



Optimization of the Particle Size Distribution of White Sugar Using Six Sigma Methodology

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Abstract —The main objective of this paper is to provide a full six sigma approach to control several indicators characterizing the variability of the white sugar's particle size at the crystallization station in an emerging African country such as Morocco. To do this, we first defined our perimeter of study, and subsequently we defined the critical elements of quality (CTQ) of our project namely: Average Aperture (OM) with a target of 0.6mm and a variation margin of ± 0.05 and Coefficient of Variation (CV) which varies in the range [38% -45%]. To make a diagnosis of the initial state of the granulometry we have adopted the study of repeatability and reproducibility as well as the study of the process capabilities. In order to trace the root causes of the variability in the crystallization process, we carry out an in-depth analysis of our process by adopting the experimental plans. The study of desirability identified the combination of variable parameters that jointly optimize the Average Aperture and Coefficient of Variation. To verify the validity of the resulting model we realized monitoring and we recalculated the new capability indicators and the level of quality achieved characterized by Z process. In order to put our process under continuous control we have carried out control charts. At the end of this project we quantified the gains, a 22.51% reduction in non-quality in white sugar.

Keywords- Six Sigma; DMAIC; Experimental Plans; Crystallization; Particle Size Distribution; Desirability Study; Study Case.

I. INTRODUCTION

The opening up of the Moroccan economy to foreign trade and the exacerbation of competition on the international scale have forced the Moroccan company to upgrade. The Moroccan sugar industry has experienced increased competition in the world. The evolving needs of the customer, privatization and openness to the world market impose the perpetual search for excellence. To improve competitiveness, to meet the needs of the internal and external market and to accompany consumer trends, it is necessary to align with the quality characterized mainly by the Particle Size Distribution of white sugar. It is in this perspective that this project is " Optimization of the Particle Size Distribution of White Sugar Using Six Sigma Methodology ". To do this, we adopted the six-sigma approach in order to attack the large variability in the Particle Size Distribution of white sugar.

Six Sigma or 6 Sigma is a registered trademark of Motorola Designating a structured management approach to improve process quality and efficiency [1-6]. The Six Sigma method was first applied to industrial processes [7][8] before being extended to all types of processes, including logistical [9-11], commercial, services [12-14], healthcare [15-18], and energy-saving processes [19]. The notion of quality is closely linked to that of variability. we can even define non-quality as a variability with respect to an expected reference. Now the fight against variability is one of the basic concepts of Six Sigma. The Six Sigma approach brings its rigorous methodology in the approach of the improvement of the quality of production and reduction of waste in the sense of lean management. It also brings the managerial structure that makes all its strength. In fact, Six Sigma is not a method; it is also a way to organize the business so that we can be able to achieve these significant advances in performance, what has been achieved in this project. Since we aim to improve a current process and an existing product performance which does not meet customer expectation, we apply the five-phase define, measure, analyze, improve and control (DMAIC) methodology. To define our project, we used the CTQ diagram which translates the needs of the client into measurable requirements, namely the Average Aperture, noted OM, whose target is 0.6mm with a tolerance of ± 0.05 mm and the Coefficient of Variation, noted CV, varying in the Interval [38% -45%]. We studied the performance of our measurement system by the study of repeatability and reproducibility as well as the performance of the crystallization process and precisely the performance of the first jet by the study of the capabilities. To analyze the influencing factors, we used the experimental designs which allowed to emerge with two models satisfying the two requirements of the customer. The desirability study made possible the optimization of both parameters (OM, CV) together. In order to put the process under continuous control we proposed control cards.

II. DEFINE THE PROBLEM

2.1. Problematic

Crystallization station provides the extraction of sugar crystals in syrup through three successive jets. The draft aims to extract maximum crystals; it represents 70% of overall yield and a purity of 99.99%. Particle Size Distribution is an important characteristic of sugar. It is not only a quality feature but also an important technological parameter. Indeed, the distribution of the grains size is important insofar as it is a decisive factor for the storage in silos, for its dispatch as

well as for the molding of the sugar in pieces. Indeed, sugar is better appreciated when its crystals are uniform and regular sizes. This work involves reducing the variability of white sugar by controlling the Particle Size Distribution parameters essentially characterized by:

Average Aperture (OM): which gives the average size of the crystals. It represents the opening of the sieve which let pass half of the grains and retain the other half [1].

Coefficient of Variation (CV): which is a standard deviation expressed as a percentage, representing the dispersion of the measurements around the mean opening.

The aim of our project is to achieve an average opening (OM) 0.6 with a tolerance of $\pm 0.05\text{mm}$ and a coefficient of variation (CV) in the range [38% -45%].

The present project concerns only crystallizers of the first jet, the other crystallizers come out of our perimeter of study illustrated in figure 3.

What?	The non-homogeneity of the grains and the variability in the grain size distribution of white sugar
Who?	All project stakeholders: Group customers; Refining Service; Storage
Where?	Crystallization station
When?	Since the start of the crystallization station in 2009 and the start of particle size analysis of white sugar
Why?	The uncontrolled crystallization parameters
How?	How will we solve the problem? by adopting the six sigma methodology

Figure 2: 5W1H Questions

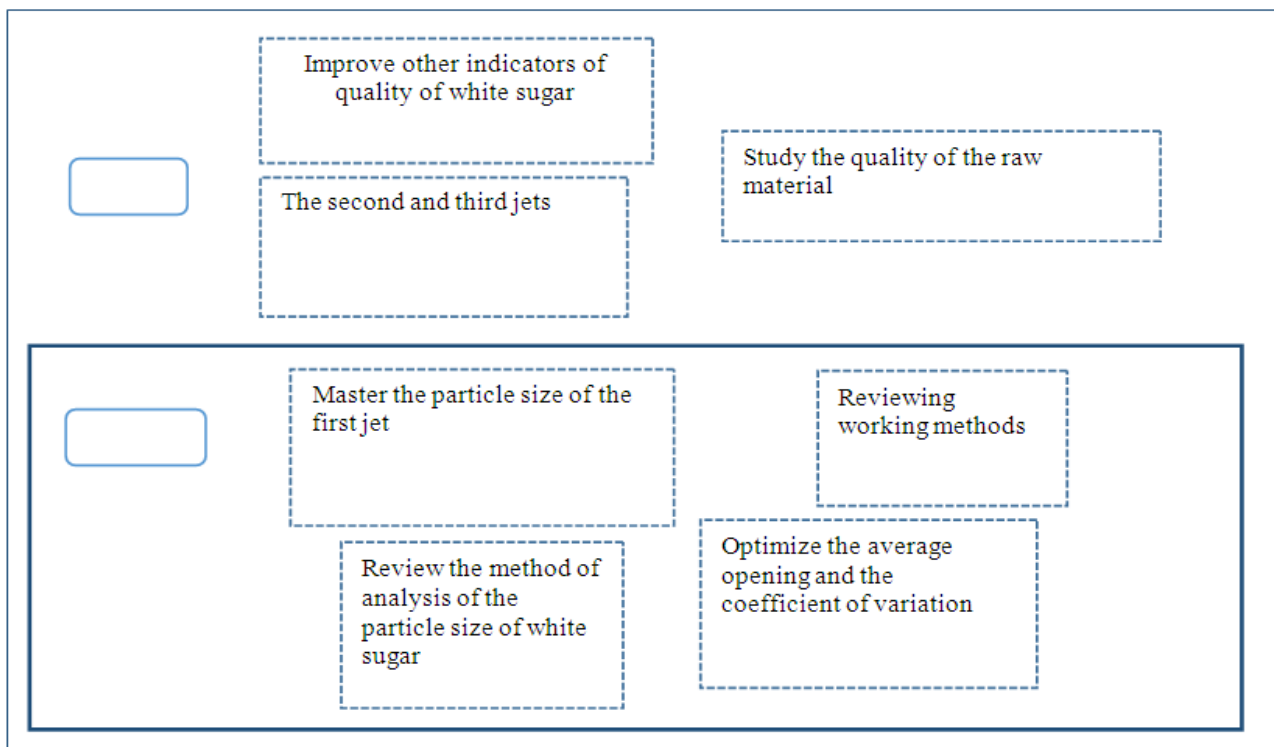


Figure 1: In/Out Diagram

This will be done by studying the sources of variability in these parameters and then proposing improvements at this level. Our project is SMART (Specific, Measurable, Achievable, Reasonable, is limited in time).

2.2. The raw sugar refining process

Refining is the method to have a high purity refined sugar from raw sugar. The raw sugar refining process is as follows:

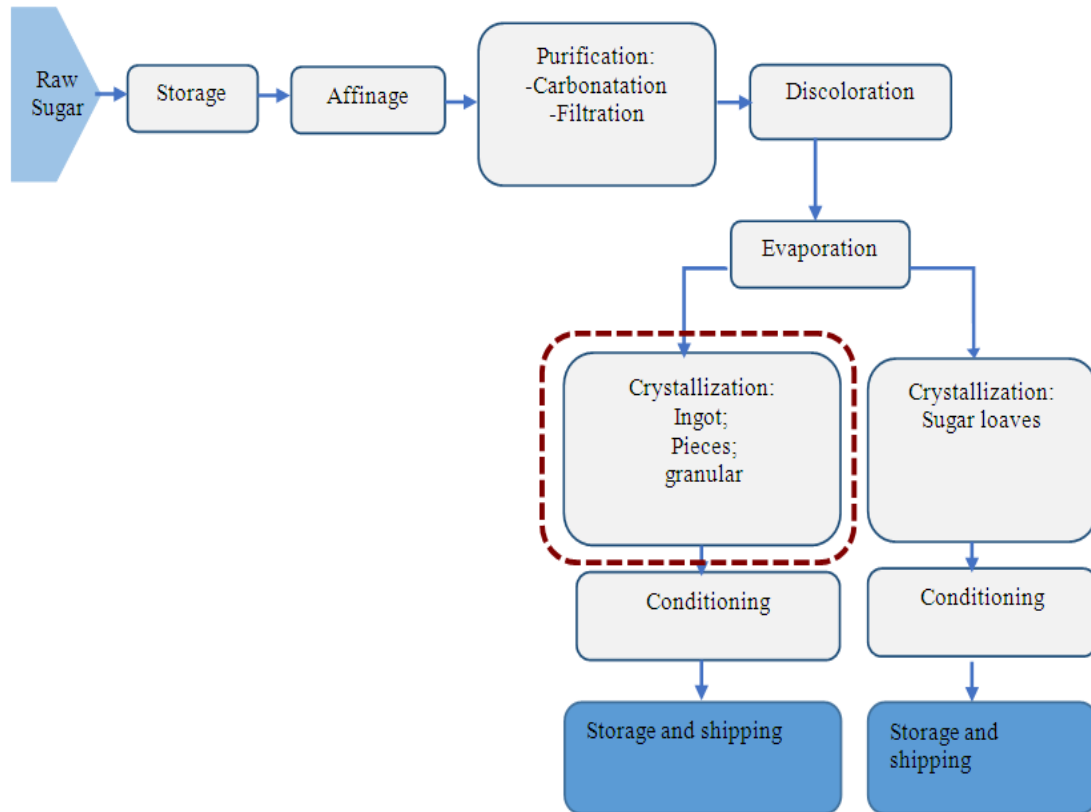


Figure 2: The raw sugar refining process

2.3 Description of the first jet

The first jet is the most important jet, either quantitatively by about 70% of all production, or qualitative as the sugar at the outlet of this jet has a purity equal to 99.99%. The first step at the start of a crystallization is the evacuation. Once the vacuum established, the "massecuite" is prepared by covering sufficiently the heat exchanger by the standard liquor which corresponds to 25% of the volume of the device. The liquor is concentrated until it reaches the point of supersaturating. The beam must remain submerged all the time, it's for this reason that the evaporated water is compensated by continuous introduction of syrup, which is regulated by a Brix meter. Once the graining point is reached (point of supersaturating), a seeding is performed in order to initiate the formation of the other crystals. The seeding is carried out by minimizing the heating vapor of the apparatus in order to avoid the dissolution of the crystals, which corresponds to a brix of 78% to 80%. The seeding must be followed by a stabilization period (waiting time or starting of the magnification). It is a phase of maintaining supersaturating around the value of the seeding point. The rise lasts about 30 to 40 minutes until the first clamping. This first clamping cooked is the most delicate phase in so far as the risk of conglomeration of the crystals is great. When the maximum level is reached (clamping phase) the supply of syrup is stopped, and evaporation of the massecuite is continued. Cooking ends with the pouring and washing of the appliance. After the apparatus has been returned to atmospheric pressure by a vacuum breaker and the agitator is stopped, the drain valve is opened and the massecuite flows into a mixer.

2.4 Cartography of the first jet

To visualize the inputs / outputs of the first jet and the various control parameters we have carried out the following SADT mapping:

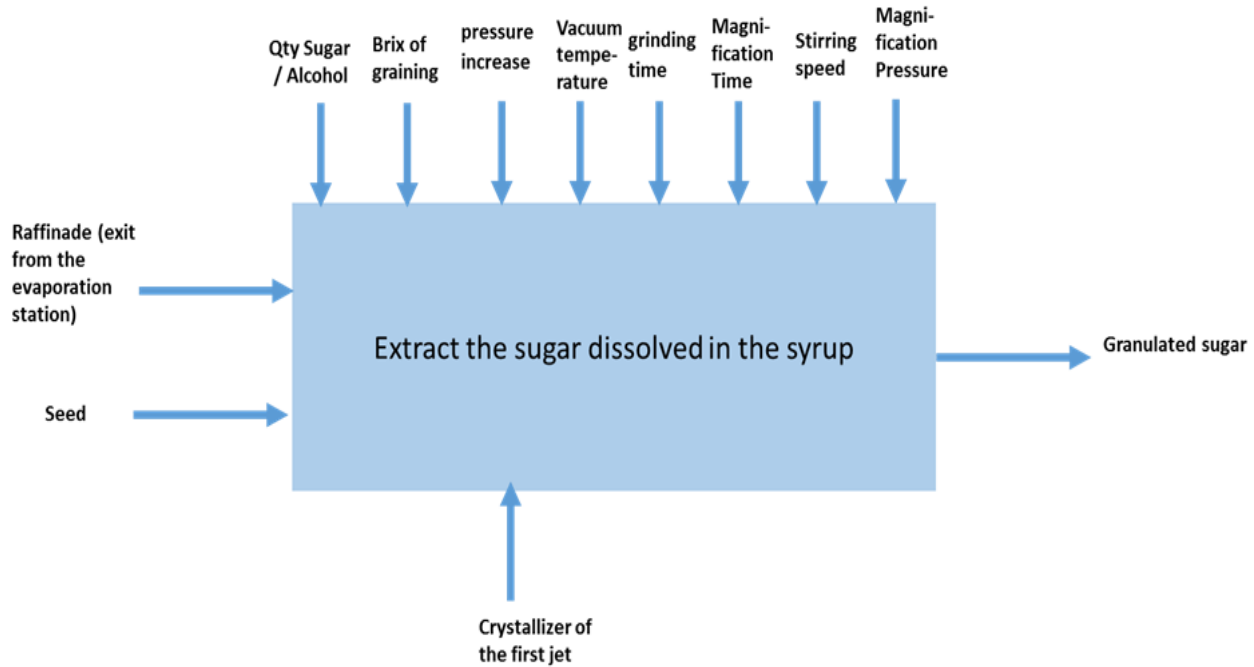


Figure 3: SADT diagram of the first jet

2.5 Critical To Quality

Granulometry is the study of the grain size distribution of white sugar. The fineness of white sugar is characterized by: opening Medium (OM), the Coefficient of Variation (CV), the percentage of Coarse (% GG), the percentage of fine grains (% GF). The two indicators Medium Aperture (OM) and Coefficient of Variation (CV) are considered the most critical for the client. The CTQ diagram below defines the critical characteristics for the client, their targets, and their limits:

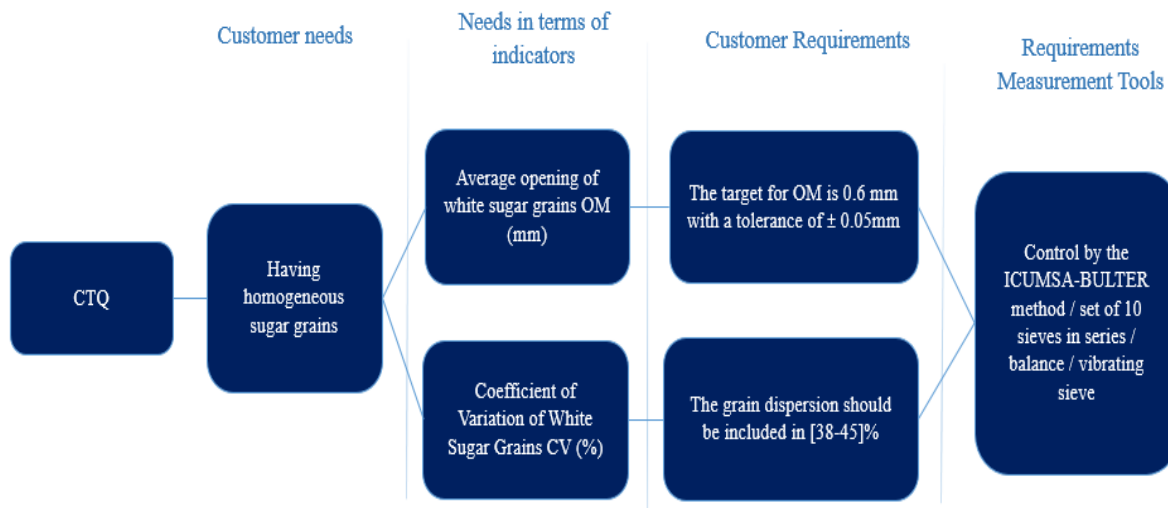


Figure 4: CTQ Diagram

The black box allows to visualize the transfer function defined by the relation $Y = F(A, B, C \dots)$ which is established between the response to optimize Y and the process parameters (A, B, C ..). We seek to demonstrate by statistical evidence the non-random nature of the relationship between the input parameters and Y.

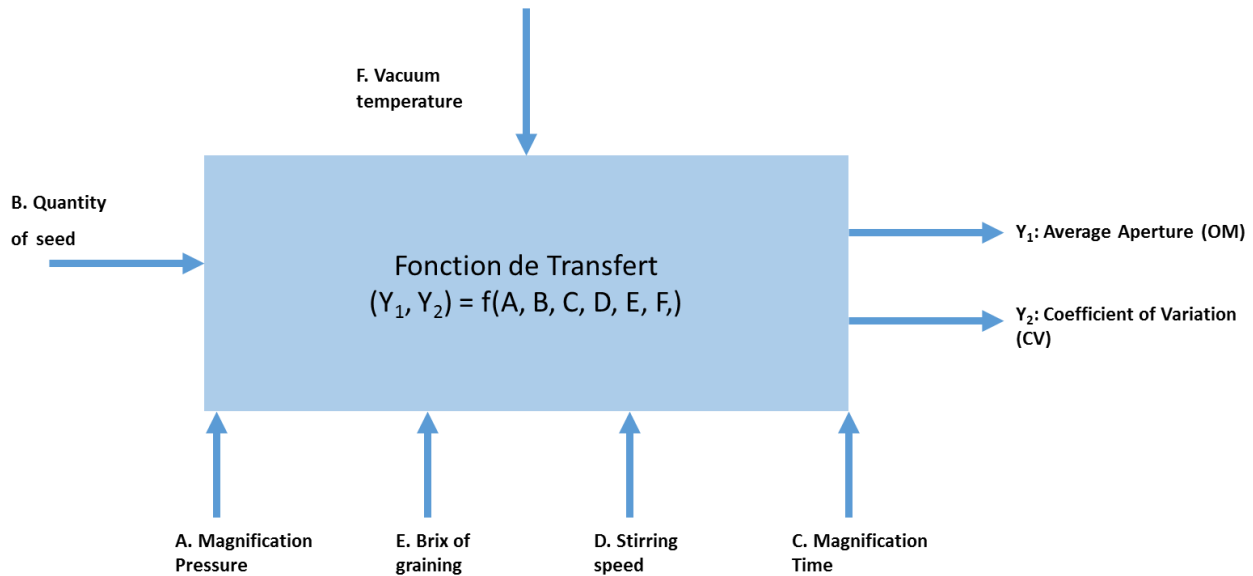


Figure 5: Black box of the process

III. MEASURE: THE PROCESS OF MEASUREMENT AND DATA COLLECTION

3.1. Validation of means of measurement

The measuring means must be attached to the calibration chains and possess properties of accuracy, linearity and stability. This will be the first check to be carried out. The method of sugar analysis corresponds to "Method GS2 / 9 - 37 (2007) for the determination of the particle size distribution of white sugar and plantation sugars by sieving", taken from the ICUMSA - Methods Book. This method is applicable to all white sugars and brown sugars. Several methods are used to determine the OM and the CV of the sugar. They are cited by the ICUMSA repository: POWERS method, RENS method, RRSB method, BUTLER method. The method used by the study laboratory is the Butler method.

The refinery's laboratory is certified ISO 17025 version 2005, which guarantees the accuracy, linearity and stability of the CTQ measurement means.

3.2. Study of Repeatability and Reproducibility (R & R)

We carried out the R & R study on samples taken over five days and each sample was analyzed and repeated by the same operator and by another operator in an independent manner. We have taken the measurements of the two characteristics: OM and CV. The results of the study are presented in Figure 9.

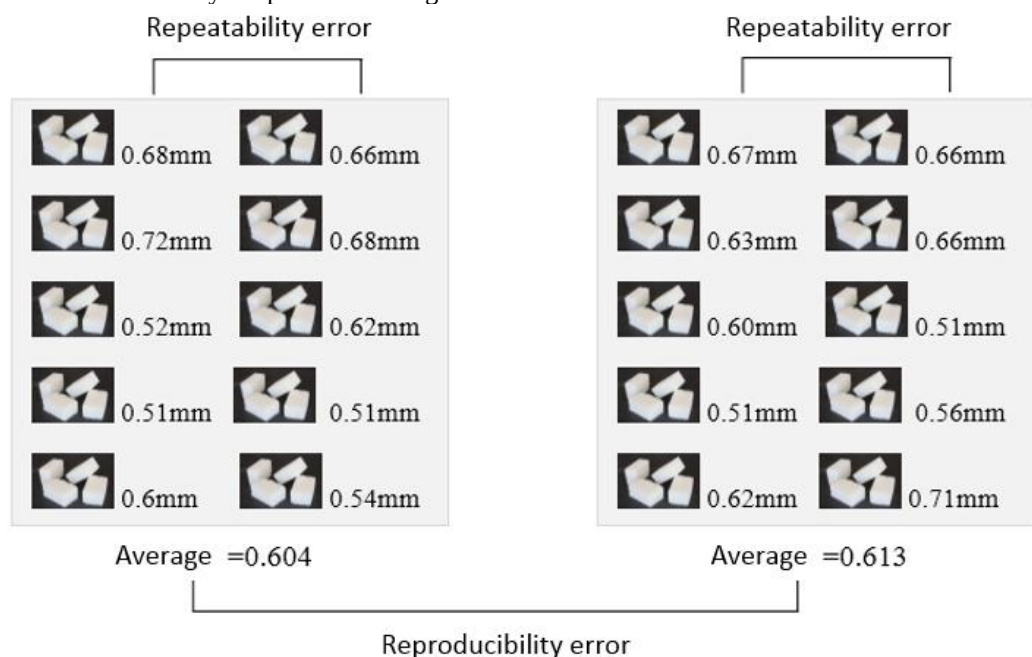


Figure 6: R & R study for OM

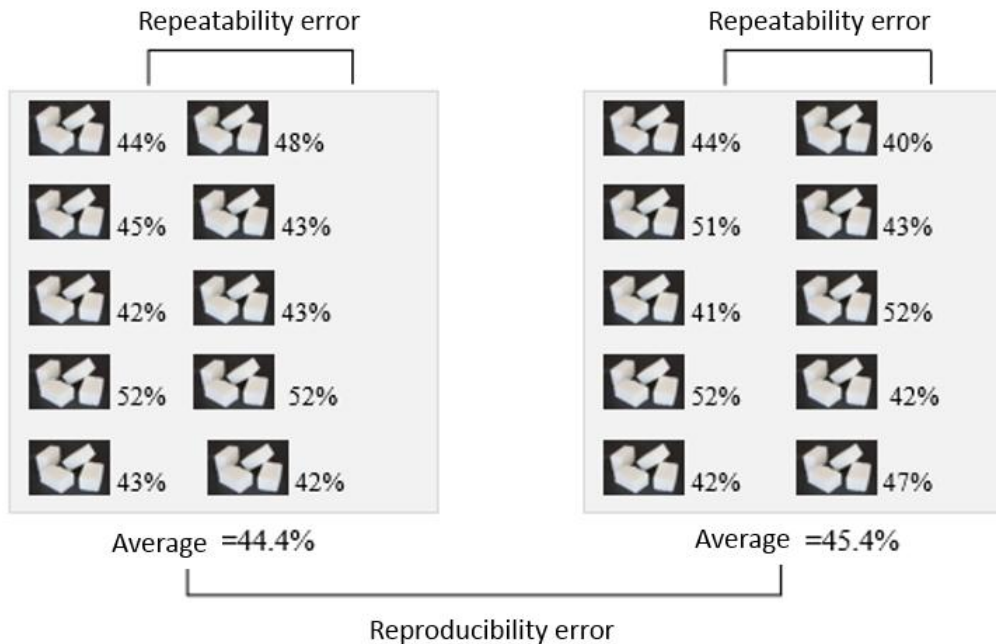


Figure 7: R & R study for CV

Gage R&R Study: OM versus Parts; Operators

ANOVA Table with All Terms

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Parts	4	0,051404	0,049731	0,012433	3,33	0,135
Operators	1	0,000339	0,000419	0,000419	0,11	0,754 x
Parts*Operators	4	0,014931	0,014931	0,003733	1,10	0,406
Repeatability	11	0,037450	0,037450	0,003405		
Total	20	0,104124				

α to remove interaction term = 0,05

Gage R&R Study: CV(%) versus Parts; Operators

ANOVA Table with All Terms

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Parts	4	303,967	303,641	75,910	24,24	0,205
Operators	1	0,008	0,006	0,006	0,00	0,968 x
Parts*Operators	4	12,526	12,526	3,131	1,55	0,254
Repeatability	11	22,167	22,167	2,015		
Total	20	338,667				

α to remove interaction term = 0,05

Figure 8 : Result of the Study R & R from Minitab Software

The ANOVA table for the repeatability and reproducibility study Minitab software shows what measures the variation sources are statistically significant. We have noted that both the Parts and Operators factors and their interaction in the R & R study (for OM and CV) have a P value greater than $\alpha = 0.05$, which shows that statistically we do not have an R & R problem. In addition, and depending on the time and frequency of molding, we have standardized the sampling that will be carried out.

3.3. Measuring and evaluating process performance

Since the means of measurement is capable and the method of sampling fair, we have set up observation campaigns of the process. The results are presented on the control charts Fig. 10. We tested the normality of the data by the Anderson-Darling test. The results are shown in Fig. 11. With a significance level of 0.05, the Anderson-Darling normality test for OM shows that the data follow a normal distribution since P-value = 0.111 > 0.05. This is not the case for CV which does

not follow the normal distribution since $p < 0.005 < 0.05$. The analysis of the capability of the OM Fig. 12 gives: $C_p = 0.43$ therefore the level of capability is very insufficient. As a direct consequence, there is a performance problem with $P_p = 0.41 < 1.33$. We also have $CPL=0.22 < CPU=0.65$, $CPK=0.22=CPL < CPU$; So we have a problem of decentering. The CV does not follow a normal distribution. It is for this reason that we used a non-normal capability Fig. 12 study which gave: $P_p = 0.26 < 1.33$ then we have a problem of very low performance, and $Ppk = 0.11 = PPU < PPL$ then the process is Not centered.

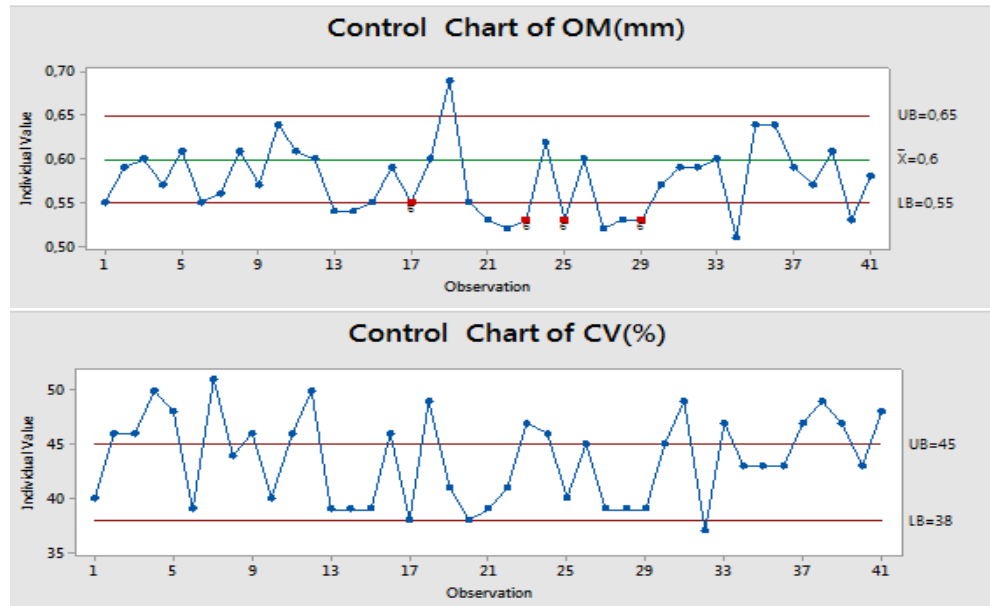


Figure 9: Average of the Opening (OM) and Coefficient of Variation (CV) Control Charts – Minitab

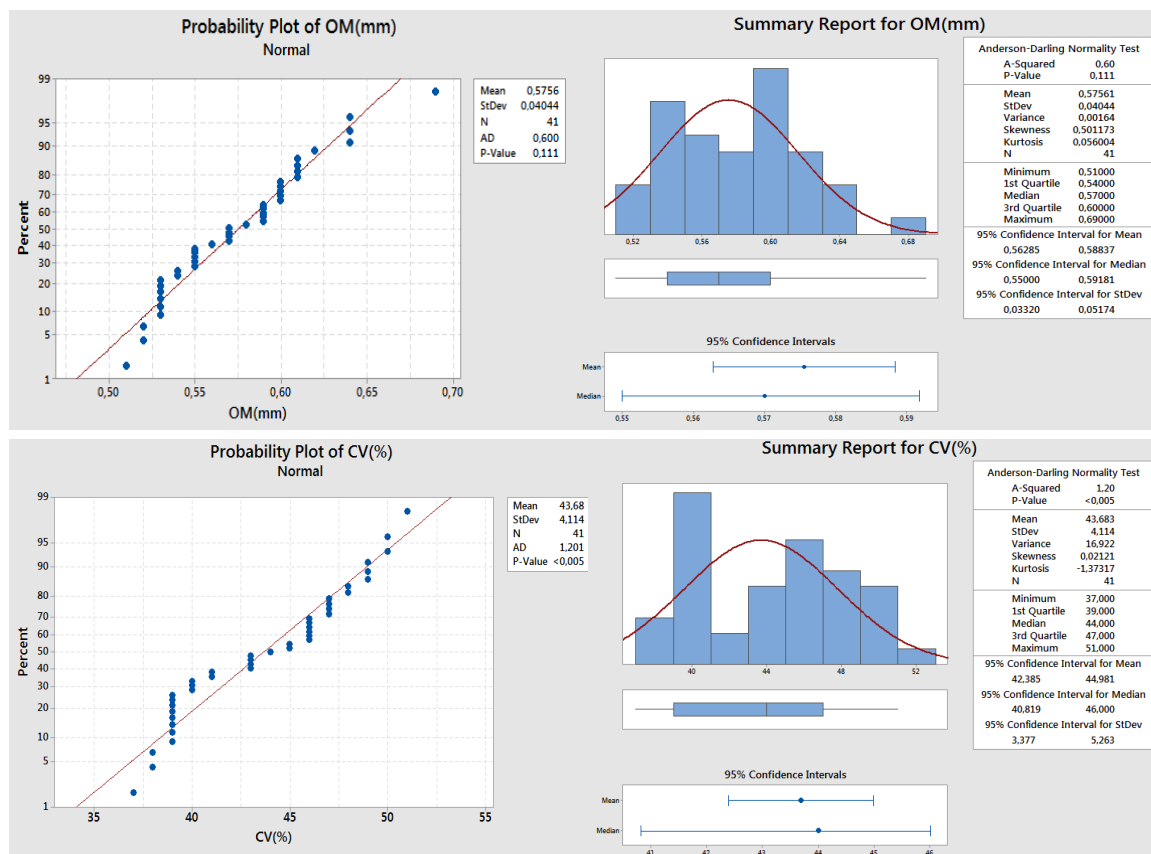


Figure 10: Normality Test for OM and CV - Minitab

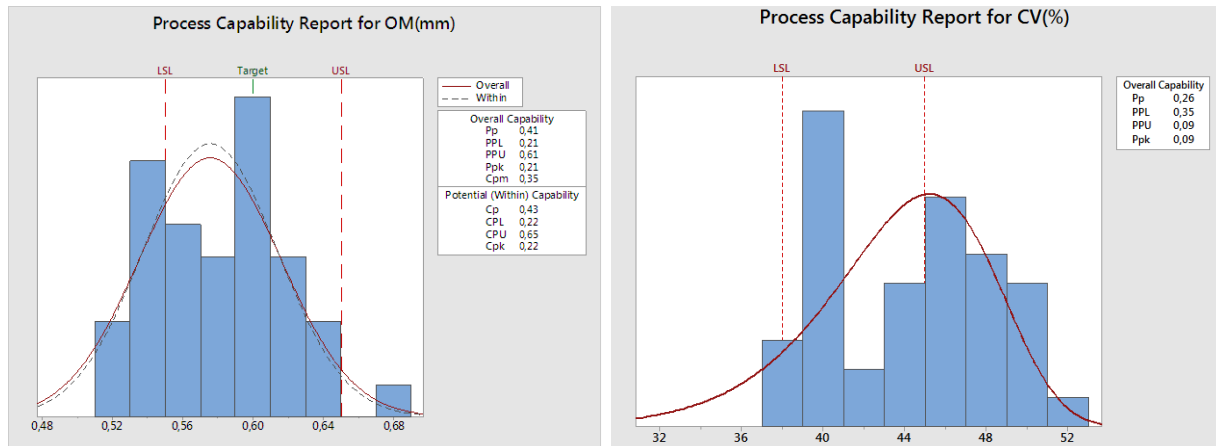


Figure 11: OM and CV Process Capability – Minitab

IV. ANALYSE: THE FACTORS INFLUENCING PARTICLE SIZE SISTRIBUTION

During the diagnosis of the crystallization station, we were able to identify several factors, as parameters that could possibly influence the Particle Size Distribution of white sugar, which are:

- The quantity of seed introduced for graining;
- Cooking vacuum temperature / pressure;
- The grinding time of the sugar used for the preparation of the seed / average opening (OM) of the seed sugar;
- The amount of isopropyl alcohol in relation to the amount of sugar used in the preparation of the semen;
- Vapor pressure during the rise phase;
- The speed of agitation;
- The duration of the magnification phase (waiting time);
- The Brix of graining (the supersaturating indicated by the Brix);
- Vapor pressure during the magnification phase.

To study the effect of these parameters, we proceeded by the experimental design method with the exception of three factors that must be fixed in advance:

- The grinding time of the sugar used for the preparation of the seed / average opening (OM) of the seed sugar = 3h30;
- The quantity of sugar of 5 kg with a total volume of 7.7 l of isopropyl alcohol (i.e. 13 l of seed obtained);
- The vapor pressure during the rise phase = 900mbar.

4.1. Screening Plans

We have chosen to work with the Plackett and Burman plan, which is a simplified fractional factorial plan that allows us to quickly select the most influential factors. This is a preliminary study to be used to prepare an optimization plan. The experimental field of the screening plane is illustrated in Table 1. The experimental matrix obtained for 6 factors screening plane is a Hadamard matrix with 8 experiments is shown in Table 2 with both responses OM and CV.

Table 1: Factors and Field of Study

Factors	Unit	Level -	Level +
A. Magnification Pressure	mbar	800	1000
B. Quantity of seed	l	15	20
C. Magnification Time	s	100	300
D. Stirring speed	Tr/min	375	600
E. Brix of graining	%	78	80
F. Temperature	°C	72	75

Table 2: Factors and their Field of Study

Test Number	Factors						Responses	
	A (mbar)	B (L)	C (s)	D (Tr/min)	E (%)	F (°C)	OM (mm)	CV (%)
1	1000	20	300	375	80	72	0,48	41,127
2	1000	20	100	500	78	72	0,51	49,308
3	1000	15	300	375	78	75	0,49	41,206
4	800	20	100	375	80	75	0,72	36,915
5	1000	15	100	500	80	75	0,55	40,243
6	800	15	300	500	80	72	0,41	46,266
7	800	20	300	500	78	75	0,61	40,608
8	800	15	100	375	78	72	0,52	46,265

Table 3: Screening Experiment Matrix

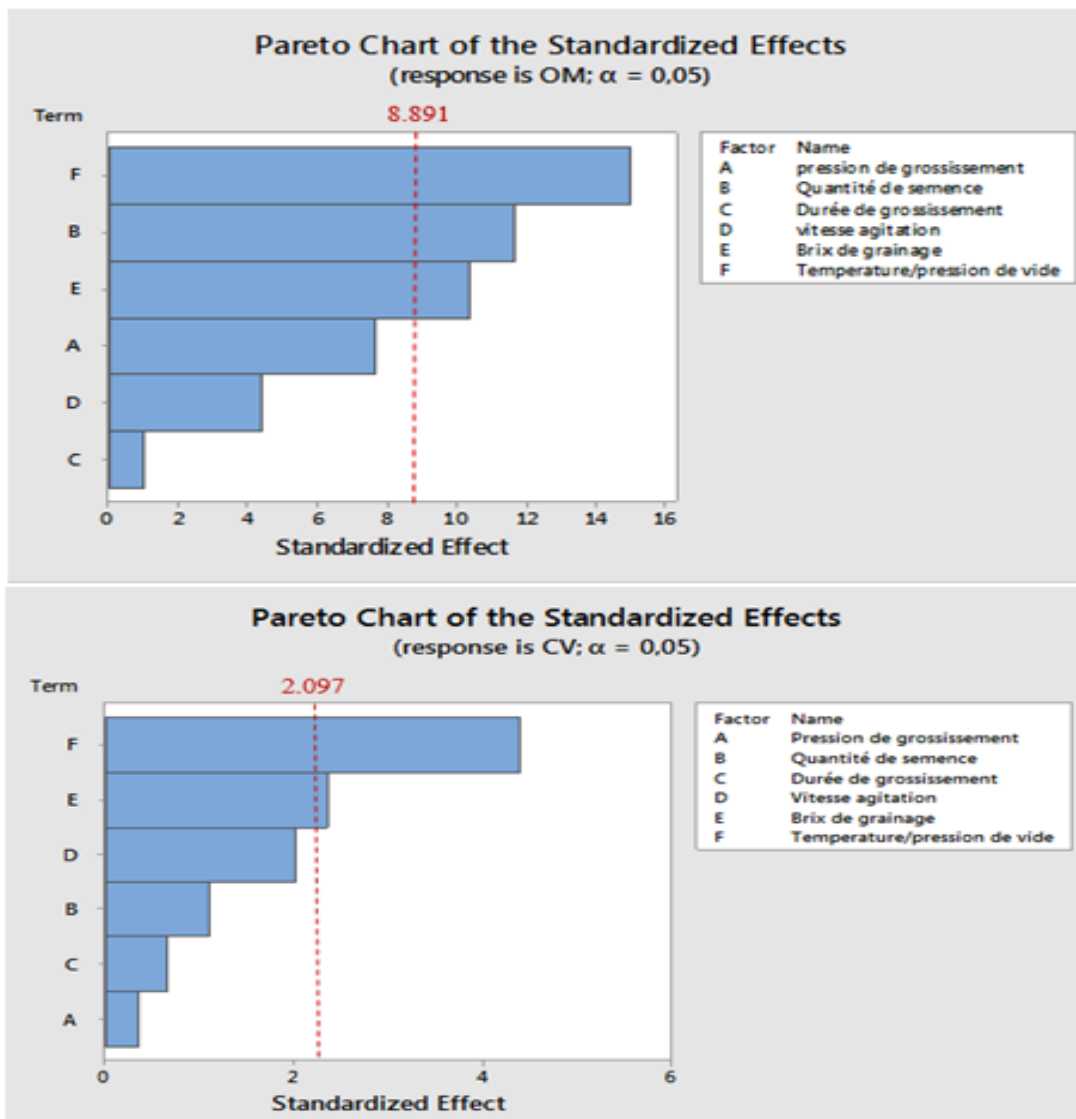


Figure 12: Graphical study of the effects of OM and CV responses

According to the study carried out, it follows that the parameters that have a statistically significant influence on the two CTQ (OM and CV) are: F : Temperature; B: Quantity of seed E: The Brix of graining. These factors will be retained for the optimization plan.

4.2. Optimization plans

In this step we generate a modeling plan and we carry out each test and we measure the answers obtained. These data make it possible to model OM and CV according to the factors with strong influence on the particle size distribution and their interactions. The experimental domain of the planes is illustrated in the table 3. The experimental matrix obtained for an experimental plan of 3 factors is 23 repeated tests, that is to say 16 tests in total. It is represented in the table 4.

Factor	Unit	Level -	Level+
B. Quantity of seed	L	15	20
F. Temperature	°C	72	75
E. Brix of graining	%	78	80

Table 4: Factors and their Field of Study

Test Number	Factors influencing grain size			Responses	
	Quantity of seed (L)	Temperature (°C)	Brix of graining (%)	OM (mm)	CV (%)
1	20	75	78	0,617	44,407
2	20	75	80	0,489	53,098
3	20	72	78	0,409	44,572
4	20	72	80	0,476	46,922
5	15	75	78	0,508	41,881
6	15	75	80	0,621	42,342
7	15	72	78	0,447	42,273
8	15	72	80	0,394	39,482
9	20	75	78	0,64	43,919
10	20	75	80	0,534	49,118
11	20	72	78	0,404	44,848
12	20	72	80	0,473	47,301
13	15	75	78	0,574	41,238
14	15	75	80	0,595	39,551
15	15	72	78	0,463	43,1
16	15	72	80	0,395	44,54

Table 5: Experimental Matrix

4.3. Results of the modeling plan

Figure 14, 15 and 15 respectively show the graphs of the effects, the graphs of the interactions and the Pareto diagrams allowing to see the factors and the interactions with statistically significant influence.

$$OM = 0.50244 + 0.06981 B - 0.03906 (A*B*C)$$

$$CV = 44.287 + 2.486 A + 1.329 (A*C)$$

Such as: A: temperature; B: Quantity of seed and C: Brix graining.

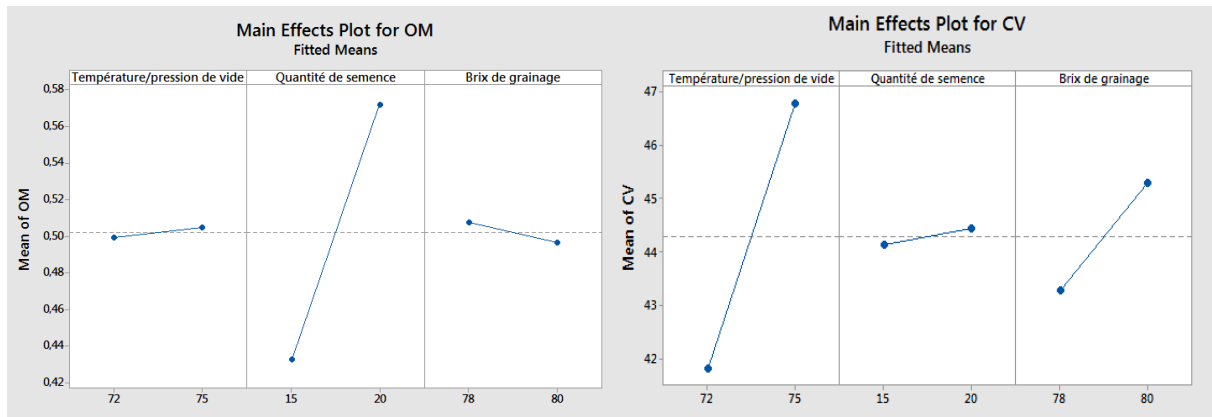


Figure 13: Effects graphic for OM & CV – Minitab

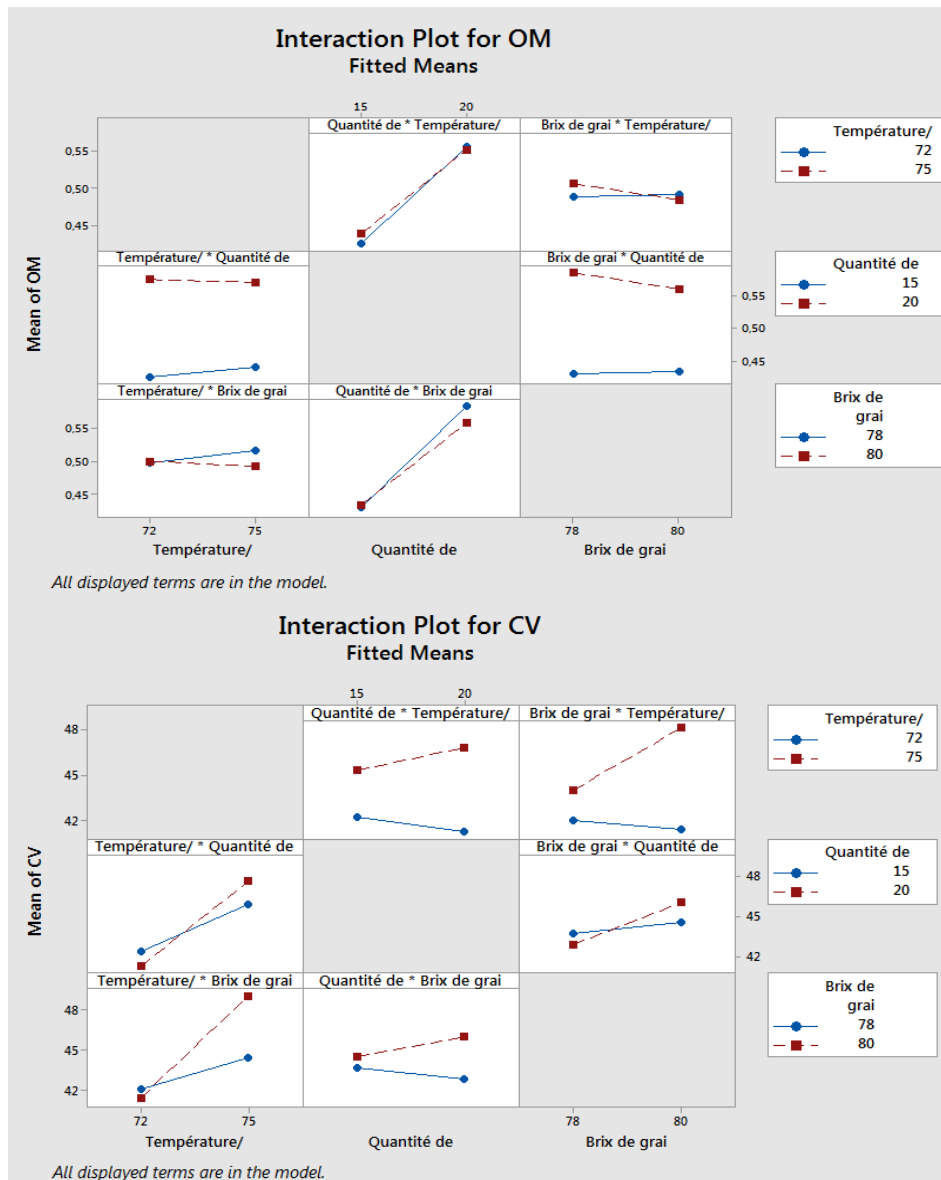


Figure 14: Graph of interactions for OM & CV – Minitab

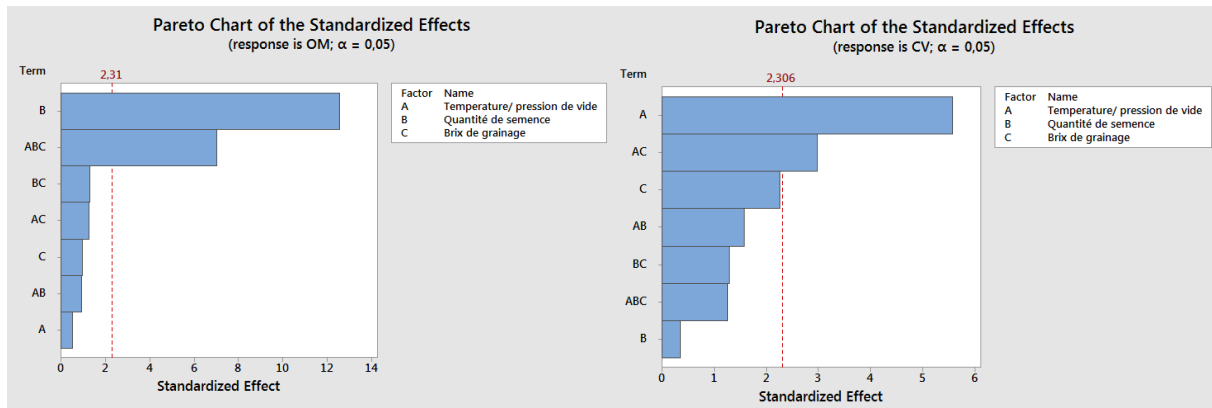


Figure 15: Pareto Diagrams

The previous study made it possible to have the mathematical models for the OM and the CV. As previously stated, we want to optimize both responses at once, OM should be between 0.55 mm and 0.65 mm and CV between 38% and 45%. So we do a desirability study.

V. IMPROVE: OPTIMAL POINT

4.4. Desirability Study

The composite desirability study makes it possible to find the optimal conditions that will bring us closer to the desired answers. We have chosen as the importance value of the two responses 1 since both present the same level of importance for the clients. Table 5 shows the parameters of the desirability function. This study allowed us to identify the combination of the variable parameters that jointly optimize the two responses fig.17. The desirability value (0.9891) is close to 1 which indicates that the parameters appear to reach satisfactory results for all the responses. The individual desirability for OM and CV is 0.99798 and 0.98021, respectively. This study gave the optimal values for the three factors presented in Table 6.

Response	Goal	Lower	Target	Upper	Weight	Importance
CV	Target	38	41,5	45	1	1
OM	Target	0,55	0,60	0,65	1	1

Table 6: Parameters of the desirability function

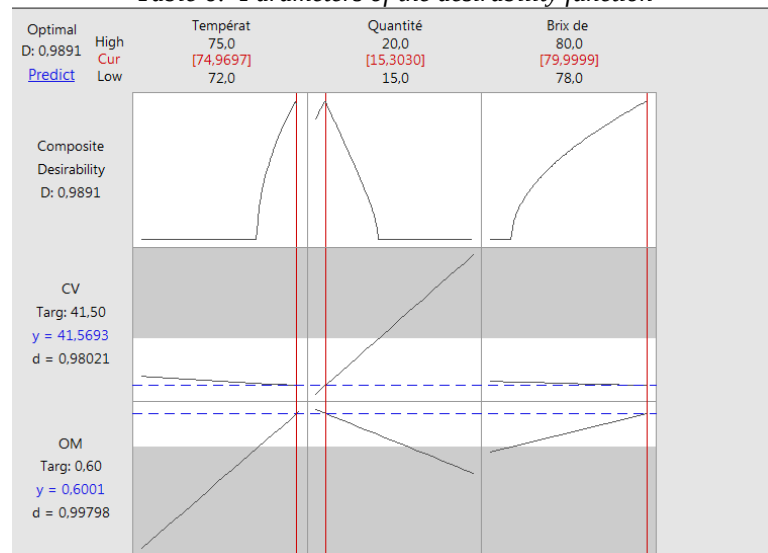


Figure 16: Desirability study for the first jet –Minitab

factors		Predicted Responses		Desirability	
Temperature	74,9697°C≈75°C	OM	0,6001	d(OM)	0,99798
Quantity of seed	15,303L≈15,5L	CV	41,5693	d(CV)	0,98021
Brix graining	79,99%≈80%			D global	0,9891

Table 7: Results of the desirability study for the first jet

To ensure the validity of the values obtained by all of the above study we chose to perform tests in accordance with the different values of the parameters obtained. Table 7 shows some of the results. After fixing the parameters in their optimal values, we observed that the two responses OM and CV are within the tolerance intervals. The boxplot of fig. 18 compares the distribution of the two responses before and after the improvements.

	Factors influencing grain size						Responses	
Test Number	Quantity of seed (L)	Vacuum temperature (°C)	Brix of graining (%)	Rise pressure (mbar)	Grinding time (h)	Quantity of sugar / Qty Alcohol (L)	OM (mm)	CV (%)
1	15	74,5	80	900	3,5	13	0,589	41.502
2	15,5	74,7	80	900	3,5	13	0,594	39.331
3	15	74,9	80	900	3,5	13	0,631	40.225
4	16	75	80	900	3,5	13	0,611	41.003
5	16,5	74,7	80	900	3,5	13	0,597	41.123
6	15,5	75	80	900	3,5	13	0,601	42.015
7	16,5	74,5	80	900	3,5	13	0,618	41.917
8	16	75	80	900	3,5	13	0,581	41.622
9	15	74,8	80	900	3,5	13	0,571	41.851

Table 8: Parameter Check Plan

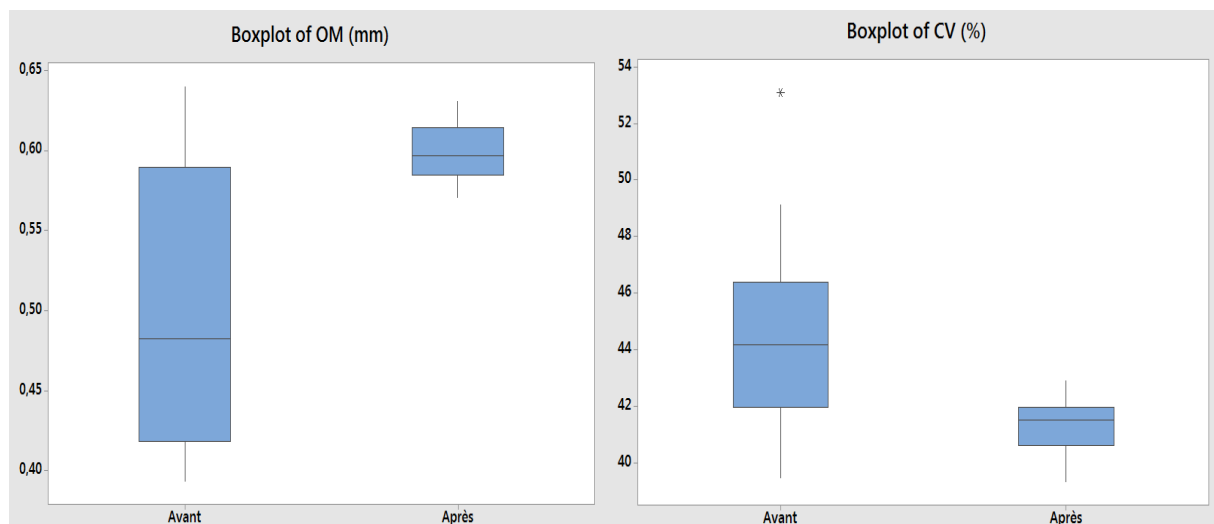


Figure 17: Boxplot of OM & CV before and after improvements

4.5. New Process Capability

We made 154 samples over 14 days, 11 samples per day to see the percentage of values that are now statistically included within the OM and CV tolerance range. We have recalculated the new capability of the process. For the Average Opening (OM): We have $1.00 < C_p = 1.04 < 1.33$ so our process is improved compared to the initial state whose value was 0.43. So we have $C_{pk} = CPL \approx CPU = 1.03$ so our process is centered around the mean OM = 0.6mm. For the Coefficient of Variation (CV): We obtained a capability of $C_p = 1.61$ which implies that our process has improved.

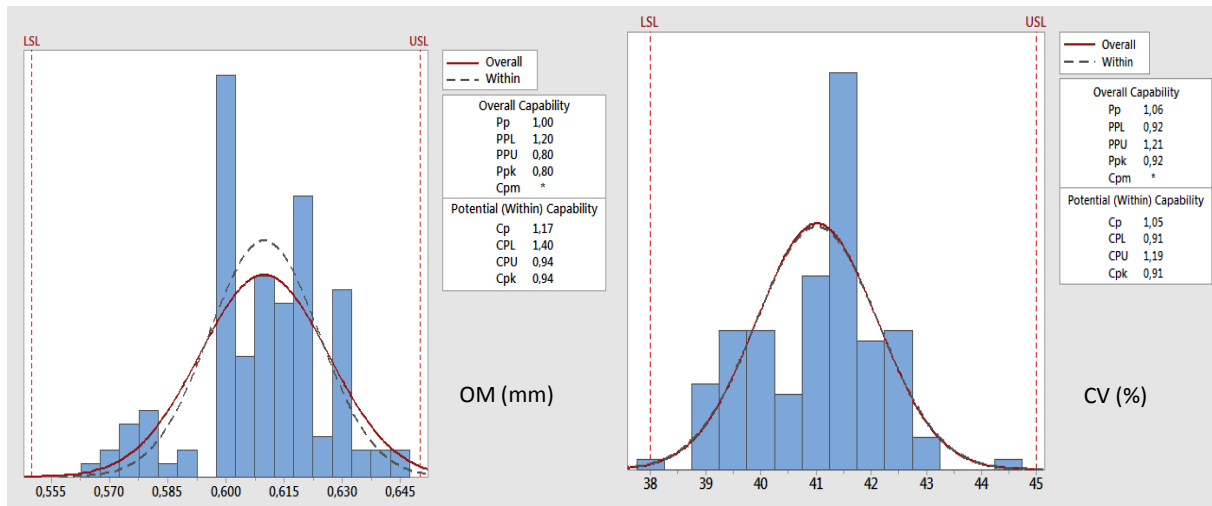


Figure 18: New Process Capabilities for Both Responses

VI. CONTROL AND STANDARDIZE

The standardization of the process control parameters ensures that the project lasts in time and that the proven solutions continue to be applied correctly within the company.

5.1. The sustainability Audit

It aims to ensure that CTQ (Critical to Quality), which are OM and CV, are well measured and monitored, that a minimum of formalization exists and that the procedures are applied correctly. This audit will enable the work team to ensure the sustainability of the results of this project. We propose to do a monthly audit following an audit grid and to see the evolution of the Particle Size Distribution of the white sugar in a daily way and to act in case there is a problem as soon as possible.

5.2. Control Charts

To follow daily the process and indicators and continuously carry out improvement actions, we realized the control charts in a spreadsheet shown in Figure 20.

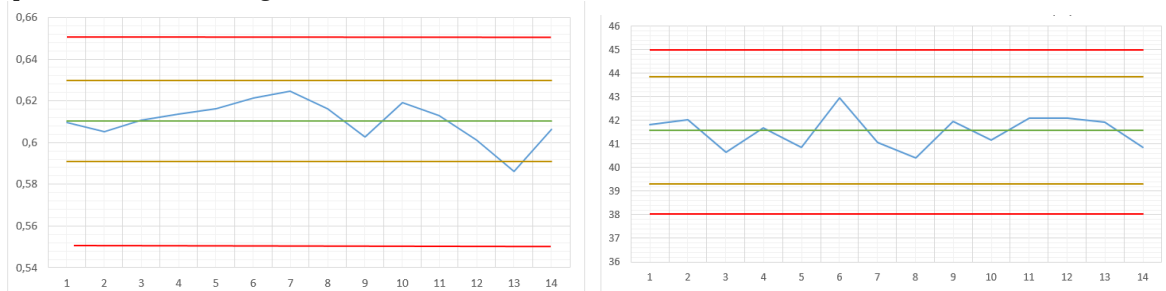


Figure 19: Control Charts

VII. QUANTIFICATION OF EARNINGS

6.1. Z process

To compare the initial state and the current state, we compare the Z-process (quality level achieved) of the two responses OM and CV. The Z process of OM and CV were respectively 0.54 and -0.06. After the study and implementation of an optimal model as well as the standardization of the parameters values influencing the white sugar granulometric state, we reached a level of quality Z of OM and CV of 2.40 and 2.74. Table 8 summarizes the qualitative gains from this project.

	Average Aperture (OM)		Coefficient of Variation (CV)	
	Before Improvement	After Improvement	Before Improvement	After Improvement
C_p	0.43	1.17		1.05
P_p	0.41	1.00	0.26	1.06
$Z_{process}$	0.54	2.40	-0.06	2.74

Table 9: Summary of qualitative gains

6.2. Money earnings

White sugar that meets quality requirements increased from 69.7% to 92.21%. The refinery reaches a daily output of 2650 tons; Of which 50% of its capacity is destined for export, ie 1325 tones. The total cost of producing one ton of granulated white sugar is \$ 700/ton. We then gained a cost of non-quality equal to $(92.21\% - 69.7\%) \times 1325 \text{ tones} \times 700\$/\text{Tone} = 0.208\text{M}\$/\text{day} = 76.2 \text{ Millions } \$/\text{year}$.

VIII. CONCLUSION

In this case study of the particle size distribution of white sugar optimization within the refinery by the Six Sigma approach, we aim to improve the quality of white sugar mainly characterized by indicators Opening Average (OM) And Coefficient of Variation (CV). To achieve our goal, we adopted the Six Sigma approach through the six steps of the DMAICS. We have first defined the key indicators of white sugar quality, namely: OM whose target is 0.6mm with a tolerance of $\pm 0.05\text{mm}$ CV varying in the range [38% - 45%]. We then studied the performance of our measurement system by the study of repeatability and reproducibility as well as the performance of the crystallization process of the first jet by studying capability and performance indicators. To analyze the influence factors, we used the experimental design and the desirability study which allowed to optimize both parameters (OM, CV). To put the process under continuous control we proposed control charts. The implementation of the standardization sheets in parallel with the proposed audit schedule will ensure the sustainability of the improvements. The results were considerable: the process capability has increased from 0.41 to 1.00 for the OM and from 0.26 to 1.06 for the CV, and Z process from 0.34 to 2.40 for the OM and from -0.06 to 2.74 for The CV. This translates directly into gains of 22.51% of production which costs MAD 76.2M\$/ year. In order to ensure the sustainability of this success and to establish a continuous improvement loop, we propose to study the other two jets to improve the total efficiency of the crystallization station.

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