

PROJECT 1 OF THE CAREER

INTRODUCTION

1.1 **Project:** Mitigation of Raw Material Quality Impact and Its Process on Sugar Colour.

Institution: Universidad del Valle - Cenicaña (Sponsor company).

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Position: University Practitioner in Chemical Engineering.

Location: Candelaria, Valle del Cauca - Colombia.

Date: August 1, 2016 - January 31, 2017.

BACKGROUND

1.2 I developed the second part of the project presented to Colciencias, "Mitigation of raw material quality impact and process conditions in the colour of final product", funded by Cenicaña, which was my work in my professional elective "Industrial practice in chemical industries" in my Chemical Engineering undergraduate program in 2017.

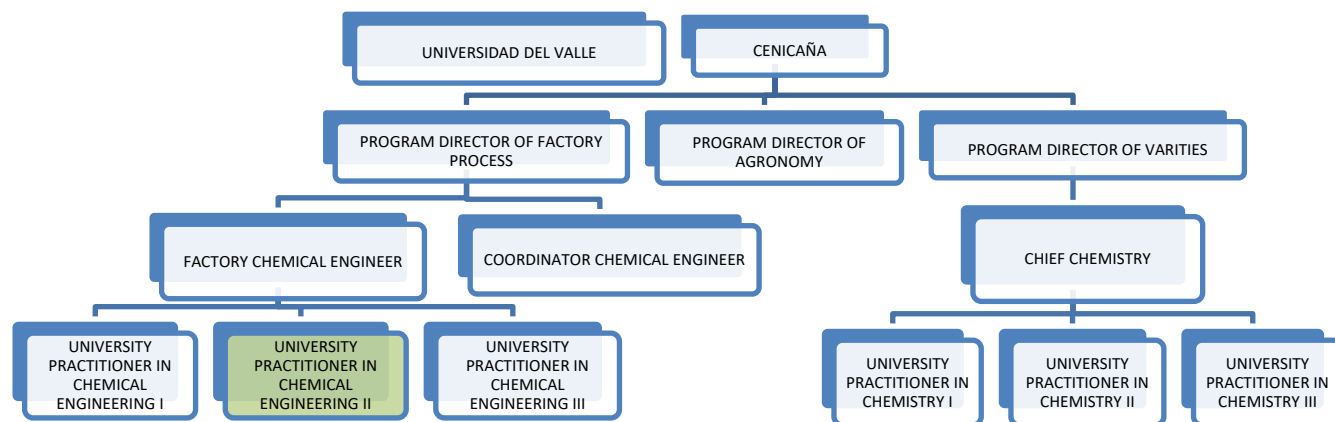
1.3 Cenicaña, the Sugarcane Research Centre, is a private, non-profit corporation founded in 1977 by Asocaña, the Association of Sugar Cane Growers of Colombia, and financed by sugarcane suppliers and sugar mills, such as Carmelita, Central Tumaco, and Incauca, among others.

1.4 Cenicaña promotes innovation by managing research and development projects in line with the strategic planning of the agro-industrial sector. It provides specialised services in information and documentation, information technology, economic and statistical analysis, technical cooperation, and technology transfer in research programs and agricultural production processes.

1.5 The project consisted of the analysis of methodologies to reduce the impact of changes in cane quality on the colour of the materials' production process of direct and refined white sugar, with the following considerations and objectives:

- Perform colour balance throughout the sugar production process.
- Implement methodologies to identify and quantify colourant compounds and colour precursors.
- Identify operating practices and establish control strategies to reduce the use of colouring compounds in process materials and finished products.
- Evaluate the most appropriate technologies to mitigate the effect of changes in raw material quality on the colour of process materials and sugar.

1.6 Organisational structure for the project:



1.7 As a University Practitioner in Chemical Engineering, my responsibilities were:

- Perform colour balance in the sugar production process in each of the unitary operations until clarified cane syrup (mills, juice clarification, sludge filtration, evaporation and clarification of cane syrup), identifying the stages that most contribute to the colour of the process materials.

- Identify operating practices and establish control strategies that contribute to the reduction of colouring compounds in process materials and in the finished product (direct and refined white sugar).
- Evaluate the most appropriate technologies to mitigate the effect of changes in raw material quality on the colour of process materials and finished product (direct and refined white sugar).

PERSONAL ENGINEERING ACTIVITY

- 1.8 One of the most influential aspects of sugar quality is colour, which is determined by factors such as the percentage of mechanical harvesting, the quality of hand harvesting, and climatic conditions, as well as cane varieties and operating practices that generate colourants. Sulphitation of materials is the most widely used method to reduce colour in the sector at low cost (on average \$180/TC). However, it has disadvantages, such as restrictions on residual sulphite content in sugar, environmental emissions, and a limited range of action on colouring compounds, among others. Therefore, I realised the need for an exploratory study of the cost-benefit ratio of new technological alternatives for colour reduction in intermediate materials, to ensure the quality of the finished product.
- 1.9 I initiated the search for information on the sugar cane process, its operational variables, cane types, colouring compounds and laboratory analysis in the context of the sugar industry. Then, I performed a thorough analysis and concluded that evaporation is the most critical stage of the process that contributes to the colour of the sugar. I evaluated the behaviour of colour in evaporation at María Luisa's sugar mill (without automatic control), taking the following samples, as shown in **Figure 1**.

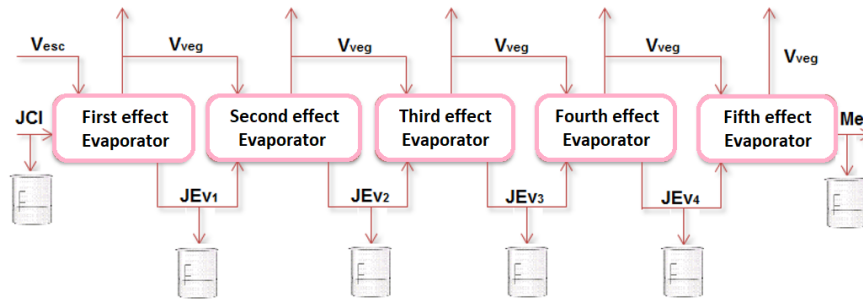


Figure 1. General scheme of sampling in the separation train (own authorship).

- 1.10 To ensure that the samples were representative, I considered the residence time of the juice in each of the evaporators as follows:

Table 1. Residence time during the evaporation train for taking point samples.

Juice Flow (m ³ /h)	Date	Residence time (min)					
		Evaporator 1	Evaporator 2	Evaporator 3	Evaporator 4	Evaporator 5	TOTAL
55.7	15/09/2016	1.9	1.4	0.9	1.1	1.5	6.8
39.1	19/09/2016	2.7	1.9	1.3	1.5	2.1	9.5
35.7	22/09/2016	3.0	2.1	1.4	1.7	2.3	10.5

The samples I obtained from the previous activity I analysed for pH, °Brix and colour; **Table 2** shows this procedure:

Table 2. Methodology for the determination of pH, °Brix and colour.

Methodology	Equipment and/or materials	Remarks
pH measurement	pH meter	Record the pH of the juice in the process
Dilution of the juice	Distilled water	Must be between 5 and 15° Brix
Filtration	Filter paper	Celite is added to the filter paper to improve and accelerate filtration.
Measurement °Brix	Refractometer	It takes into account the dilution to determine the actual value
Dilution of the juice	Distilled water	Up to 5° Brix
Increase or decrease of pH	pH meter	Up to pH 7 with NaOH or HCl
Filtration	0.45 µm filter	Cellulose acetate membrane filter
Absorbance measurement	Spectrophotometer	Measurement at 420 nm

I analysed the results obtained to evaluate the effect of the operation variables on the colour of the process materials. I requested the sugar mill to provide the evaporator level, temperature, and pressure values during the three monitoring periods. See **Figures 2, 3, 4** and **5**. The results obtained for colour, °Brix and pH:

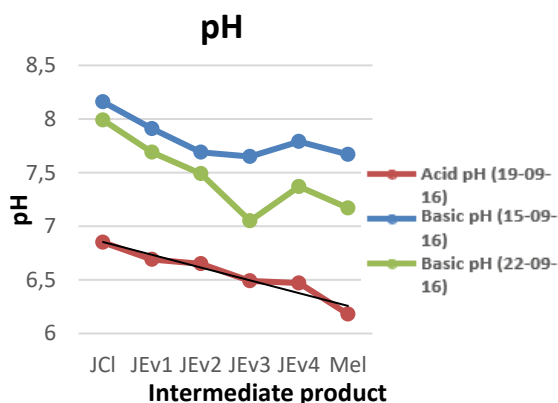


Figure 2. pH of the intermediate products of the evaporation train of María Luisa's sugar mill.

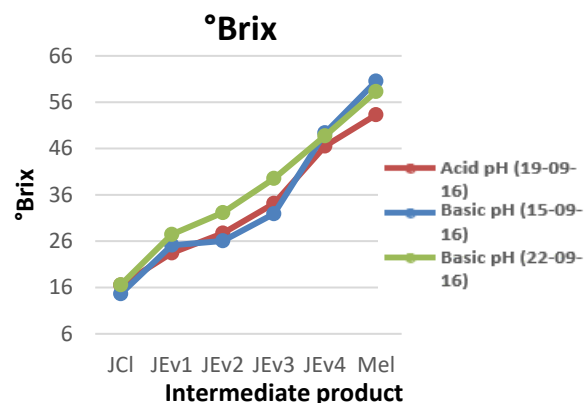


Figure 3. Brix of the intermediate products of the evaporation train of María Luisa's sugar mill.

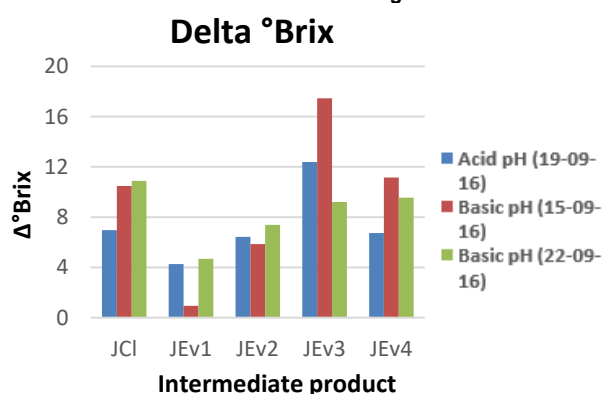


Figure 4. Delta of °Brix of the evaporation train of the María Luisa's sugar mill

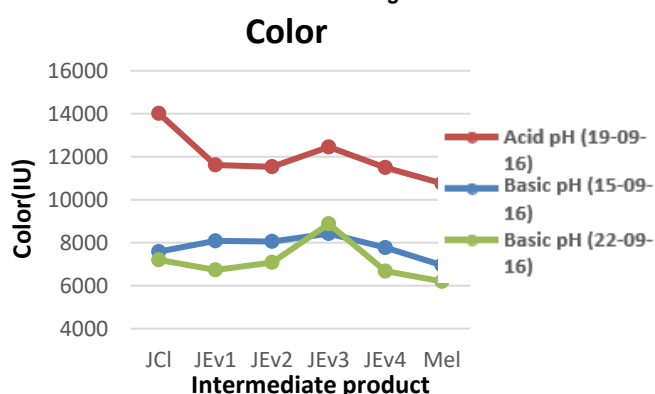


Figure 5. Colour of the intermediate products of the evaporation train of María Luisa's sugar mill.

Note: On 09-15-16, the evaporators were clean; on 09-22-16, they were dirty; and on 09-19-16, they were in an intermediate state.

*IU=ICUMSA Units

- 1.11 I analysed the results to identify a trend in the evaporator 3 condition that favoured the highest colour formation in the case of basic juices and one of the highest in the case of acidic juices, and the operating conditions of the sugar mill during sampling to explain the colour formation. I established the relationship between the most influential variables in the process —temperature, level, and pressure —with each evaporator and determined that the level value was crucial. See Fig.6

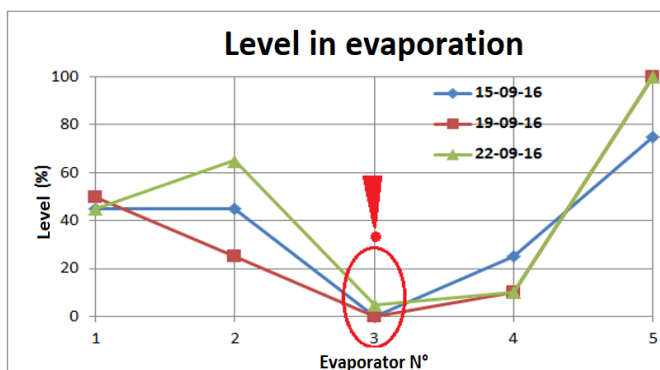


Figure 6. Level along the evaporation train for each monitoring of María Luisa's sugar mill (own authorship).

From **Figure 6**, I observed the low level in evaporator 3. According to Rein, the optimum liquid level in the tubes is between a quarter and a half of the height of the tubes, measured from the calandria (33% in the first evaporator, down to 20% in the last one). The low level in evaporator 3, along with the fact that the temperature it presents was very close to exhaust steam, could be the reason for the increase of colour, since there exists little liquid the heat transfer would cause a sharp rise in the temperature of the liquid, causing the juices that are ascending through the tubes to reach their caramelisation and part of these would be remained encrusted. I shared these findings with sugar mill personnel to ensure optimum levels in their evaporators and the importance of automating the level control.

- 1.12 I analysed that the second stage contributed considerably to the generation of sugar colour, after milling and before clarification. I carried out comparative tests of diluted juice with or without the addition of ascorbic acid and heating at 80°C, and its effect in attenuating colour. For this purpose, I took samples of the diluted juice from the mill area at various points throughout the process for testing and analysis. I subjected the samples to different experimental conditions to observe the effect of temperature and/or ascorbic acid on colour attenuation with 500 mL of juice, thus:
- 80°C, no ascorbic acid
 - 80°C, 0.2 g ascorbic acid
 - Ambient temperature, no ascorbic acid
 - Ambient temperature, 0.2 g ascorbic acid

Subsequently, I performed pH, °Brix and colour analyses. **Table 2** shows this procedure. See **Figures 7 to 10**.

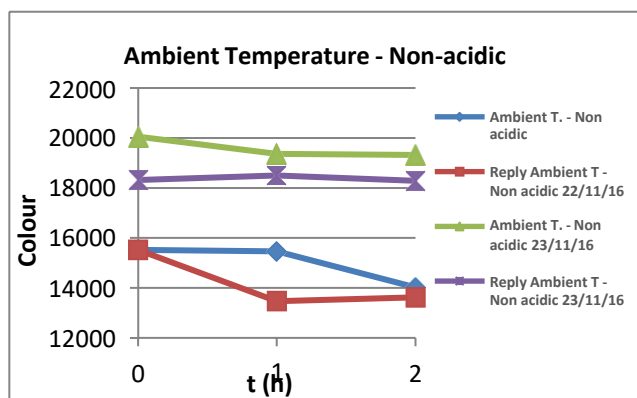


Figure 7. Colour of diluted juice, subjected to ambient temperature and without the addition of ascorbic acid.

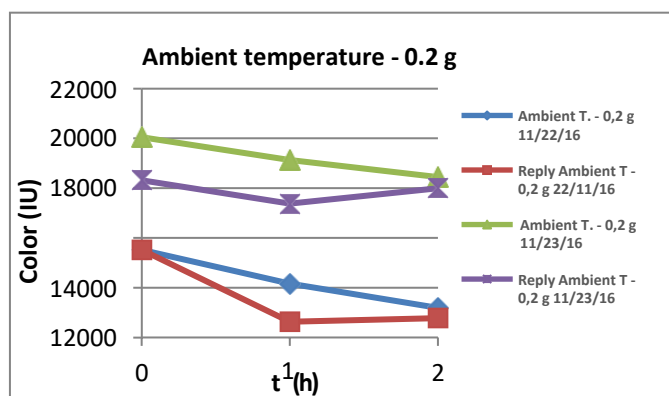


Figure 8. Colour of diluted juice, subjected to ambient temperature and with 0.2 g of ascorbic acid.

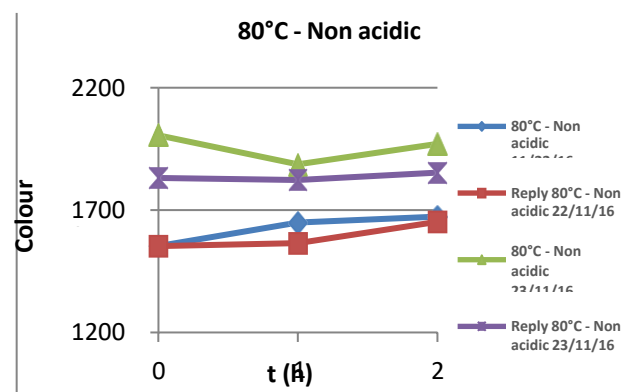


Figure 9. Colour of diluted juice, subjected to 80°C and without the addition of ascorbic acid.

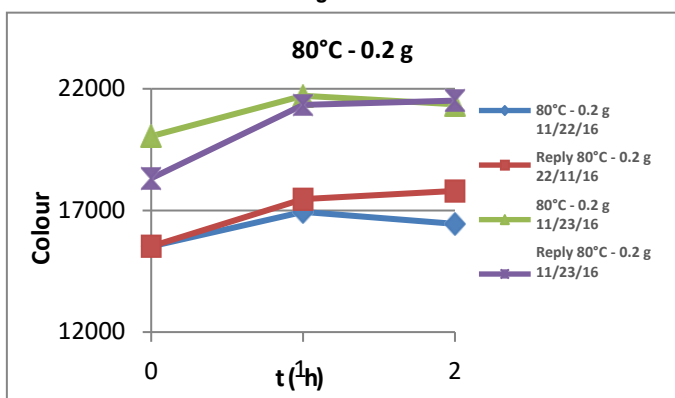


Figure 10. Colour of diluted juice, subjected to 80°C and with 0.2 g ascorbic acid.

- 1.13 From the analysis of the information, I concluded that, by adding only ascorbic acid, there is a significant colour reduction (7% and 5% for days 22 and 23, respectively), which gives indications that it can be used as a compound to attenuate the increase in colour by degradation, although it is not significant. In addition, increasing the temperature to 80°C did not affect the colour reduction in this compound.

1.14 I performed an analysis at the laboratory level to relate the operative variables that had an impact on the increase of sugar colour, such as temperature, pressure, pH and Delta °Brix. The next step was the benchmarking of decolourising alternatives that I researched, classified and proposed decolourising technologies for cane sugar, identifying alternatives that attenuate colour formation, which I classified into the following groups:

- **Ion exchange materials and adsorbents:** *Ion exchange resins, granular or powdered activated carbon, **Sepiolite** and **Octadecylsilyl silica gel****.
 - **Oxidisers:** *Ozone, hydrogen peroxide and Fenton's reagent.*
 - **Sulphitation:** *gaseous and liquid sulphur dioxide*
 - **Filtration:** *Ultrafiltration*
 - **Clarifying aids:** *Carbonation and cationic polyamine*
- *The alternatives marked in bold are the proposed alternatives.*

1.15 I developed the schemes for the alternatives based on the information collected through technological research and benchmarking, as well as laboratory and industrial assemblies. See **Figure 11** for activated carbon and **Figure 12** for ozone, which are used as examples of two of the 13 technologies in total:

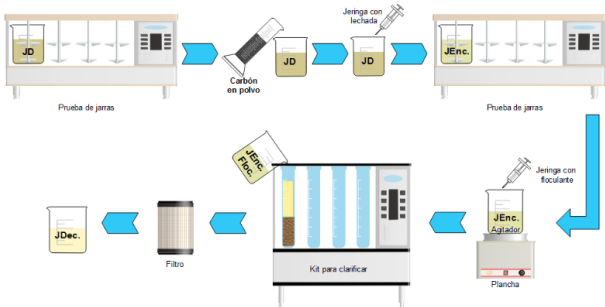


Figure 11. Laboratory assembly - Granular activated carbon

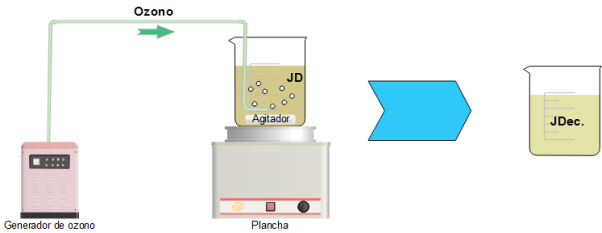


Figure 12. Laboratory assembly - Ozone (hot and cold)

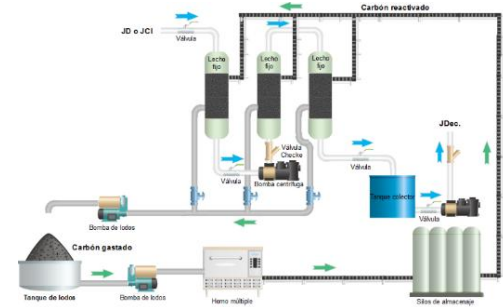


Figure 13. PFD - Granular activated carbon

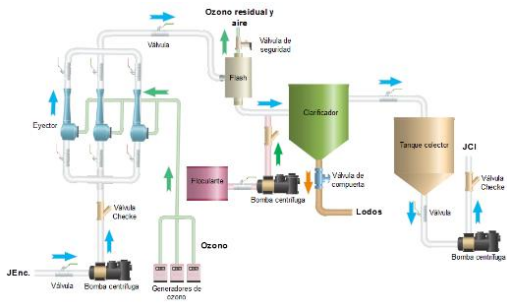


Figure 14. PFD – Ozone in hot

1.16 I considered the criteria to evaluate the technical and economic feasibility. These were:

- ❖ Economic (45%)
 - Capital - Purchase of equipment (18%)
 - Operation - Supplies and services (17%)
 - Maintenance (10%)
- ❖ Decolourisation efficiency in %IU (35%)
- ❖ Risk (20%)
 - Environmental (12%)
 - NFPA 704 classification (8%)
 - Human health (5%)
 - Flammability (1%)

Table 3. Scores per position in each criterion

Position	Score
1 st	13
2 nd	12
3 rd	11
4 th	10
5 th	9
6 th	8
7 th	7
8 th	6

1. Decolourisation efficiency

Table 4. Classification by decolourising efficiency

Alternative	Decolourisation efficiency(%UI)	Score	Position
Granular or powdered activated carbon	60	60	4 th and 5 th
Powdered activated carbon	80	80	2 nd
Ion exchange resins	75	75	3 rd
Sepiolite	38	38	9 th
Ozone	60	60	4 th and 5 th

2. Risk

- Environmental: I considered the environmental impacts of technology based on the International Chemical Safety Cards.
- NFPA 704 classification: I coordinated with Human Resources personnel the health, flammability, reactivity and special care risks by technology and inputs in flammability, reactivity, and specific hazard.

1.17 I included the "Complexity - Advantages, Disadvantages and Limitations" section, which is not linked to the criteria part of the technologies, presenting the advantages and disadvantages of the decolourising alternatives and their limitations in the process. I obtained the following results:

Table 5. Overall results and score for the risk criterion, (Own Authorship).

Alternative	Risk (20%)														
	Environmental (12%)			NFPA 704 classification (8%)											
				Human health (5%)			Flammability (1%)			Reactivity (1%)			Specific hazard (1%)		
	Position	Score	Real score	Position	Score	Real score	Position	Score	Real score	Position	Score	Real score	Position	Score	Real score
Granular activated carbon	1 st to 5 th	11	1,320	1 st to 4 th	11,5	0,575	11 th to 13 th	2	0,020	8 th to 10 th	5	0,050	1 st to 10 th	8,5	0,085
Powdered activated carbon	1 st to 5 th	11	1,320	1 st to 4 th	11,5	0,575	11 th to 13 th	2	0,020	8 th to 10 th	5	0,050	1 st to 10 th	8,5	0,085
Sepiolite	1 st to 5 th	11	1,320	5 th to 8 th	7,5	0,375	1 st to 8 th	9,5	0,095	1 st to 7 th	10	0,100	1 st to 10 th	8,5	0,085

I based my assessment on the risks involved in handling the materials required for decolourising alternatives, considering their characteristics and NFPA 704 and ISCS (International Chemical Safety Cards) standards.

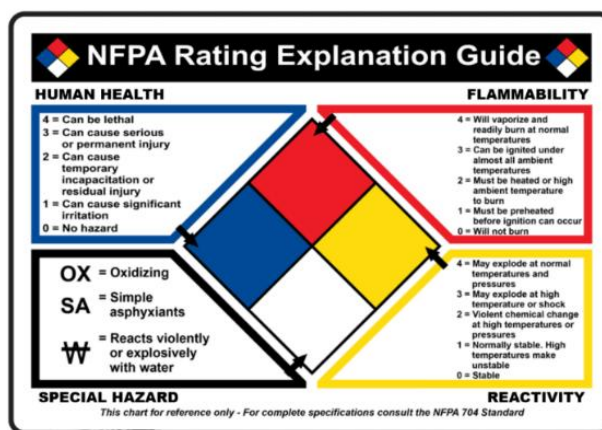


Figure 15. NFPA 704

And with the next step:



Figure 16. Scale for measuring and scoring the risks of each technology

The above scale ranges from the lowest to the highest, where 0 indicates no risk of any kind, and 4 indicates fatal, irreversible, and/or incalculable damage. See **Table 6**:

Table 6. Final ranking of alternatives. (Own Authorship).

Alternative	Finalscore	Final position
Carbonation	10,990	1 st
Powdered activated carbon	8,915	2 nd
Sepiolite	7,830	3 rd
Cationic polyamine	7,295	4 th

- 1.18 With the above results, I established the next stage of the project, which was presented to the work team in a presentation at the company, printed and record the work, so future practitioners in chemical engineering could continue with the subsequent stages, which would be the application of the proposed technologies at the laboratory and industrial levels, in accordance with the schedule of activities allocated to the project. See Table 7.

Table 7. Timeline. (Own Authorship).

Description of the activity		Start		End	
		Month	Year	Month	Year
1	Quantify the colour contribution to the diluted juice of each part of the sugarcane	3	2015	9	2015
	Estimation of the effect of operating variables.	7	2015	12	2015
	Perform colour balancing throughout the sugar production process.	5	2015	9	2015
	Results report Objective 1	1	2016	2	2016
2	Apply available techniques (spectrophotometric and chromatographic).	1	2015	12	2015
	Implementation of spectrophotometric	7	2015	6	2016
	Results report Objective 2	6	2017	7	2017
3	Characterisation of colouring compounds and precursors in Objective 1.	1	2016	8	2016
	Design and propose control strategies in the processing area.	7	2016	12	2016
	Results report Objective 3	1	2017	2	2017
4	Evaluate technically viable technological and process alternatives.	8	2016	2	2017
	Reduce the impact of changes in cane quality on the colour.	8	2017	10	2017
	Final results report	10	2017	12	2017

SUMMARY

- 1.19 I concluded that tests should be performed at both the laboratory and industrial levels, specifically in the sugar cane process with carbonation, powdered activated carbon, and Sepiolite, to obtain high-quality sugar with colour mitigation as the final product. I established the next stage of the project by performing tests at the laboratory level of the decolourising alternatives selected in the preliminary report, considering that the juice is at the same conditions (harvest time, variety, manual or mechanical cut and burned or in green); since these have a significant effect on the generation of colour.