APPLICATION OF STATISTICAL TECHNIQUES TO CONTROL PARTICLE SIZE DISTRIBUTION IN GLAZES

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1. INTRODUCTION

Particle size distribution in glazes is one of the properties that influence the surface appearance of the glaze after firing. Variation in particle size can lead to variations in the gloss of the fired glaze and to variations in glaze melt viscosity, which can cause changes in the surface tension, in other words the roughness of fired glaze. Therefore, it is important to control this characteristic in order to avoid variations in the appearance of the finished product.

The best tools for controlling one or more variables are statistical control charts. A control chart shows the behaviour of a process with the data displayed in time order. The objective of control charts is to detect changes in the process which could result in defective pieces as early as possible.

In this study, a statistical control of particle size distribution in a glaze has been carried out in order to monitor variations in this property and observe their effects on the fired glaze. For this purpose, an X-S control graph was produced with the aim of detecting possible changes during the milling process, which could produce defects in the appearance of the fired glaze. To create the graph the D10, D50 and D90 percentiles of each of the millings made with the same glaze composition were determined with a laser diffraction instrument, considering that these three values would define the behaviour of the particle size distribution.

Lastly a process capability study was carried out. For this the Cp and Cpk capability indexes were determined.

The Cp index correlates the variability of the process itself with the specification limits established for the product or service resulting from it.

To be considered a Capable Process it is necessary that Cp is greater than or equal to 1.33.

The Cpk index is the Value that characterises the relationship between the process mean and its distance from the specification limit, which means that the process will give a less accurate result. In order to consider that a process is operating within the specification, Cpk must be greater than or equal to 1.33.

2. EXPERIMENTAL PROCESS

The study was carried out under laboratory conditions. The mill used was an alumina ball mill with a ball charge of 800 g. The chosen glaze was a matte glaze used for firing porcelain tile.

300g of glaze was weighed and wet milled in the ball mill. The D10, D50 and D90 percentiles of the slip were then measured. This process was repeated several times, only varying glaze milling time. The resulting slips were applied at the same density and viscosity on to a half engobed porcelain tile, using an applicator with a 4 mm opening. The tile was fired in an industrial cycle at a maximum temperature of 1190 ºC. Once the piece was fired, the differences were examined between the different glaze coatings. This was intended to establish certain tolerances when performing the following millings. The results are shown in table 1.

Milling time (minutes)	D10(μm)	D50 (μm)	D90(μm)
4	0.41	6.36	35.08
5	0.39	6.02	32.50
6	0.40	6.41	30.29
7	0.39	6.08	29.26
8	0.36	5.60	26.97
10	0.38	5.25	24.69
12	0.37	4.51	20.89
14	0.37	4.42	20.70
16	0.36	3.97	19.31
18	0.37	9.91	18.24
20	0.36	3.84	17.56

Table 1.

The glaze surfaces obtained with milling times of 7, 8, 10 and 12 minutes hardly showed any noticeable difference. At shorter milling time, the surface was more matte and at longer milling times, it was glossier. For this reason the following values were chosen as upper tolerance limits (UTL) and lower tolerance limits (LTL):

For D10: UTL = 0.39 μm LTL = 0.36 μm

For D50: UTL = 6.08 μm LTL = 4.51 μm

For D90: UTL = 29.26 μm LTL = 20.89 μm

X-S CONTROL CHART

Further glaze millings were performed but this time controlling that the parameters were within the tolerances given above. The percentiles for each milling were determined. To create the charts, samples were defined, each one of them comprising the measurements of five millings. The mean value and scatter for each sample were thus determined from the following equations:

$$X = (\Sigma xi)/n \qquad S = \sqrt{\frac{\sum (X - xi)^2}{(n-1)}}$$

SAMPLE	D10		D50		D90	
	X	S	X	S	X	S
1	0.37	0.01	6.37	0.61	22.40	2.12
2	0.36	0.00	4.80	0.52	24.79	1.98
3	0.39	0.01	5.45	0.54	23.11	2.70
4	0.37	0.00	5.91	0.48	28.47	2.45
5	0.39	0.00	6.06	0.60	27.75	2.69
6	0.38	0.01	4.84	0.65	24.28	3.01
7	0.37	0.00	4.96	0.47	24.04	2.06
8	0.37	0.01	5.16	0.36	27.62	2.79
9	0.39	0.00	5.48	0.28	29.06	3.12
10	0.39	0.01	5.04	0.34	21.97	2.45
11	0.38	0.01	5.25	0.68	25.50	2.56
12	0.36	0.00	4.65	0.50	26.14	3.18
13	0.38	0.01	5.07	0.52	22.06	2.16
14	0.39	0.01	5.31	0.65	25.37	2.78
15	0.38	0.01	4.69	0.47	24.51	2.66
MEAN VALUES	**X= 0.38**	**S = 0.01**	**X = 5.27**	**S = 0.51**	**X = 25.14**	**S = 2.58**

From the mean values of X and S, the control limits were calculated in order to construct the means chart from the equations:

Upper control limit: $UCLx = X+A \cdot S$

Centre limit: $CLx = X$

Lower control limit: $LCLx = X-A \cdot S$

The value of A is derived from a statistical table where the values of A are indicated as a function of the number of measurements. In this case, where n=5 and A= 1.427. Thus, for each percentile one obtains:

PERCENTILE	UCLx	CLx	LCLx
D10	0.39	0.38	0.36
D50	6.00	5.27	4.54
D90	28.82	25.14	21.45

The limits were then calculated so that the scatter graph could be constructed:

Upper control limit: $UCLs = B_4 \cdot S$

Centre limit: $CLs = S$

Lower control limit: $LCLs = B_3 \cdot S$

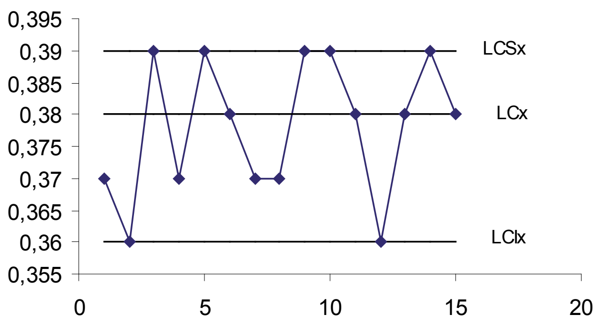
As in the foregoing case, B4 and B3 are tabulated constants for each sample size. In this case, B4 = 2,089 and B3 = 0.

The values obtained are shown below:

PERCENTILE	UCLs	CLs	LCLs
D10	0.02	0.01	0
D50	1.06	0.10	0
D90	5.38	0.10	0

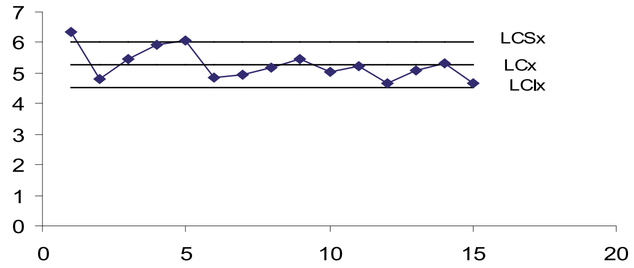
Finally, the charts obtained were drawn:

CHART OF X MEAN VALUES FOR D10



All the values are under control

CHART OF X MEAN VALUES FOR D50



Two of the values are not under control. This could be due to bad measurement by the measuring apparatus. These were eliminated and the limits recalculated. On doing this, again one value is not under control, so it was eliminated and the limits recalculated again. Finally the following limits were obtained for the D50 percentile:

UCLx = 5.77 CLx = 5.06 LCLx = 4.34

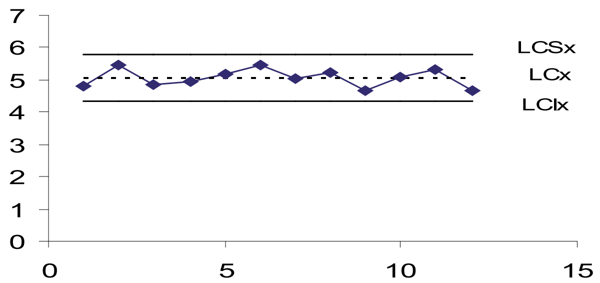
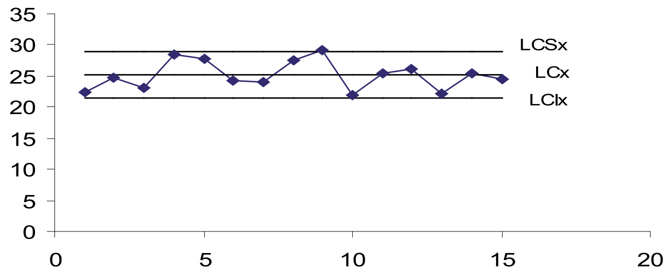


CHART OF X MEAN VALUES FOR D90



All the values are under control.

CHART OF S SCATTER VALUES FOR D10

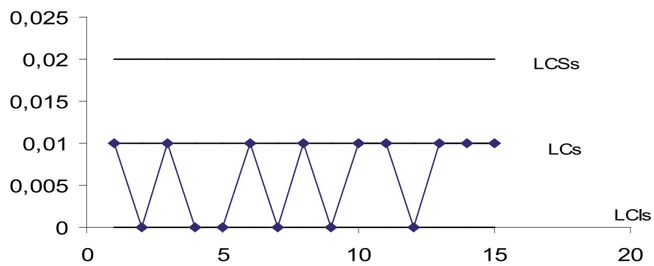
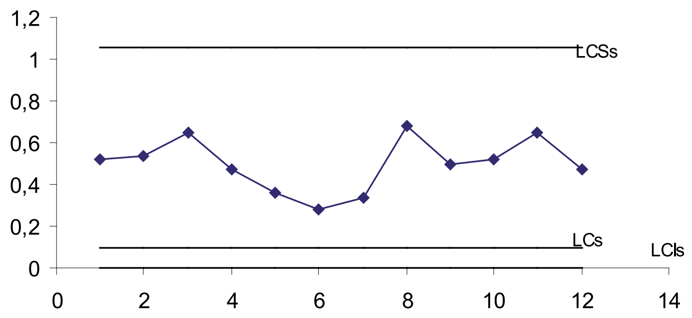
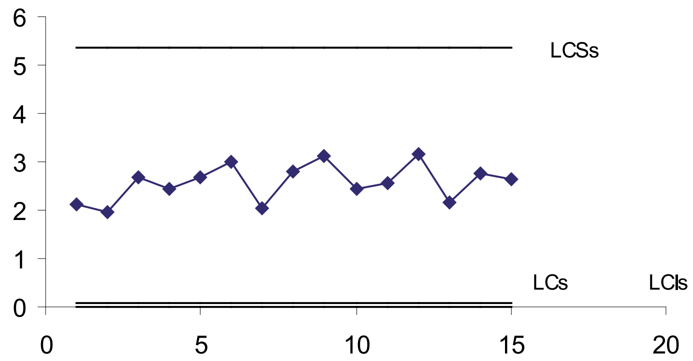


CHART OF S SCATTER VALUES FOR D50



SCATTER GRAPH S FOR D90



CAPABILITY STUDY

The capability indices Cp and Cpk were determined. For this the following equations were used:

Capability index Cp: $Cp = (UTL - LTL)/6\sigma$

Capability index Cpk: $Cpk1 = (X - LTL)/3\sigma$

$Cpk2 = (UTL - X)/3\sigma$

The most restrictive value of Cpk1 y Cpk2 will be Cpk.

Standard deviation: $\sigma = S/c_2$ c_2 is a tabulated constant that depends on sample size.

where $n = 5$ and $c_2 = 0.8407$

The following values were thus obtained for each percentile:

D10: Cp = 0.5 Cpk = 0.3 (incapable process)

D50: Cp = 0.4 Cpk = 0.3 (incapable process)

D90: Cp = 0.5 Cpk = 0.5 (incapable process)

Therefore, after carrying out the capability study, it was concluded that, although all the variables are within the limits in the control charts, the process is not capable of complying with the specified tolerance, so that the possibilities need to be examined of changing process or product design.

REFERENCES

[1] Verdoy, P; Mateu, J; Sagasta, S; MANUAL DE CONTROL ESTADÍSTICO DE CALIDAD: TEORÍA Y APLICACIONES. Published by Universidad Jaume I, 2006.