progressive urbanization, aging of ecological infrastructure, and high population density [9,13]. The Member States of the European Union, in agreement with the Water Framework Directive (WFD), are obliged to use and protect their water resources rationally. These include the proper treatment of wastewater.

This study aimed to apply a methodology of analysis of results through control charts, aiming at accurately monitoring the results for a wastewater treatment plant from a slaughterhouse in a specific region of Alentejo in Portugal. The parameters analyzed were pH, biological oxygen demand for five days (BOD_5), chemical oxygen demand (COD), total suspended solids (TSS), and total phosphorus (TP).

According to Reilly et al. [14], slaughterhouse wastewater presents a biological risk to humans and other animals due to the presence of pathogens, pharmaceuticals, and toxic chemicals used for plant cleaning [15]. This harmful potential means that the disposal of slaughterhouse and dairy waste is often subject to local legislation which has been put in place to protect public health [16]. For example, COD in slaughterhouse wastewater often requires a 95% reduction, with similar levels of treatment being required for TP before final discharge into the environment [15]. Currently, the UK dairy processing and slaughterhouse industries use technologies such as chemical dosing, reverse osmosis, anaerobic digestion, dissolved air floatation, and membrane bioreactors to treat their wastewater. They have been obtaining good results [17–19].

The construction of control charts will help guide analysts when there are results outside the control lines, making it possible to reassess the results faster if necessary. This method will assist in managing the quality of the results of the water analysis laboratory.

2. Materials and Methods

2.1. Description of the Study

The School of Agriculture has a water quality control laboratory accredited by ISO 19025, where water quality is analyzed. This laboratory is located in Beja, Portugal. The technological process of the wastewater treatment plant analyzed includes mechanical, biological, and chemical treatment of wastewater. The values of pollution indicators for sewage discharged into the municipality to the slaughterhouse may not exceed the following values: pH—19, BOD_5—500.0 mg $O_2{\cdot}L^{-1}$, COD—1500.0 mg $O_2{\cdot}L^{-1}$, TSS—1000.0 mg$\cdot L^{-1}$, Pt– 25 mg $P{\cdot}L^{-1}$ these values are accorded between the company and the municipality.

The objective was thus to create control charts for the pH, BOD_5, COD, total suspended solids, and total phosphorus parameters in analyzing a wastewater treatment plant from a slaughterhouse in Alentejo for 18 months (2021–2022).

2.2. Methods

The methodology was made according to Figure 1. The laboratory receives the wastewater samples, analyzes them creates a database. The work was applied from this step forward, where the data was analyzed, and the control charts were created.

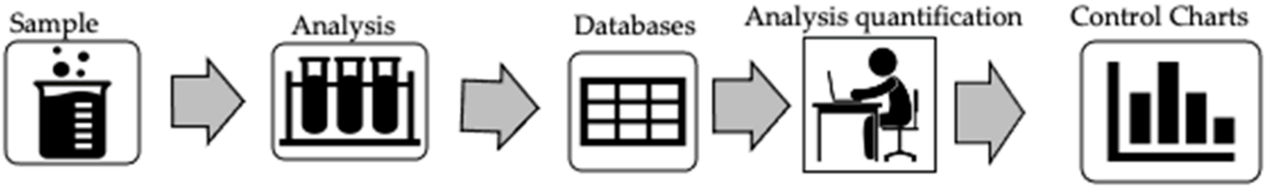

Figure 1. Process method.

The creation of the control charts is structured with an upper control line (UCL), a lower control line (LCL), the process means or target (CL), and the observed points. This statistical tool shows the evolution over time of a characteristic, allowing the identification of the purpose of variations and assisting in a continuous improvement of the process to produce itself according to the specifications keeping the process under statistical control. Statistical control is ensured through the lines of the control limits that allow real-time analysis of the progress of the process.

This represents the statistic related to the variable of interest in case one or more points exceed the control limits, indicating that the process has a problem [20].

For pollution indicators in treated sewage, control charts were with the boundaries of the helplines, and the control lines and the center line were determined considering the tree-sigma rule for the normal distribution N (μ, σ) [9,21,22].

Lower control line (*LCL*):

$$LCL = \mu - 3\sigma \tag{1}$$

Lower warning line (*LWL*):

$$LWL = \mu - 2\sigma \tag{2}$$

Lower helpline (*LHL*):

$$LHL = \mu - 1\sigma \tag{3}$$

Centre line (CL):

$$CL = \mu \tag{4}$$

Upper helpline (*UHL*):

$$UHL = \mu + 1\sigma \tag{5}$$

Upper warning line (*UWL*):

$$UWL = \mu + 2\sigma \tag{6}$$

Upper control line (*UCL*):

$$UCL = \mu + 3\sigma \tag{7}$$

where: μ = average of the analyzed values, and σ = standard deviation of the analyzed variable.

To analyze the technological reliability and stability of the wastewater treatment station in Alentejo, Portugal, with the use of control charts, some specifics were necessary; the coefficient of technical reliability was used, as in other authors [9]:

$$WN = \frac{x_{sr}}{x_{dop}} \, [-] \tag{8}$$

where WN = plant reliability factor $[-]$, x_{sr} = average value of the analyzed pollution index in treated sewage $[\mathrm{mg \cdot dm^{-3}}]$, and x_{dop} = permissible value of the analyzed pollution index in treated sewage $[\mathrm{mg \cdot dm^{-3}}]$.

To determine the effectiveness of wastewater treatment, it was calculated according to the following formula:

$$\eta = \frac{S_s - S_0}{S_s} \times 100\% \tag{9}$$

where: η = reduction of a particular pollutant index in treated sewage [%], Ss = value of the pollution index in raw sewage $[\mathrm{mg \cdot dm^{-3}}]$, and So = value of the pollution index in treated sewage $[\mathrm{mg \cdot dm^{-3}}]$.

To verify interruption or instability of the effluent treatment process from control charts, we can use the following parameters according to Andraka [23]: eight consecutive points on one side of the central line, one point outside the control limits, two of the three points outside the $\pm 2\sigma$ warning lines and four of five consecutive points beyond the $\pm 1\sigma$ extension lines [9].

Control charts have three fundamental objectives: reducing variability, monitoring, and estimating process quality parameters [24]. In constructing control charts, it is essential and valuable to distinguish the two phases of implementation and construction.

In phase 1, a set of process data is analyzed retrospectively to understand the variation of the process over time, evaluate the stability of the process, and model the performance of the process under control. This last step is usually carried out by estimating the parametric model in phase 2, in which the process has been previously estimated. Phase 1 thus corresponds to a retrospective check of the process where the experimental control limits are calculated, while phase 2 concerns monitoring the process itself [25].

Phase 2 begins after collecting a set of process data under stable conditions and is representative of the performance of the process under control. In phase 2, a control chart is used to monitor the process, comparing the sample statistics for each successive sample as extracted from the process with the control limits [26].

3. Results

The results show the control charts for the inlet and outlet effluents of an existing wastewater treatment plant in a slaughterhouse in Alentejo, Portugal.

Figures 2 and 3 show the inlet and outlet effluent behavior for the pH parameter. The control charts show the variation during the 30 days of treatment for wastewater.

It can be seen that after treatment, the results are stabilized; this is the possible interpretation of the analysis definition of the control lines. Figure 4 shows the COD inlet results; compared to Figure 5, the COD outlet shows an improved system. For Figure 5, there is a first point close to the LWL, but it is not necessary to take control because the next point is far away from the LWL. Only two points cross the control lines, and this behavior does not jeopardize the treatment outcome.

Analyzing the results for Figures 6 and 7. it is possible to see the same stability of the results at the outlet of the treated effluent with only one point crossing the UCL control line. Due to all the results obtained having this same point outside the standard, it is believed that the treatment was ineffective on this day.

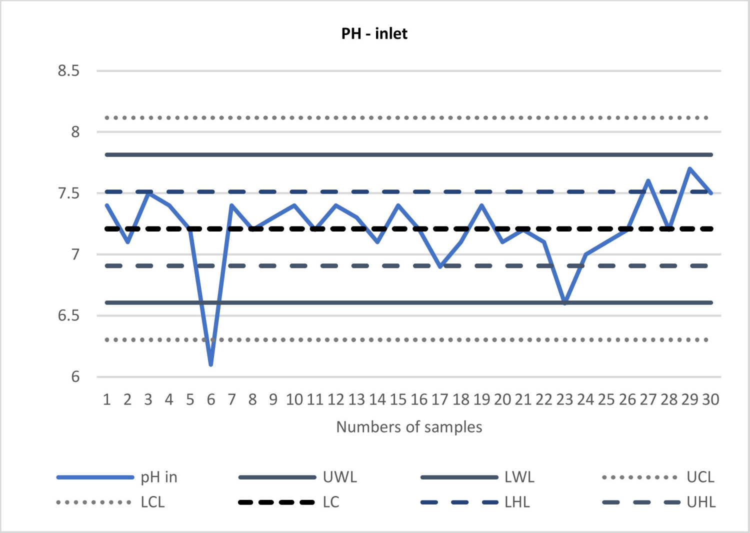

Figure 2. pH inlet.

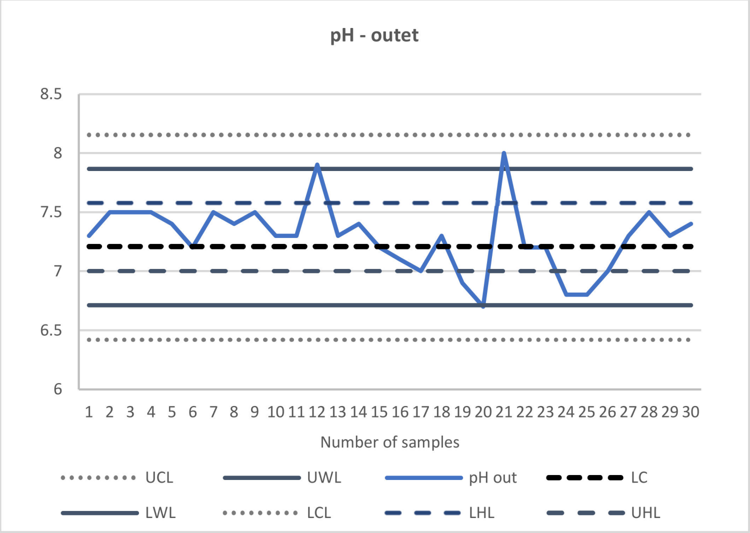

Figure 3. pH outlet.

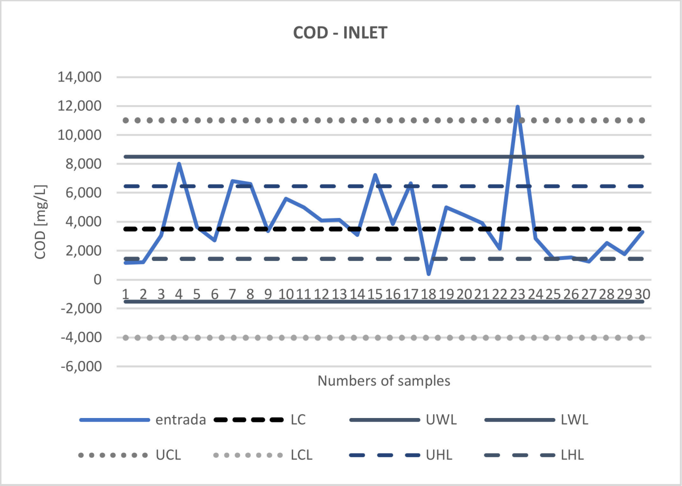

Figure 4. COD inlet.

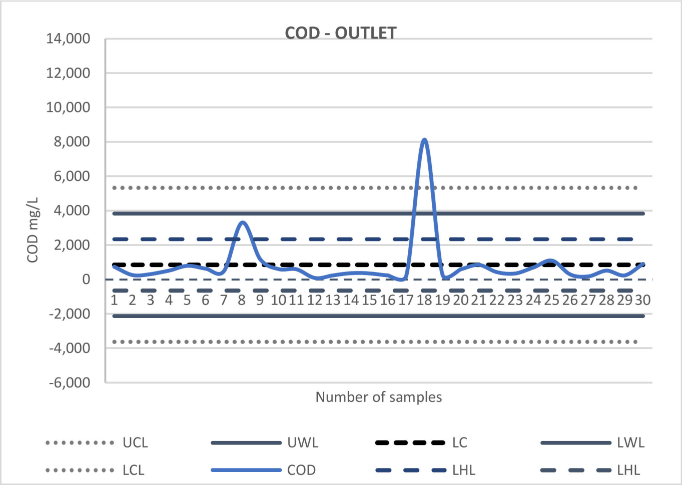

Figure 5. COD outlet.

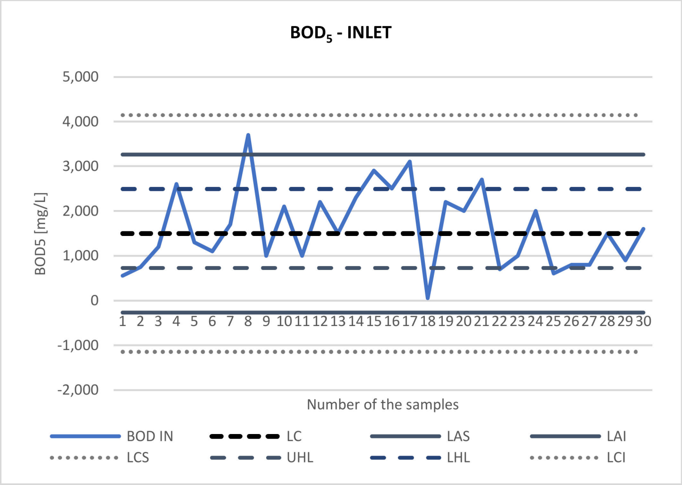

Figure 6. BOD$_5$ inlet.

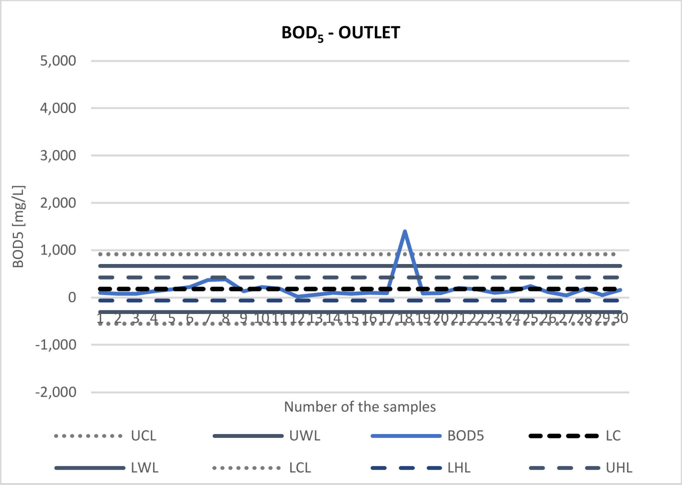

Figure 7. BOD$_5$ outlet.

For total phosphorus results, Figures 8 and 9 show similar results. The resulting line remains stable and only crosses the UHL line once; the results stay close to the LC control line across all samples.

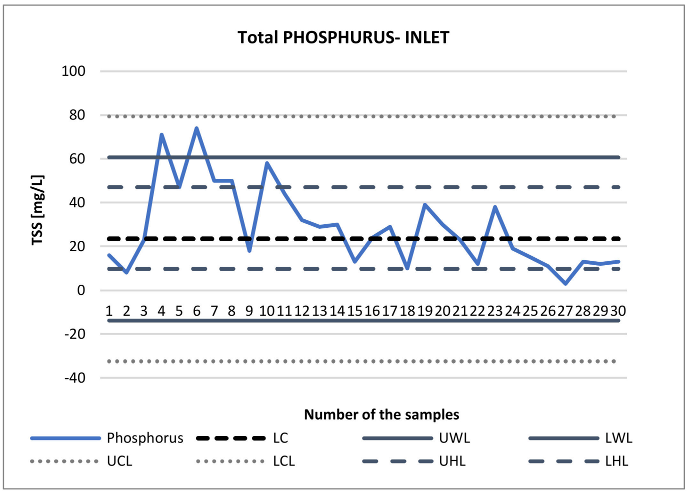

Figure 8. Total phosphorus inlet.

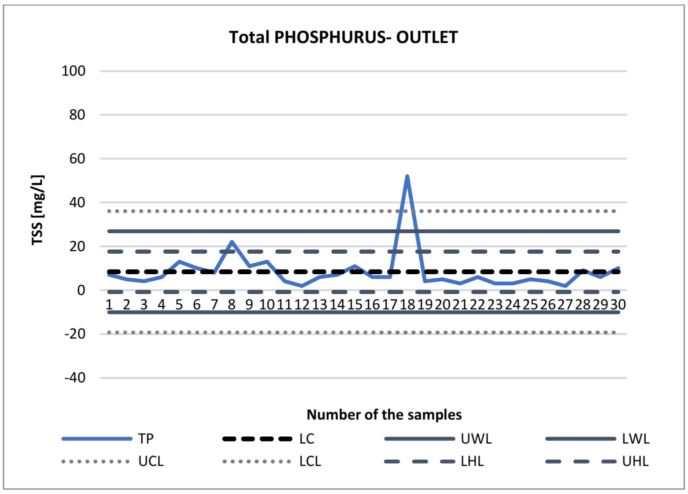

Figure 9. Total phosphorus outlet.

For the TSS inlet chart (Figures 10 and 11) as a sample to reach the UWL line when observing the TSS outlet chart, it can be noted that the post-treatment results are stable, except on three occasions. In this situation, it may alert the company to a possible problem in the treatment in that specific period.

All control charts obtained were analyzed, and most points are under statistical control in Figures 2–11. In accordance with Montgomery [26], only rule 1, the presence of special causes of variation, was evaluated, and there was only one particular cause. With the process under statistical control in phase 1, it passes to phase 2, which consists of monitoring the process.

The purpose of using the rules is to increase the sensitivity of control charts. However, care must be taken when using a set of rules, as an excessive number of false alarms may occur. The higher the number of rules to be used, the greater the number of false alarms [26].

Table 1 shows the technological reliability coefficient against the average efficiency of the analyzed pollutants removal in the sewage treatment plant. It can be seen that the efficiency of reduction from treatment is good enough to reach more than 70% in all parameters analyzed, with better results for the BOD_5 indicator, reaching 89%.

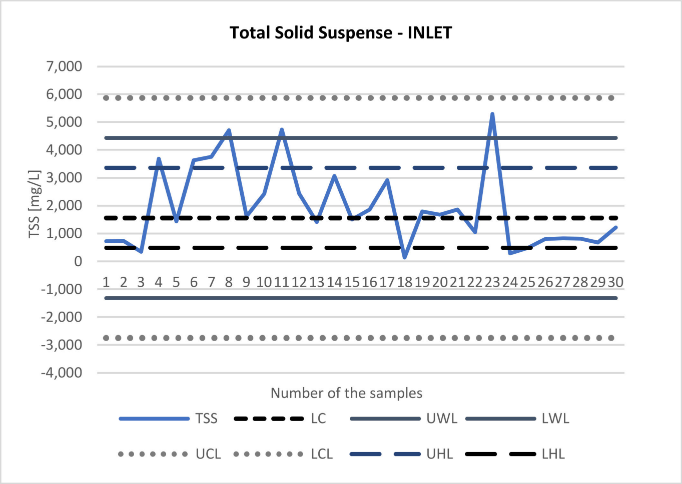

Figure 10. Total suspended solids inlet.

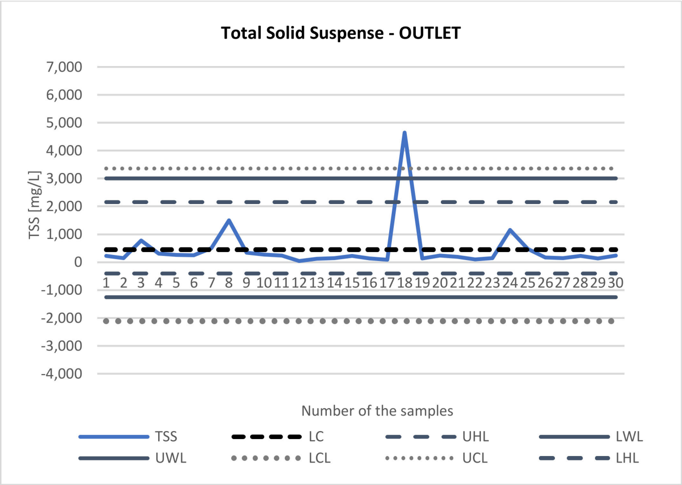

Figure 11. Total suspended solids outlet.

Table 1. Value of the technological reliability coefficient against the average efficiency of the analyzed pollutant removal in the sewage treatment plant in the Alentejo slaughterhouse.

Indicator	BOD_5	COD	TSS	TP
Reliability coefficient WN	0.37	0.57	0.45	0.84
Reduction Efficiency η (%)	89	78	76	70

Explanations: BOD_5 = biochemical oxygen demand, COD = chemical oxygen demand, TSS = total suspended solids, TP = total phosphorus.

The BOD_5 parameter showed the lowest rehabilitation coefficient (WN) of 0.37 and, consequently, the most excellent technological reliability, which indicates a very satisfactory result for this parameter. For COD and TSS parameters, the rehabilitation coefficient showed results of 0.57 and 0.45, respectively, indicating values that can be considered reasonably good. In the case of TP, the indicated value is within the legislation in force. However, it is the highest value for the rehabilitation coefficient and consequently has the lowest treatment efficiency among the analyzed parameters since the indicators are inversely proportional. A study by Mlyński [27] used the indicator to prove the technological reliability of the treatment plant, where it obtained results below 1. However, this was slightly higher when compared to the results obtained in the present study.

4. Discussion

Although there were some lower deviations in the LC, most of the results were stable after the treatment, which can be seen from the construction of the control charts.

The graphs demonstrate stability after water treatment, except for occasional events in a given period. These points that suffered oscillation may indicate a lower treatment efficiency because this anomaly is in the same period and different parameters such as COD and BOD_5. For the TSS input graph as a sample to reach the UWL line by looking at the TSS output graph, it can be noted that the post-treatment results are stable, except for three points that, in this situation,, may alert the company to a possible problem in the treatment in that specific period. Another potential cause of this difference in results may be related to the error in sample collection.

In sample 18, all parameters were altered beyond the control lines UCL, UWL, and UHL, except for the parameter pH. This behavior leads us to believe that the wastewater treatment plant had some technical problems that led to the inefficiency of treatment or stoppage of the process on this day. It is verified that only with this sample we obtained this change. In the previous and subsequent samples, the results were satisfactory within the control lines, as expected. According to Nagendra and Rai [28], the chart series size, sample size, and sampling interval are the three main factors in detecting changes efficiently. Thus, for further investigation of the cause of this variation, a larger number of samples is required. In a similar study Moore [29] suggests that in relation to the Shewhart chart of averages (x^{-}), the errors depend on the degree of non-normality and the sample size (or subgroup). These errors can be reduced by using a larger sample size.

Studies related to wastewater treatment from slaughterhouses indicate the need for efficiency in treatment due to environmental risks and human health. When comparing the results obtained by Really et al. [14] in their studies for COD (75%) and BOD_5 (85%), the results obtained in this study show a good removal efficiency for values of 78% and 89%, respectively. These data assist in analyzing results compared to the values established in the decrees of laws in force for Portugal, such as 236/98 [30].

According to Decree-Law No 236/1998 [30], phosphorus in wastewater may have a concentration of 10 mg P/L in the forms of orthophosphates, polyphosphates (P_2O_7), and organic phosphorus [31]. Wastewater treatment is carried out to avoid risks to public health, pollution of water resources, and the environment in general. It is essential to be able to control these results. The work performed by [32] created control charts similar to this work and obtained values of 95% for removal efficiency and 0.49 for the rehabilitation coefficient for TP. The values are better than those found in Table 1 for the TP, and a justification for these results is the origin of the residual water. Although there is a similarity in the construction of the control charts, we cannot compare the results since they are wastewater from different sources.

These results help in the analysis of data in the laboratory, thus creating a greater control, so when a result is outside the line control (LC) in the respective control chart, it can already be said that there was an error in the treatment process, facilitating the search for corrections.

5. Conclusions

The study's main objective was based on the application of statistical control of the process of analyzing wastewater from a food industry company, more specifically in the meat sector (a slaughterhouse). From the experimental point of view, it started by diagnosing the work of data control from the results of the construction of control charts to identify the main problems and situations to be corrected and monitored.

Most of the results of the studied parameters in question oscillated around the central line and did not show any crossing of the control lines or grouping of samples below or above the characteristic lines.

The control charts were effective for what was proposed, and variations could be observed in the analyzed period. These results confirm that the treatment is stable according to the rehabilitation coefficient and treatment efficiency results.

It can be observed that on the 18th day of collection, all parameters exceeded the UWL. This behavior is due to a possible isolated failure of treatment. It can be observed in all control charts constructed, thus concluding the objective of the work where the rapid detection of variations in small scales allows the identification of such causes of variability. However, it is possible to state that all points are under statistical control, and this chart can then be used in the laboratory routine.

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Abbreviations

BOD_5	Biological oxygen demand
CL	Centre line
COD	Chemical oxygen demand
LCL	Lower control line
LHL	Lower helpline
LWL	Lower warning line
TP	Total phosphorus
TSS	Total solid suspense
UCL	Upper control line
UHL	Upper helpline
UWL	Upper warning line
WFD	Water framework directive

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