COLOUR DEVELOPMENT

(Origin of colors, development during processing ,Prevention and Removal)

A case study at Mirpurkhas Sugar Mills

ABSTRACT

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The color of sugar is most important commercial attribute and sugar mills are spending much resources to comply with the market requirements on the color of their product . When one say "colorant" he means, the material causing the color.
Pure sucrose has no colorant but it appears colorant due to inclusions of a plenty of complex compounds. Hence the colorant is the generic term used to cover a variety of components which contributes to the color of the sugar. The sugar colorants are very extremely complex in nature and not easy to quantify.

There can be a variety of sources from where the sugar colorant originates but mainly they can be summarized in two major sources as under.

1. Plant pigments

   Natural colorants which originates from sugarcane itself. These consist of the organic components like plant pigment, waxes etc. etc. The intensity of such colorants is the function of the many contributory factors viz. cane variety, Interestingly the color within the different parts of the sugarcane stalk varies considerably.
   During clarification these decomposes to form polyphenolic compound with subsequent enzymatic browning.

2. The color development during the processing.

   The majority of such colorants is formed from the following two types of chemical reactions:

   (i) The color formed due to the Alkaline Degradation (Hexose Alkaline Degradation Product) and Melanoidins –type material resulting from reaction of amino acids and reducing sugars. These are formed comparatively at low temperature and high pH.

   (ii) Caramel type color products, which are produced by thermal degradation of sucrose and reducing sugars, triggered on high temperature.

The color management is a very significant and paramount function of a technologist. This management can be categorized as a variety of activities like the identification and the measurement of the colorants, prevention of formation of new coloring compounds during the process and the last but not the least is the removal of coloring compounds.

Recently Pakistan Sugar Industry has developed more sensitivity about the quality of sugar for the use of consumers in general but in particular for the pharmaceutical and beverages use.

Most of sugar mills are trying to upgrade their plants to achieve modern standards. Within its limited resources Mirpur Khas Sugar Mills is one of the sugar mills who has achieved nearly all international quality standards in sugar. In this presentation some factors are cited which causes color development. steps taken for analysis and prevention , also has a reference of processes applied in Mirpurkhas Sugar for color removal.

INTRODUCTION:

COLORANTS:

" sugar color with its associated impurities and precursors is complex mixture of diverse composition , difficult to describe in practical terms" (smith & Gregory)

Sugar colorant is not a single molecular species, but it is a complex mixture of compounds. (Baunsgaard 2000) .

They can be divided into two categories, natural -plant pigments, color precursors and those formed during sugar processing. During sugar manufacturing some of these compounds are removed and many of them undergo numerous reactions leads in the formation of further dark color compounds.

Coloring substance are undesirable impurities in sugar cane juice mainly are organic non sugars, They have adverse effects on crystallization process and refine sugar product . Due to complexity of Sugar colorant it is not easy to quantify them hence having limited sources at Mipurkhas sugar mills color is only measured as the total effect of all colorants on light absorbance @ 7.0 pH.

Mirpurkhas sugar mills one of the sugar mills in Pakistan that produces refine sugar of high quality that comply utmost all international standards.

This paper briefly reviews current knowledge on color and the associated color precursors, development of colors at different stages of processing.

It also out lines quantification of colors at different stages in MSM, processes / techniques viz. (Juice Defecation, Juice clarification , refinery of melt liquor through carbonation, phosfloatation, filteration, and treatment with PAC.) employed by Mirpurkhas sugar mills for prevention and removal.

Anx-1 : Colorant Classification (according to their properties) (purolite)

	Natural Colorants / Cane Pigments			Color Development During Juice Processing		
Colourants Name	Flavonoids (polyphenols), anthocyanin, flavones Melanins	Phenolics (Benzoic, cynamic, chlorogenic , gallic & cafeic acid)	Chlorphylis Xanthrophylis Carotene	Melanoidins	HADPs	Caramels
Reaction Products	Oxidised by enzyems, form colored compound.	Generally Colorless, Oxidise/re-act with amines-iron, form colored product	Green pigments , removed in clarification	Maillard Reaction Products	Hexos Alkaline Degradation Product-s	Sucrose, Glucose and Fructose Degradati-on at high temperat-ure

Anx-2 Properties of colorants (SB Davis)

PROPERTY	**Monomeric**	**Intermediate**	**Polymeric**
COMPOSITION	**Plant pigments Mainly flavonoids,**	**Factory colorants eg. ADF-**	**Factory colorants e.g. Melanoidins and caramals**
MOLECULAR WEIGHT MW -Daltons	**< 1000**	**1000-2500**	**>2500**
IONIZATION	**Neutral at low pH**	**Cat-ionic below pH 5, An-ionic above pH 6**	**Same as intermediates**
POLARITY	**Least polar**	**Intermediate**	**Polar**
TYPE	**Weak acid**	**Weak acid + amphoteric**	**Amphoteric**
Indicator value IV	**5-40** **pH sensitive**	**3-4** **Intermediate**	**1-2** **pH insensitive**

Plant pigments

These are Primarily, chlorophyll, xythophyle, sacharatin, tannins and flavonoids / anthocyanins, (polyphenols) and phenolic compounds.

They Make two third color in raw sugar. (smith &Paton 85).

Their Purple / Red pigment changes into dark green with addition of lime (R.B.L Mathure)

These are unstable in neutral or alkaline solutions, and decomposed by heat,

These are Largely removed or destroyed in clarification, but small amount is found in polymeric colorant in raw sugar. (Godshall and grimm 94)

Chlorophyll, Xanthophyll and carotene are:

- **insoluble in water**
- **eliminated early in the cane-juice processing.**
- **changes green/orange to yellow color in alkaline solution and give colors with ferric ions. (Harbone,84)**

Phenolic compounds

These are Present in plant as Esters. The most common phenolic ester in cane is chlorogenic acid, an ester of caffeic acid with quinic acid . Ranges from colorless to yellow. (MA Godshall)

Juice continue come in contact with metallic iron surfaces and tend to increase intensity of color due to absorption of iron salts. These form dark colored polyphenols with Iron in ferric state specially in acidic medium.

Melanin is reaction product of amino acids with phenolics as well as very dark enzymatic products of phenolics. (Godshall). They have detrimental effect on color of sugar as they crystalizes with sugar.

Oxidized enzymatically to polymeric color in cane juice (prior to heating)

Flavonoids

These are pH-sensitive colorants of low molecular weight .They are oxidized by enzymes and form enzymic browning colorants of high molecular weight.

They are not removed to any great extent during clarification and hence included within the raw sugar crystal. Their Ionization increases with increase in pH

COLOR PRECURSOR

Color Precursor are colorless chemically related plant pigments, form colour in processing. Simple phenols, reducing sugars and amino acids are significant color precursor

Amino nitrogen derivatives (amino acids/aspartic acid) can react with reducing sugars on heating, to form melanoidins which are polymeric type colorants.

Phenolic derivatives (benzoic, cinnamic and caffeic acids) are highly reactive and form colored complexes with iron. They can also polymerize enzymatically to form color complexes in the early stages of processing before heating. (P.smith, N.H, Paton)

General observations of sugar researcher.

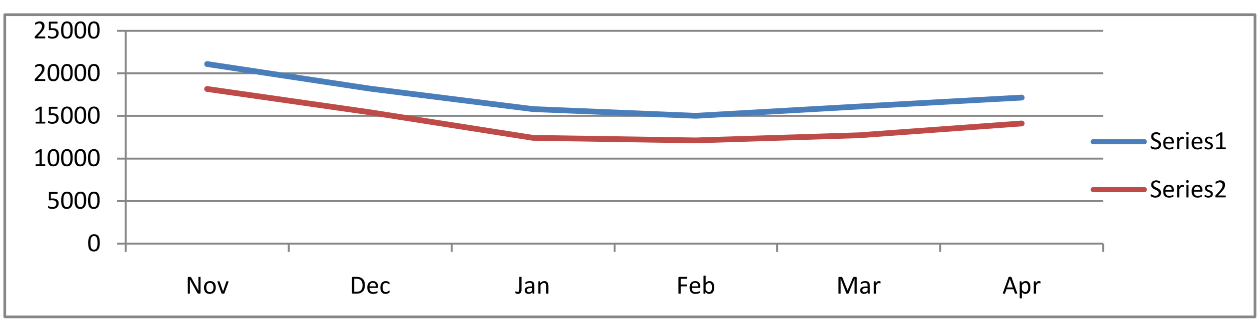
The quantity, quality and type of pigments and colorants in juice are dependent upon:

- **The variety and maturity of sugar cane**
- **Climatic condition**
- **Agricultural practices (use of nitrogenous fertilizers) stored by plant as amino acids.**
- **Cut to crush delays**
- **Amount of trashes incorporated into the crushed cane**
- **Soil condition**
- **Juice extraction practices. (lionnet, Reid, Anon, Ivin, Doyle) .**

Seasonal trend.

"Its seasonal trend that the colors are higher in the start and end of season and the lowest in the middle when the cane quality is at its best.. This confirms that the color due to the cane is a very significant proportion of the total color in the mills." **Smith (1990)**

Anx-3, MSM Mixed juice color trend



Anx-5, Colorant Controlling work at MSM

Particulars	Average Results	
SEASON	2006-07, 2007-08 (Two seasons)	2012-13 , 2013-14 (Two seasons)
Tops & Trash % in Cane	3 - 5 %	Less than 1 %
Inferior Variety % Cane (Tritron, Bansi, B-4360, NIA-98, GT-11, PR1000, BF-241, S-95 etc)	8 - 10 %	less than 2 %
Burnt cane	0.4%	0.08%
Immature Cane %	2 - 4 %	Less than 1 %
Dextran quantity in First Expressed Juice	9000 ppm	4000 ppm
First Expressed Juice Color IU	17220	14035

Anx-6 Pics of poor quality sugar cane

In 2006-07 & 07-08 colors were on higher sides there were many reasons viz. %age of tops and trashes, immature cane, stale cane, high ratio of polysacharides /dextran, inferior cane varieties were on upper side then last two years. %age of immature cane raised the R.S % and polysaccharides, those caused color formation and hurdles in process.

<u>Enzymic reaction</u>

Phenols may be oxidized to dark colored polymers by enzymic oxidation or non-enzymic oxidation, and these polymers contribute to the color of raw sugar.

The enzymes involved in browning are polyphenol oxidases (PPO) (N.H.Paton)

Over the Half of color of raw juice is due to enzymatic reaction. (Coombs & Baldry 1978).

Rate of enzymic oxidation that leads to browning depends on the concentration & nature of phenols, enzyme activity, pH and temperature. (Macheix et al., 1990)

Enzymic oxidation reaction continues until the enzyme is inactivated by heat.

<u>Color extraction, formation in milling</u>

The color –sugar ratio increases along the milling train from 20-30%.

Excessive maceration and juice extraction also effect the color

withdrawal. The higher extraction gives the higher juice color. (P.smith, N.H, paton).

You can see it in anex: 13 primary juice colors are 14035 IU while Mixed juice colors 16295 IU.

Dextran has indirect effect in color formation. Dextran is formed by the attack of bacterium Leconostic Masscentriodes resulting decomposition of sucrose unit into glucose & fructose. Bacteria utilizes the energy of glucose-fructose bond for dextran formation (Stanier et al. 1987) .

Delayed in cut to crush time, stale and burnt cane, supply, temperature, moisture and residence time , poor sanitation at mills enhance the production of dextran. The dextran formation raises many problems in sugar processing by increasing juice viscosity and poor clarification and crystal elongation) . (kitchen 88, Morel due boil 91, Muller 81).

Dextran not only causes the color formation but also has harmful effect through out the processing. (Kim & Robyt, 95)

Openion of Technologists:

it was reported that for each 1% addition of tops to clean cane, the color of clear juice was increased by 1.3%, (chen ,85)

Green leafy trashes and tops has dexterous effect on color and turbidity of juice and they also increase the polysaccharides in juice. (purchase 91)

Steps taken by MSM for prevention.

For prevention of all above contamination or reduction , Good quality cane was procured , farmers were motivated for cultivation of good quality cane.

The sanitation, (mechanical cleaning with hot water or steam at requisite areas) and biocide/ bleaching powder application , usage of pensilien were empolyed to reduce or diminishes the microbiological growth or inversion of sucrose resulting reduction in color.

In 2006-7 & 7-8 Primary and Mixed juice colors were 17220 , & 20302 respectively and in 2012-13, & 13-14 these were 14035 & 16295 Hence total reduction found 18.5 %

Color removal and formation during clarification

Clarification removes 20-25% of input colors including low and

some high molecular weight, (P.smith & N.H.Paton)

Some new Colorants also form during clarification, such as polymers from the

alkaline degradation products of reducing sugar and oxidation products of phenolics.

Melanins in cane juice is very little removed in clarification and this colorant

constitutes about 60 % of the color in the clarified juice (Paton 1992)

Presence of baggacillo causes biological contamination and increase in color of

clear juice. It increases with application of lime and heat. (Mathur).

Prevention steps at MSM:

-Temperature and pH of juices properly maintained.

-Retention of low pH juice avoided.

- To reduce contamination of baggacillo rotary screen of Slot 0.35 mm at mill house

and 0.25 mm for screening clear juice were Installed. (after 20067- 07-08)

- Same capacity of clarifiers were used for 7000 TCD, hence, retention time reduced,

Input colors were also lesser, therefore 13.87% reduction in color obtained during

clarification.

Anx-7 Analysis of clear juice

	Particulars	Unit	2006-07 & 2007-08	2012-13 & 2013-14
Clear Juice	Color	IU	17846	14209
	p.H	value	7.0	7.1
	R.S	% on Solids	4.3	4.0
	P^2O^5	PPM	55	57
	TSS	G/L	6.5	2.4
	CaO	PPM	932	826
	Clarity	%	94.5	95.3
	Clarification Effect	%	8.6	9.2

-In 2006-07 and 07-08 color removal in clarification was 12.09 % and in season 2012-13 and 13-14 it is 16.39 %

- In season 2012-13 & 13-14 Color of CJ were obtained 20.38% lesser as compared to season 2006-07 & 07-08

<u>Color development across evaporators:</u>

- **Thermal decomposition of fructose & hexose mainly, and glucose to lesser extent.**

  occurs under alkaline conditions, increase with time and temperature. The reaction products are brown in color and acidic in nature. Leading to inversion in sucrose, and further color formation, Amines are to be thought involved in the reaction.

- **Degradation occure in evaporators (even operating Under normal conditions)**

  mostly in first two effects.

- **Increase in glucose % sucrose ratios. pH and purity drops and colors develops.**

- **pH drops is due to formation of organic acids. & scaling out of basic calcium salts.**

At Mirpurkhas Sugar Mills:

-During seaon 2006-07 & 07-08, input colors were on higher side , Robert evaporators were used as first effect due to long residence and contact time of juice with tubes potentially large sucrose losses and color formation due to inversion.

-In last two years FFE were used as first effect color formation and inversion losses reduced to 4.27% & color reduction obtained 30.52% respectively as compared to 2006-07, 07-08

Anx-8, Color development across evaporator (based on P.smith & Paton findings)

particulars	season 2007-8 and 2008-9					
	pH	RS%	% sucrose Inverted	Brix %	Color	% color developed
Clear Juice	7.0	4.19		13.4	17750	
After 1st Effect	6.8	4.33	0.180	27.1	18103	
After 2nd Effect	6.7	4.38	0.063	39.8	18253	4.39%
After 3rd Effect	6.6	4.41	0.038	50.1	18374	
After 4th Effect	6.5	4.43	0.025	58.3	18468	
After 5th Effect	6.5	4.44	0.013	65.2	18530	
	season 2012-13 and 2013-14					
particulars	pH	RS%	Inversion rate	Brix %	Color	
Clear Juice	7.1	4.03		13.8	14320	
After 1st Effect	6.9	4.15	0.150	29.7	14453	
After 2nd Effect	6.9	4.19	0.050	41.3	14563	3.05%
After 3rd Effect	6.8	4.22	0.038	51.2	14650	
After 4th Effect	6.7	4.24	0.025	60.8	14716	
After 5th Effect	6.7	4.25	0.013	69.3	14756	

<u>Averag chlorogic acid & flavonoids in Raw juice , by HPLC, (N.H Paton) Average Phenolics & Amino Nitrogens .</u> G.R.E (Lionnet)

Parameters	**Results**
Chlorogenic Acid	**10- 1060 mg/kg solids**
Neutral Phenolics (as sinapoyl glucose)	**110- 620 mg/Kg solids**
Flavonoids (as sinapoyl glucose)	**170-1340 mg/Kg solids**

<table>
<tr><td colspan="3">SYRUP</td><td colspan="3">Raw Sugar</td></tr>
<tr><td colspan="2">PHENOLICS ppm on Brix</td><td>Amino Nitrogens
ppm on brix</td><td colspan="2">Phenolics ppm on brix</td><td>Amino Nitrogens
ppm on brix</td></tr>
<tr><td>Caffeic cid</td><td>Gallic Acid</td><td rowspan="2">500 - 900</td><td>Caffeic Acid</td><td>Galic Acid</td><td rowspan="2">5 – 8</td></tr>
<tr><td>4000- 7000</td><td>800 - 1400</td><td>55 – 90</td><td>12 – 22</td></tr>
</table>

<u>Color formation in Pan boiling</u>

- **Melanoidins & Caramals : are the main colored product during pan boiling. Melanoidins form from Maillard type reactions(known as browning reaction) of amino acids with reducing sugars (non-enzymic browning). (gross & coombs 76) (M.A Goshall)**

- **Produced with the application of high temperature at high brix and low purity, also form with low heat over long periods. (newell 1979)**

  During pan boiling colors not only increases but crystalization steps also partitons the colorants between crystal & mother liquor.

- **Melanoidins increase the viscosity of sugar solutions and hence interfere in the boiling and crystallization stages.**

- **Color increase in each grade of boiling from A to C masscuite. (P.smith).**
- **Returns of B and C sugar typically increase color of A- massecuite. (Munday et al 1968)**
- **10-20 % color formation in C massecuite occurred during massecuite storage in the crystallizer. (N.H.Paton & H.R.Delaney)**
- **Caramals produced by thermal degradation and condensation reactions of sugars.**

  True Caramel is never formed under normal conditions , working on vacuum boiling.

- **Source of caramel formation is sometimes due to the crystalline crust on the surface of the coils or calandria tubes of the vacuum pans. If pan is not properly washed after the boiling strike.**
- **Factors which could effect color are circulation, Pan design (head,stirrer,tubes sizes, striaght sides no body flaring) , Temperature , Nature of impurities, size of crystal, and pan operation practices etc. (peter Rein)**

  In 2006-7 to 2007-8 colors of A/B/C Masscuits were 17385,38926 &73246 Respectively, Which were 24, 23 & 16% higher than last two years.

  Reasons were , during last two years A & B masscuites were boiled in continueous pans, good designe Ciurculators and jigging steam coils were mounted in batch pans, for temperature control Indian automated multi jet condensers were installed. Input colors were in a lesser amount of specially those which include in crystal therefore partition co-efficient and Purging efficiency improved and B/C sugar of lesser colors returned to pan for seeding, Consequently less amount of color obtained .

<u>Partition co-efecient & Purging efficiency *:*</u>

The partition coefficient is the ratio of massecuite color to the sugar color, Indicates the extent of inclusion of color in sugar crystal, a high partition coefficient indicates low inclusion in the crystal.

Purging effecincy is the ratio of impurities in Massecuite removed in molasses, Influenced by partition co-efficient. High partition co-efficient high purging efficiency.

The polymeric pH-sensitive caramels, alkaline degradation products and some quantity of melanoidins are removed by affination and crystallization

Alkaline degradation products and caramel have small tendency to deposit within the crystal mostly remain on the surface ,and remove in molasses.

Melanoidins attached within the crystal, not easily to remove in affination process.

Anx-9, MSM Purging efficiency /partition co-efficient

Raw Massecuite						
Particulars	season 2006-07 and 2007-08			season 2012-13 & 2013-14		
	A	B	C	A	B	C
Massecuite Color	17385	38926	73246	13109	29759	61470
Molasses Color	33797	67100	111680	25552	52394	95223
Sugar Color	974	10751	26271	666	7124	20217
Purging Efficiency	97.2	94.80	91.48	97.46	96.83	92.95
partition co-efficient / Ratio	17.86: 1	3.62: 1	2.79: 1	19.69: 1	4.18: 1	3.04: 1

COLOR DEVELOPMENT DURING STORAGE :

Color developed at the high storage temperatures , especially in PAKISTAN where the temperature reaches 48° C.

Color Increase with, both time and temperature . opinions are divided as to whether this color formation is due to caramelisation or melanoidin formation

It was proposed that the 0.05 % reducing sugars present in sugar crystal would under go slow caramelisation catalyzed by ash constituents especially carbonate, during storage.

Color formation could also be due to the reaction of polyphenole, not removed by conventional clarification method, and iron from factory equipment. (M.J. Kort).

Mother liquor within coglomerates crystal also causes color formation .

When refine sugar was made from sulphitation process, color formation was higher during storage, as unstable sulphited carbonyls are prone to losing the sulphite groups and release SO_2 over the time, resulting color returned

Anx-10, Color development during storage:

particulars	Manufacturing process	
	DRCS	DRC with Phosfloatation and PAC
sugar color (initial result)	39 IU	20 IU
moisture%	0.03	0.03
storage temp C	32 C* to 44 C*	32 C* to 46 C*
Humidity%	50 to 75 %	50 to 75 %
sugar color (after 6month storage time)	58 IU	26 IU
color development %	49 %	30 %

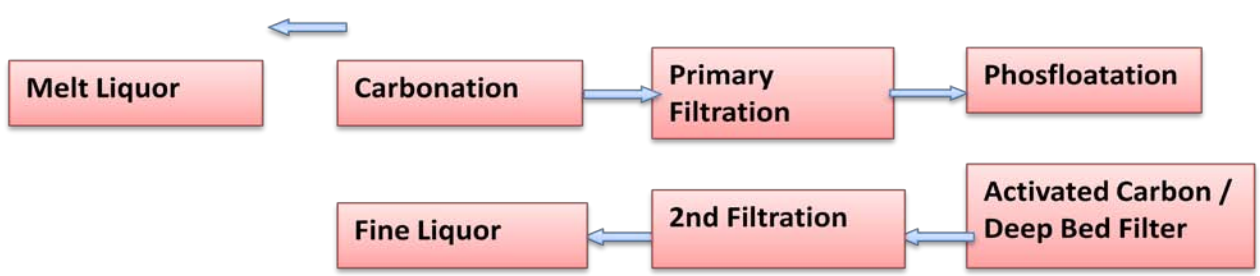
Anx-11,Color removal in different processes (SB DAVIS)

Colour Process	Phenolics	Flavonoids	Melanoidins	Caramels	ADF Products	Colour Precursors
Carbonatation	⬆ ∩	⬌	⬆	⬆	∩	
Sulphitation	⬆	⬆	⬆	⬆	⬆	⬆
Phosphatation	⬌	⬌	⬆	⬆	⬆	
Color Precipitants			⬆	⬆	⬆	
Oxidative Decolorants	⬆	⬆			⬌	⬆
Activated Carbon	⬆	⬆	⬆	⬆		
Ion Exchange	⬌	⬌	⬆	⬆	⬆	
⬆ Well Removed	⬌	Poorly Removed	∩	Formed in Process		

Process combination & selection

To produce good quality refined sugar and to achieve cost effective removal of colorants many processes have been developed over the years. In MSM different combination of processes were tried and found carbonation , phosfloatation with PAC is the better one in existing scenario. Input colors were +/- 20000 and final product was achieved of 20 -ICUMSA.

ADDITION OF PAC



Journey of color 20000 IU to 20 IU

Particulars	Average Results of Color IU		
	2006-07 to 2007-8	2012-13 to 2013-14	% of Color Decrease
Primary Juice	17,220	14,035	18.50
Mixed JUICE	20,302	16,995	16.28
Clear Juice	17,846	14,209	20.38
Syrup	18,631	14,638	21.43
A. Massecuite	17,385	13,109	24.60
A. Heavy	33,797	25,552	24.40
A.Sugar	974	666	31.60
B. Massecuite	38,926	29,759	23.55
B. Heavy	67,100	52,394	21.92
B.Seed	10,751	7,124	33.73
C. Massecuite	73,246	61,470	16.08
Final Molasses	111,680	95,223	14.74
C. Magma	26,271	20,217	23.04
C. Light	39,874	31,538	20.90
C. Seed	12,668	8,895	29.78
Melt Liquor	1,100	739	32.81
Clear Liquor	638	347	45.55

Particulars	Average Results of Color IU		
	2006-07 to 2007-08	2012-13 to 2013-14	% of Color Decrease

Sulphited Liquor	338	Process Changed	

Phosfloatation Out Liquor	Process N/A	226	
D/Filter + GAC out Liquor	Process N/A	183	
Fine Liquor	287	175	39.23
R1 Massecuite	460	281	38.85
Run Off 1st	881	543	38.40
R-1 sugar	39	20	49.04
R-2 Massecuite	1,077	662	38.53
Run Off 2nd	2,094	1,241	40.72
R-2 Sugar	61	35	42.30
R-3 Massecuite	2,483	1,474	40.64
Run Off 3rd	4,872	2,735	43.86
R-3 sugar	94	52	45.16

Conclusion

Current knowledge on origin of colors, their development during processing are shared.

It is clear that the topic is a complex one, Color formation of various types occurs throughout sugar processing.

color analyses was carried out through out the seasons, and data compared for 4 seasons

Its evidently showing that with the continuous improvement in milling , processing and cane quality and trying different color removal techniques, improvement in colors brought and found that existing process is better one according to MSM scenario.

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