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Statistical Appreciation of Pellets Quality for Measured Characteristics

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Abstract
The purpose of the work is to highlight two statistical methods for assessing the quality of the pellet batches, respectively the s method and the R method. The practical applicability of the two methods referred to the unit density of the pellets and the ash content, were determined on the basis of some standardized methodologies. The unit density of the pellets was used both to exemplify the s method and the R method, so a total of 3 individual case studies were analysed. The work can be a support for pellet producers from all over the world to show how the quality of pellets can be determined from the point of view of an individual characteristic. Also, in the context of some ambiguous definitions of quality, the paper applies statistical methods to find the most appropriate definition of pellet quality.

Keywords
statistical quality, pellet, briquette, s method, R method

1. Introduction

The statistical verification of the batches of pellets is a verification by sampling, respectively a small amount randomly extracted from the batch is checked, and the result obtained on the sample is extrapolated to the level of the entire batch. The main advantages of statistical control are given by the reduced checking (about 5-10%) of the products, but especially by the drastic nature of the rejection of a batch of products, when the top management will have to take urgent measures to remedy the production that generated these defective products [1].

A measurable characteristic is a characteristic that has a unit of measure, such as the effective density of pellets, ash content, calorific value, etc. Checking the quality of the batch of pellets is done from the point of view of each measurable characteristic analysed, which is why an analysis must be carried out to identify the one or the most important quality characteristics of the analysis batch. Measurable characteristics are those characteristics of quality that can be quantified quantitatively and that present a unit of measure. The elements of a verification plan for measurable characteristics are the sample size n and the acceptance constant k. The effective sample is represented by the number of products extracted from the batch in order to check their quality. The acceptance constant k is a constant dependent on the value of the acceptable quality level (AQL) and is used in making the decision to accept or reject product lots. From this point of view, the acceptance constant is defined as the lowest value of the quality parameter, considered acceptable for the characterization of the manufacturing process at the supplier [2-5].

A product quality control plan for measurable characteristics includes two elements, namely the effective sample and the acceptance constant (n and k). For this, the following determinations are necessary [6-9]:

- establishing the invariant elements of entry into the standard: N, Nv, AQL, the type of sampling and the verification method;
- the extraction from the standard of the constituent elements of the plan, namely the effective sample n and the acceptance constant k (or the decision graph).

Before starting a product quality control plan, it is recommended to carry out the following verification operations:

- to confirm that the distribution of quality characteristics is considered normal (Gaussian curve) and that the production from which the batches originate is continuous and constant;
- if the manufacturing process at the supplier is statistically stabilized, i.e., the mean square deviation is stable and homogeneous;
- to confirm that a level of verification (Nv) has been established;
- to confirm that a value of AQL has been established between the two parties and that it is found among the values given by the standard, because approximations or extrapolations of its values are not allowed;
- check if the double tolerance limits are separate or combined, and for the separate ones if the two values of AQL (acceptable quality level) have been established.

The choice of the verification method (s, σ, R) is made on the basis of economic considerations, the effective samples and the consistency of the information obtained. The σ method is the most economical in terms of sample size, but before this method can be used, it is necessary to determine the value of σ, and for this the manufacturing process at the supplier must be stable and statistically stabilized. Considering that this cannot be known from the beginning, the verification process begins by applying the s or R method. The s method assumes a slightly smaller sample than the R method, but the necessary calculations are more complex. If at a given moment, from the information provided by the statistical control sheets, it results that the values of s or R is under control, then the authority responsible in the field of product quality can adopt the continuation of the verification with the σ method. This does not mean that the values of s or R should not be calculated, because it needs to know if the variability remains under control, by making their statistical control sheets [6-10].

The choice of the verification level is made according to the level of consistency of the information, for the measurable characteristics there are three usual verification levels I, II, III and two special levels S3 and S4. Determining the level of verification and the value of AQL is the responsibility of the responsible authority and depends on several factors. When possible, it is recommended to consider the balance between costs and added value, respectively the implications that non-compliant products have on the costs of the economic unit.

In the absence of other regulations, it is indicated to use the usual verification level II, level I being used when less consistent information is sufficient, and usual level III when a higher consistency of information is required. The special levels S3 and S4 are used when it is possible to analyse only small herd samples, but relatively high risks must be tolerated, due to the less consistent information provided by the sample. In the general mode, the choice can be graded, i.e., first the value of AQL is chosen, then the actual sample and only in the last place the limit quality (LQ) [11-15].

The quality parameter q is a function of the specified tolerance limit (upper or lower), the mean and the mean square deviation of the lot, namely:

- in the case of the lower tolerance limit (Eq. 1):

$$q_i = (m - L_i)/s \tag{1}$$

- in the case of the upper tolerance limit (Eq. 2):

$$q_s = (L_s - m)/s \tag{2}$$

where: q_s and q_i are quality parameters, in the two cases; m – arithmetic mean of the lot; L_i and L_s – the lower and upper tolerance limits; s – mean square deviation of the batch of pellets.

The decision statistic is the inequality relationship between the quality parameter and the acceptance constant, being dependent on the tolerance limits, as well as the estimates of the average and the mean square deviation of the lot. Dual Separate Tolerance Limits is a term used when two tolerance limits (lower and upper) and two distinct AQL (Acceptable Quality Level) values are specified simultaneously, one for each of the two limits. Combined Dual Tolerance Limits is a term used when two tolerance limits (lower and upper) are specified simultaneously and when the specified value of AQL applies to the sum of the two limits and corresponds to the defective fraction (sum of the fractions of the two limits). Separate tolerance limits refer to individualized tolerance limits, either upper or lower.

2. Materials and Method

2.1. Method s

The s method of quality control of pellets lots is a method that allows a decision to be made regarding the acceptance of a lot by using the estimate of the mean square deviation of the lot using the mean square deviation of the sample s.

The choice and operation of a verification plan using the s method involves the following main operations:

- with the verification level Nv and the number of batch N, the code letter LC is determined;
- with the code letter and the value of AQL, the two elements of the verification plan are chosen, respectively the effective sample n and the acceptance constant k;
- sample n is extracted from the lot;
- the quality characteristic is measured for each element of the sample;
- the arithmetic mean and the mean square deviation of the measured values of the sample are determined;
- if the average of the sample is outside the specified tolerance limits, the lot could be rejected without calculating the mean square deviation s, but it is necessary to determine it in order to follow the evolution of the quality (note that the reciprocal of this statement is not true, respectively if the arithmetic mean is between the tolerance limits, the batch is not accepted immediately, and the comparison between the quality parameter and the acceptance constant must be made);
- on the basis of the calculated values (mean, standard deviation and quality parameter) and the elements of the verification plan (constant or acceptance curve) the acceptance or rejection of the lots is determined.

2.2. R method

The R method of quality control is a method that allows making the decision to accept or reject the batch by approximating the mean square deviation of the batch σ, by means of the average amplitude of the subgroups of the values of the measurable quality characteristic R, or of the estimator R/*c*, where *c* is a correction coefficient. The subgroup of a string of values of the measurable characteristic is a set of five consecutive products. The amplitude of a string of R values is the difference between the maximum and the minimum value, respectively: $R = x_{max} - x_{min}$. The average amplitude of several amplitudes is the arithmetic mean of all subgroups in the analyzed value string. For the sample consisting of less than 10 products, the average amplitude is the amplitude of the entire sample, and for samples larger than 10 products, first the sample is divided into groups of five pieces, after which the amplitude of each group is determined, and finally the mean amplitude as the arithmetic mean of previous individual amplitudes.

The R method for checking the quality of the products is based on the principles of the s method, with the specification that the mean square deviation of the batch is estimated using the average amplitude of the checked sample. Using instead of the actual values of the lot (*m* and σ), their estimates R and *m* (or R/*c*, where *c* is a correction coefficient) the estimate *q* of the lot quality parameter, Q is obtained. In this way the standard gives us the value of the acceptance constant k, which guarantees with a high known probability the acceptability of the lot when $q \geq k$. The correction coefficient c has values between 1.9-2.3, depending on the type of verification plan and the effective sample, respectively it will increase with the increase of n. Also, the R method works with an initial condition, respectively the maximum average amplitude MAR to be greater than s. This MAR can be calculated with the following relationship (Eq 3):

$$MAR = F \cdot (L_s - L_i), \tag{3}$$

where *F* is a correction coefficient with values between 0.3-1.3, depending on the actual sample and the acceptable quality level.

The way on this method works is given by the following steps:

- with the verification level and the lot level, the LC code letter is determined;
- with the code letter and AQL, the verification plan is obtained, respectively the duplicate *n* and *k*;
- with the data provided by the measured sample, the average amplitude is calculated as follows:

- if the sample consists of more than 10 products, the results are divided into groups of five, in the order of verification, the amplitudes of each group are calculated and finally the average amplitude as the arithmetic mean of all the calculated amplitudes;
- if the sample is smaller than 10 products, the results are no longer divided into groups, considering that the average amplitude is the amplitude of the sample.

The acceptance-rejection criteria are similar to the previous methods, respectively, the parameter value of quality is compared with constant of acceptance (Eq. 4).

$$\frac{L_s - \bar{x}}{\bar{R}} \geq k_s \qquad (4)$$
$$\frac{\bar{x} - L_i}{\bar{R}} \geq k_i$$

The graphic method for the case of unique limits is solved by drawing on graph, in a system of coordinate axes with R abscissa and m ordinate, the following two lines (Eq. 5):

$$\bar{m} = L_s - k_s \cdot \bar{R} \qquad (5)$$
$$\bar{m} = L_i + k_i \cdot \bar{R}$$

2.3. Determination of pellet density

The density of the pellets was determined for each pellet separately, as a ratio between their mass and density. Before this test, the pellets were conditioned at a temperature of 20 °C and a relative air humidity of 55% for a period of 48 hours, in order to obtain a moisture content of the pellets of 10%. Also, the ends of the pellets were ground, after which the length was determined, in order to obtain a straight cylinder. If it is taken into account that each pellet has a cylinder shape, the general relationship for determining their unit density was the following (Eq. 6):

$$\rho = \frac{4 \cdot m}{\pi \cdot d^2 \cdot l} \cdot 10^6 \ \ [\text{kg/m}^3] \qquad (6)$$

where: *m* - mass of the pellet, in g; *d* - diameter of the pellet, in mm; *l* - length of the pellet, in mm.

2.4. Determination of ash content

The ash content of the pellets was determined on shredded material, which resulted from sorting the sawdust with a 1×1 mm sieve. This material was dried in a Memmert laboratory oven (Germany) at a temperature of 105 °C, until the anhydrous state. After that, the material was placed in a thin layer on a crucible and weighed on an analytical balance with a precision of 3 decimal places. Calcination was carried out in a Pro-Therm calcination oven (Ploiesti, Romania), at a temperature of 700 °C, for a period of 40 minutes. The calcination operation was considered finished when the material had a light grey colour and no more sparks were observed above the crucible. After cooling in the desiccator, the crucible with the ash was weighed again, and by the difference to the mass of the crucible, the mass of the calcined ash was found.

The calculation relationship of the calcined ash content was as follows (Eq. 7):

$$A_c = \frac{m_s}{m_{cc}} \cdot 100 \ \ [\%] \qquad (7)$$

where: A_c – ash content, in %; m_s – mass of sample, in g; m_{cc} – mass of calcined ash, in g.

3. Results and Discussion

The results will be exemplified for three individual cases, the first representing the s method and the case of the lower tolerance limit in the case of pellet density, the second one for the R method and the case of the lower tolerance limit for the pellet density, and the third will be about the R method in the case of content of calcined ash.

3.1. Method s, case of lower limit of tolerance for pellets of energetic willow

This method refers to the use of the standard deviation s, applied to the analysis of the density of energetic willow (*Salix viminalis*) pellets, the numerical and graphic method. The application corresponds to the lower tolerance limit of 1120 kg/m^3 according to the Code of Good Practice /BioGen/UK and DIN plus/Germany. For this it had a batch of 1200 pieces of energetic willow pellets that are subject to statistical verification, in order to accept or reject it. According to the statistical control standards for measurable characteristics depending on the level of the batch 501-1200 and the usual verification level II, the letter J is found for indexing the verification plan. By adopting an acceptable quality level AQL = 4 and normal verification to find the two elements of the verification plan: the batch level n = 35 and the acceptance constant k = 1.39. At this moment there are all the elements to start the activity of statistical verification of the quality of the batch of pellets from the point of view of the unit density. Therefore, a sample of 35 pellets is extracted from the batch and the density is checked, obtaining the values from the Table 1.

The problem of acceptability or rejection of the batch of analyzed pellets can be solved both numerically and graphically. The numerical procedure is solved based on the data from the Table 2.

Table 1. Density values of energetic willow pellets

990.5	1197.6	1103.4	1018.3	1149.2	1043.9	1219.4
1006.2	1158.4	1116.1	1020.1	1089.4	1138.7	1070.4
1031.8	1270.6	1096.2	1213.1	1156.4	1008.0	1103.3
1078.4	1028.6	1018.9	1230.6	1135.3	1154.4	1117.2
1027.2	1063.7	1069.8	1040.1	1093.8	1030.8	1001.9

Mean: 1101.01 kg/m^3
Standard deviation: 78.4 kg/m^3

Table 2. The numerical decision procedure for the application corresponding to the method s

No.	Data	Values
1	Sample size (from statistical standards)	53
2	Sample mean (from calculation)	1101.01 kg/m^3
3	Standard deviation of sample (from calculations)	78.4 kg/m^3
4	Lower tolerance limit (from pellet standards)	1120 kg/m^3
5	Acceptance constant (from the statistical standard), k	1.39
6	The quality parameter (from the calculation), q	0.24
7	Decision: Quality parameter q = 0.24 < Acceptance constant k = 1.39. The batch is rejected.	

From Table 2 it can be seen that, although the arithmetic mean of the sample of 1101.01 kg/m^3 is very close to the lower tolerance limit of 1120 kg/m^3, the batch is rejected mainly due to the fact that the acceptance conditions of the analyzed batch are not met, respectively that the quality parameter is greater or equal than the acceptance constant.

In the case of the graphic procedure of method s and the case of the lower limit of tolerance, the line of the general equation $m = L_i + k \cdot s$, respectively $m = 1120 + 1.39 \cdot s$, is represented, an equation that passes through the point (0; 1120) and has a slope tangent of 1.39, on a system of rectangular axes $s0m$, as can be seen in figure 1.

The current point with the coordinates (78.4; 1101) is placed on the graph made previously, noting that it is placed in the rejection zone (if the graph is made on graph paper). It is observed that the current point is very far from the linear equation separating the acceptance and rejection zones. For the ease of determination, the intersection of the equation line s = 78.4 with the equation line $m = 1120 + 1.39 \cdot s$ is made, obtaining the point M(78.4; 1229). The ordinate of this point of 1229 is higher than the ordinate of the current point of 1101, that is, the current point is located below the acceptance line, that is, in the rejection zone. It is observed that the final result is the same regardless of the procedure, i.e. if the numerical or graphic method was used.

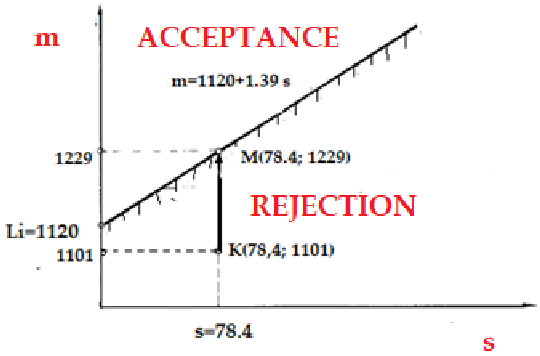

Fig. 1. The graphic procedure in the case of the application with method s, the lower limit of tolerance

3.2. Method R, the case of the lower limit of tolerance, the density of energetic willow pellets

As in the previous case, it is considered the case of the lower tolerance limit, at a density value of 1120 kg/m^3. The same batch of 1200 pieces of *Salix viminalis* pellets from the case of the previous application is considered, which will be analyzed with the average amplitude R method. At code letter J there is a sample of 35 pellets, and at an acceptable quality level AQL = 2.5 finds the acceptance constant k = 1.57. The 35 values of the sample are divided into subgroups of five pellets, as can be seen in the following Table 3. The amplitude of each subgroup is then determined, as the difference between the maximum and minimum value, after which the average of these amplitudes is made.

Table 3. The division of the sample into subgroups in the case of the R method

No.	Value of unit density, kg/m^3						
1	990.5	1197.6	1103.4	1018.3	1149.2	1043.9	1219.4
2	1006.2	1158.4	1116.1	1020.1	1089.4	1138.7	1070.4
3	1031.8	1270.6	1096.2	1213.1	1156.4	1008.0	1103.3
4	1078.4	1028.6	1018.9	1230.6	1135.3	1154.4	1117.2
5	1027.2	1063.7	1069.8	1040.1	1093.8	1030.8	1001.9
R_i	87.9	242.0	97.2	212.3	67.0	146.4	217.5
Rm	142.2						

An average amplitude of 142.2 kg/m^3 is found. To use the numerical determination process, the values necessary for the calculation are centralized in the following Table 4.

Table 4. Data of the numerical procedure of the application from the R method

No.	Specifications	Values
1	The sample size	35
2	The number of subgroups	7
3	The sample mean	1101
4	The average amplitude of the sample	142.2
5	Lower tolerance limit	1120
6	The quality parameter, q	$q = \frac{(\bar{m}-L_i)}{\bar{R}}$ = 19/142.2 = 0.13
7	Acceptance constant, k	1.57
Decision	Quality parameter q = 0.13 < Acceptance constant k = 1.57. The batch is rejected	

It is observed that, as when using method s, in this case the batch of pellets is also rejected, showing that whatever method is applied, the final result is the same. The application of the graphic procedure is done by transforming the relationship between the quality parameter and the acceptance constant into an inequality, whose line at the limit $m = L_i + k{\cdot}R$, is transposed into the plane (Figure 2), dividing the space into two zones, one of acceptance and the other of rejection.

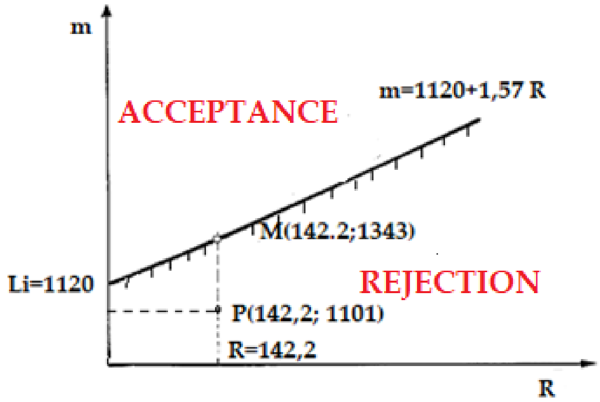

Fig. 2. The graphic procedure in the case of the application with R method

It can be observed that the current point P is located below the linear equation, respectively in the rejection zone, as was also determined when the numerical evaluation method was used. As in the previous case of the graphic method, the vertical line R = 142.2 is drawn on the graph, which will intersect the line dividing the space at the point M(142.2; 1343). Since the current point has an ordinate of 1101 lower than the intersection point of 1343, it means that it is under the line of delimited spaces, that is, in the rejection area of the lot.

3.3. The case of the upper limit of tolerance, method s, the ash content of larch briquettes

The ash content of briquettes is stipulated at a maximum of 0.7% by the Swedish standard SS 18 71 20 and group 1 briquettes (better quality). This limiting value represents the upper limit of tolerance, a case that is solved according to a certain statistical methodology, with a numerical and graphical procedure, procedures that will be further exemplified in the case of the ash content of larch briquettes. For this, it is considered that we have a batch of 600 larch briquettes, which will be analyzed from the point of view of the measurable characteristic, namely the ash content. The analysis methodology stipulated in the Romanian standards SR ISO 3951-5: 2009 and STAS 3160/3-84 is applied.

The usual verification level II is chosen at the code letter J, and at the acceptable quality level AQL = 4%, the effective sample of 35 pieces and the acceptance constant k = 1.39 are found. At the beginning, each sample is transformed into fine powder, by grinding and sorting with a 1×1 mm sieve, the 35 sample samples are created, the ash content test (ASTM E-1755-01, 2003) is performed for each sample, obtaining the following 35 values of the calcined ash content (Table 5).

Table 5. Calcined ash content values of larch briquettes

0.681; 0.667; 0.597; 0.477; 0.667; 0.698; 0.432;
0.592; 0.652; 0.492; 0.374; 0.549; 0.485; 0.649;
0.654; 0.682; 0.693; 0.547; 0.523; 0.612; 0.701;
0,384; 0.483; 0.477; 0.594; 0.699; 0.687; 0.701;
0.654; 0.672; 0.659; 0.692; 0.695; 0.684; 0.562.

The problem of acceptability or rejection of the batch of analyzed pellets is solved first by the numerical method, based on the data in the Table 6.

From Table 6 it can be seen that, although the arithmetic mean of the sample of 0.65% is lower than the upper limit of 0.7% of the ash content, but also very close to the lower limit of the upper tolerance, the lot is rejected mainly due to the fact that the acceptance conditions of the analyzed batch are not met, namely that the quality parameter is greater than or equal to the acceptance constant.

In the case of the graphic procedure of method s and the case of the upper limit of tolerance, the general equation line $m = L_s - k{\cdot}s$, respectively m = 0.7 – 1.39·s, is represented, an equation that passes through the point (0; 0.7) and has a slope tangent of 1.39, on a system of rectangular axes $s0m$, as can be seen in Figure 3.

Table 6. The numerical decision procedure for method s and ash content

No.	General data	Values
1	Sample size (from statistical standards)	35
2	Arithmetic means of the sample (from calculation)	0.65%
3	Sample mean square deviation (from calculations)	0.05
4	Upper tolerance limit (from pellet standards), L_s	0.7%
5	Acceptance constant (from the statistical standard), k	1.39
6	Quality parameter (from calculation), $q = \frac{(\bar{L_s} - m)}{s}$	1.0
7	Decision: The quality parameter q = 1.0 is lower than the acceptance constant k = 1.39. Conclusion: the batch is rejected.	

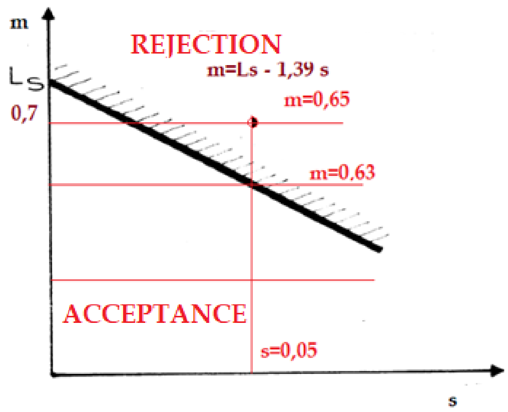

Fig. 3. The graphic procedure in the case of the application with method s, the upper limit of tolerance

The current point with the coordinates (0.05; 0.65) is placed on the graph made previously, noting that it is placed in the rejection zone (if the graph is made on graph paper). It is observed that the current point is very far from the linear equation separating the acceptance and rejection zones. For ease of determination, the intersection of the equation line s = 0.05 with the equation line m = 0.7 – 1.39·s can be made, obtaining the point M(0.05; 0.63). The ordinate of this point of 0.63 is lower than the ordinate of the current point of 0.05, that is, the current point is located above the right of acceptance, that is, in the rejection zone. It is observed that the final result is the same regardless of the procedure, i.e., if the numerical or graphic method was used, in both cases the decision to reject the batch of pellets was obvious.

4. Conclusions

The s and R methods for assessing the quality of product batches are the closest standardized methods for determining the quality of pellet batches.

The statistical methods for evaluating the quality of the pellet batches are based on the main statistical parameters of tendency and dispersion, respectively the arithmetic mean, the standard deviation and the amplitude of the measured values.

Regardless of the procedure used (numerical or graphic), the result of accepting/rejecting the lot will be the same.

The examples for unit density of pellets and ash content can be continued for other measurable characteristics such as bulk density, fine material granulometry, upper and lower calorific value, volatile and fixed carbon content and many others.

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