PLANTATION OF A NEW FORMULA TO ASSESS PURITY OF FINAL MOLASSES

***Hayat-ur-Rahim Khan**
hayatsalarzai99@yahoo.com
hayat.salaarzai@gmail.com
&
Muhammad Shafiq Khan**
engr.shafiqrao@gmail.com

Abstract:

Loss of sucrose in final molasses is a major financial loss to a sugar factory; all efforts are to be made to curtail this loss. The apparent purity concept is unable to quantify the degree of molasses exhaustion but the Target Purity Formula can be used as bench mark to fulfill this purpose. Target Purity formula is a function of mono saccharides and inorganic components. A number of Target Purity Formulas have been presented in the past. Thirty tests of analysis of molasses exhaustion were collected from various sugar mills of the country in three years. All available Target Purity Formula applied on these tests to find an appropriate formula which can be used to quantify the exhaustion of final molasses for our country. Evaluating some of the Target Purity Formulas, a new formula is being planted **(40.86-14.60 log Rs/A)** and checked against all thirty tests analysis of final molasses exhaustion and found suitable for our country.

Introduction:

The loss of sucrose in final molasses is the biggest loss which has to suffer by a Sugar Factory. This loss has a significant impact on the profitability of a Sugar Mills directly or indirectly. It is essential to obtain a reliable data on final molasses exhaustion to minimize sucrose loss. The normal measurement or apparent purity obtained in Sugar Mills Laboratory is not accurate enough to check sugar loss in final molasses. To assess the degree of exhaustion achievable, a **"Bench Mark"** is required. Which is luckily available in the form of **"Target Purity"** formula.

Target purity is the lowest final molasses purity achievable in a Sugar factory. It reflects the dependence of optimum final molasses exhaustion on the ratio of two main factors Reducing Sugar (RS) and Ash (A) ratio. Presence of reducing sugar (mono saccharides) or Fructose + Glucose in the juice has an ability to decrease the solubility of sucrose in the molasses while inorganic components (Ash) tend to increase solubility.

***Process Advisor, Sanghar Sugar Mills Ltd. Sanghar. Sindh.**

**** Chief Chemist, Sanghar Sugar Mills Ltd. Sanghar .Sindh.**

RS/A ratio are used as the criterion for molasses exhaustion. It is the only factor that effects the purity of final molasses during the processing of juices and mother liquor.

The aim of this study is to find out appropriate target purity equation that can be used to quantify molasses exhaustion in our country because apparent purity concept is considered insufficient to quantify the degree of molasses exhaustion.

Over the years, a number of different Target Purity Formulas have been presented for different cane growing areas of the world. So, it has become a necessity to choose a most appropriate equation for our country also.

Target purity equations used in different parts of the world are given as under:-

Foster (1960)		$40.7 – 17.8. \log (q_{RS/A})$
Miller et al. (1998)	(No. 1)	$41.1 – 8.7. \log (q_{RS/A})$
Miller et al. (1998)	(No. 2)	$46.9 – 9.5. [1 – \exp (- 1.3 . q_{MS/A})]$
Miller et al. (1998)	(No. 3)	$39.4 – 10.6. \log (q_{RS/A})$
Miller et al. (1998)	(No. 4)	$55.1 – 18.7. [1 – \exp (- 2.6 . q_{MS/A})]$
Bruijn et al. (1972)		$39.9 – 19.6. \log (q_{RS/A})$
Rein and Smith (1981)	(No.1)	$37.7 – 17.6. \log (q_{RS/A})$
Rein and Smith (1981)	(No. 2)	$33.9 – 13.4. \log (q_{MS/A})$
Smith (1995)		$43.1 – 17.5. [1 – \exp (-0.74 . q_{MS/A})]$
Saska et al. (1999)		$42.4 – 12.3. \log (q_{MS/A})$
Gil et al. (2001)	(No. 1)	$38.5 – 7.7. \text{In} (q_{RS/A})$
Gil et al. (2001)	(No. 2)	$35.8 – 6.3. \text{In} (q_{RS/A})$

Where

RS/A = Reducing Sugar Ash Ratio
MS/A = Mono Saccharide Ash Ratio

Review of Literature:

1. The principal loss of sucrose in the boiling house is that retained in the final molasses. As sucrose crystallization from final molasses proceeds, the rate of crystallization becomes slower and slower until finally a point is reached at which no more sucrose will crystallize out at a given temperature. The molasses then is termed exhausted. The purity of exhausted molasses depends principally on water content. At the same water content, however, the nature and quantity of non sucrose constituents, having their origin in cane, govern the purity; of these reducing sugars and ash have major controlling effect (Payne, 1982).
2. Hugot (1986) gives a simple formula of expected purity of final molasses used in the Reunion i.e. expected purity = 40-4 x $\frac{\text{Reducing sugars.}}{\text{Ash}}$
3. Many operating factors, such as the character of the crystals and density of the final massecuites, the extent of the cooling in the crystallizers, and the after treatment of massecuite, effect the purity of final molasses. Viscosity is also of great importance in exhaustion of molasses, since it is one of the factors that limit the concentration of the massecuite and the super saturations of the molasses (Meade,1964).

4. Most of the sugar factories estimate their performance by the apparent purity of final molasses. However, this method of comparison in the same factory is doubtful value. With the progress of the crop and maturing of the cane the relation of sucrose to reducing sugars changes and therefore direct polarization will change and consequently apparent purity. Since in a given sugar factory the ash content in molasses varies very little, it can be disregarded for all practical purposes, and only the sucrose reducing sugars and Brix of molasses can be taken into consideration for calculation of maximum possible exhaustion of molasses (Baikow, 1967)

Target Purity Formula Calculation .

Material and Method:

In order to calculate Target Purity formula following steps were adopted in SSML laboratory:-

Apparent Brix of Molasses	:	Spindle Used
Apparent Pol of Molasses	:	Polarimeter AA10
Apparent Purity of Molasses	:	Pol x 100 / Bx

a) **Determination of Sulphated Ash in Final Molasses:**

1. Heat a platinum dish to redness in the furnace.
2. Transfer to a desiccators and allow cooling.
3. Weigh the dish on analytical balance.
4. Transfer 3-5 g of molasses to the dish and weigh.
5. Add 5 ml of 10 percent sulphuric acid.
6. Evaporate on a steam bath for 2-3 hours and then heat on hot plate until completely charred. Start hot plate on low heat and gradually increase to high heat to prevent spattering of sample.
7. Transfer to the furnace and heat at 550°C for five hours.
8. Remove from furnace; cool and carefully add 3 ml of 10 percent sulphuric acid wetting the entire dish.
9. Evaporate almost to dryness on steam bath and then heat on hot plate until sulphuric acid is volatilized.
10. Transfer to furnace again and heat at 500°C for two hours.
11. Remove from furnace, cool in a desiccator and weigh.

Calculation and Example:

Weight of empty platinum dish			32.582 g
Weight of dish plus molasses			37.332 g
Weight of molasses sample			4.750 g
Weight of dish plus ash after ignition			33.533 g
Weight of ash 33.533-32.582			0.951 g
Sulphated Ash %	=	$\frac{0.951 \times 100}{4.750}$	= 20.02

b) **True Solids** : Brix Molasses –(0.74 x Sulphated Ash)

c) **True Purity** : Sucrose x 100 / True Solids

d) **Reducing Sugar (Eynon & Lane Method)**

1. Take 5 ml A-Fehling Solution + 5 ml B-Fehling Solution in a Conical Flask.
2. Add 30 ml Distilled Water
3. Add 2-3 drops of methylenblau as indicator and mix well.
4. Heat on hot plate and titrate against Final Molasses and note the burette reading.
5. Calculation R.S. % $= \frac{0.05 \times 10 \times 100}{\text{Burette Reading}}$

e) **Total Sugar as invert in Final Molasses:**

1. Wt. 0.75 gm of molasses sample in a 100 ml beaker on analytical balance.
2. Add 20-30 ml of Distilled Water and mix well.
3. Transfer into a 200 ml flask and fill ½ with distilled water.
4. Cool on room temperature for ½ hours.
5. Add 4-5 drops of P.P. as indicator.
6. Add 10 % NAOH solution until pink color appears.
7. Complete the volume with distilled water.
8. Take 5 ml A-Fehling solution + 5 ml B-Fehling solution into a conical flask add 15-20 ml distilled water and 2-3 drops of methylenblau.
9. Titrate against final molasses and note the burette reading.

Calculation and Example:

Table

Burette Reading	Value
20.0	50.9
21.0	51.00
22.0	51.10
23.0	51.10
24.0	51.20
25.0	51.20
26.0	51.30
27.0	51.40

B. Reading = 26

Total Sugar = $\frac{51.30 \times 200}{10 \times 26 \times .75}$ = 52.62

Sucrose % = Total Sugar as invert – R.S. X 0.95

Choice of Target Purity Formula:

Thirty exhaustion molasses tests were collected from various sugar mills all over the country in three years during season 2012-13, 2013-14 & 2014-2015 calculated according to twelve Target Purity Equations.

Results are shown in Table No. 1, 2, & 3.

Table No. 1 **Season 2012-13**

Factory No.	Redu-cing. Sugar	Sulpha-ted Ash	RS/A Ratio	True Solids	Sucrose	True Pty.	Foster 1960	1. Miller 1998	2. Miller 1998	3. Miller 1998	4. Miller 1998	Brujin 1972	1. Rein & Smith 1981	2. Rein & Smith 1981	Smith 1995	Saska 1999	1. Gil et al 2001	2. Gil et al 2001
							Target Pty.	Target Pty.	Target Pty.	Target Pty.	Target Pty.	Target Pty.	Target Pty.	Target Pty.	Target Pty.	Target Pty.	Target Pty.	Target Pty.
1	14.76	14.96	0.99	85.43	31.43	36.79	40.80	41.15	40.03	39.46	37.84	40.01	37.80	33.98	34.03	42.47	38.60	**35.88**
2	13.77	15.00	0.92	88.24	31.75	35.98	41.36	41.42	40.28	39.79	38.12	40.63	38.35	34.40	34.47	42.86	39.16	**36.34**
3	11.90	18.23	0.65	77.30	34.29	44.36	**44.00**	42.71	41.47	41.36	39.83	43.53	40.96	36.38	36.40	**44.68**	41.78	38.49
4	14.40	18.27	0.79	85.54	36.67	42.87	**42.54**	**42.00**	40.81	40.50	38.81	**41.93**	39.52	35.29	35.37	43.67	40.33	37.30
5	13.78	16.79	0.82	78.17	32.03	40.97	42.23	41.85	**40.67**	**40.31**	38.61	41.58	39.21	35.05	35.13	43.46	**40.02**	37.04
6	12.01	18.40	0.65	81.28	35.14	43.23	44.00	42.71	41.47	41.36	39.83	**43.53**	40.96	36.38	36.40	44.68	41.78	38.49
7	14.12	16.90	0.84	80.09	36.40	45.45	42.09	41.78	40.61	40.23	38.53	41.43	39.07	34.95	35.03	43.36	39.88	36.93
8	13.03	17.18	0.76	85.18	36.40	42.73	**42.84**	**42.14**	40.94	40.67	39.00	**42.25**	39.81	35.51	35.58	43.88	40.63	37.54
9	12.09	17.45	0.69	87.48	37.77	43.18	**43.54**	42.49	41.26	41.09	39.49	**43.02**	40.50	36.04	36.08	44.36	41.33	38.11
10	12.20	16.33	0.75	86.41	37.96	43.93	42.95	42.20	41.00	40.74	39.08	42.38	39.93	35.60	35.67	**43.96**	40.75	37.64
11	12.87	17.66	0.73	86.03	37.31	43.37	**43.15**	42.30	41.08	40.86	39.21	42.59	40.12	35.74	35.81	44.09	40.94	37.79
12	11.96	18.07	0.66	87.45	37.70	43.11	**43.89**	42.66	41.42	41.30	39.75	**43.41**	40.85	36.30	36.32	44.60	41.68	38.40
13	15.13	15.60	0.97	86.08	37.57	43.65	40.94	41.22	40.09	39.54	37.90	40.16	37.93	34.08	34.14	42.56	38.74	35.99
14	14.67	16.60	0.88	84.79	33.51	39.52	41.66	41.57	40.41	**39.97**	38.28	40.95	**38.64**	34.62	34.70	43.06	**39.45**	36.58
15	15.10	14.60	1.03	86.49	32.17	37.20	40.44	40.97	39.88	39.24	**37.67**	39.61	37.44	33.70	33.74	42.22	38.24	35.59

Table No. 2 **Season 2013-14**

Factory No.	Redu-cing. Sugar	Sulpha-ted Ash	RS/A Ratio	True Solids	Sucrose	True Pty.	Foster 1960	1. Miller 1998	2. Miller 1998	3. Miller 1998	4. Miller 1998	Brujin 1972	1. Rein & Smith 1981	2. Rein & Smith 1981	Smith 1995	Saska 1999	1. Gil et al 2001	2. Gil et al 2001
							Target Pty.	Target Pty.	Target Pty.	Target Pty.	Target Pty.	Target Pty.	Target Pty.	Target Pty.	Target Pty.	Target Pty.	Target Pty.	Target Pty.
1	12.78	18.00	0.71	80.10	34.80	43.45	**43.35**	42.39	41.17	40.98	39.35	**42.82**	40.32	35.89	35.95	**44.23**	41.14	37.96
2	14.98	16.70	0.90	85.20	35.40	41.55	**41.54**	**41.51**	40.36	39.90	38.22	**40.83**	38.53	34.53	34.61	42.98	39.34	36.48
3	13.05	17.01	0.77	85.10	36.30	42.66	**42.75**	**42.10**	40.90	40.62	38.94	**42.16**	39.73	35.44	35.52	43.82	40.54	37.47
4	14.41	18.00	0.80	85.54	36.40	42.55	**42.42**	**41.94**	40.76	40.42	38.73	**41.79**	39.40	35.19	35.28	**43.59**	40.21	37.20
5	13.78	16.10	0.86	88.00	32.00	36.36	41.90	41.69	40.52	40.12	38.42	41.22	38.89	34.81	34.89	43.23	39.70	**36.78**
6	12.01	18.05	0.67	87.45	37.91	43.35	**43.85**	42.64	41.40	41.28	39.72	**43.37**	40.81	36.27	36.30	44.58	41.64	38.37
7	14.30	18.98	0.75	84.80	35.60	41.98	42.89	**42.17**	40.97	40.70	39.04	**42.31**	39.86	35.55	35.62	43.91	40.68	37.58
8	14.12	17.05	0.83	80.10	36.45	45.51	42.16	41.81	40.64	40.27	38.57	41.51	39.14	35.00	35.08	43.41	39.95	36.99

Table No 3 **Season 2014-2015**

Factory No.	Redu-cing. Sugar	Sulpha-ted Ash	RS/A Ratio	True Solids	Sucrose	True Pty.	Foster 1960	1. Miller 1998	2. Miller 1998	3. Miller 1998	4. Miller 1998	Brujin 1972	1. Rein & Smith 1981	2. Rein & Smith 1981	Smith 1995	Saska 1999	1. Gil et al 2001	2. Gil et al 2001
							Target Pty.	Target Pty.	Target Pty.	Target Pty.	Target Pty.	Target Pty.	Target Pty.	Target Pty.	Target Pty.	Target Pty.	Target Pty.	Target Pty.
1	15.09	15.80	0.96	85.27	35.42	41.54	41.06	**41.27**	40.14	39.61	37.96	40.29	38.05	34.17	34.23	42.65	38.85	36.09
2	15.04	15.40	0.98	86.13	34.82	40.43	**40.88**	41.19	**40.07**	39.51	37.88	**40.10**	37.88	34.04	34.10	42.53	38.68	35.95
3	14.30	19.74	0.72	84.79	35.49	41.86	43.19	42.32	**41.10**	40.88	39.24	42.64	40.16	35.78	35.84	44.12	40.98	37.83
4	12.74	18.21	0.70	79.35	34.92	44.01	**43.46**	42.45	41.23	41.04	39.43	42.94	40.43	35.98	36.03	**44.31**	41.25	38.05
5	14.90	16.70	0.89	84.80	33.50	39.50	41.58	41.53	40.38	39.93	38.24	**40.87**	38.57	34.56	34.64	43.01	**39.38**	36.52
6	12.30	16.40	0.75	86.00	38.00	44.19	42.92	42.19	40.98	40.72	39.06	42.35	39.90	35.57	35.65	43.94	40.72	37.61
7	13.05	17.00	0.77	85.09	36.30	42.66	**42.74**	**42.10**	40.90	40.62	38.94	**42.15**	39.72	35.44	35.52	43.81	40.54	37.47

Plantation of a New Formula:

It is obvious from Table No. 1, 2 &3 that results of Target Purity and True Purity Difference (TPD) of following four equations are very marginal as compared to other eight equations.

1.	Foster	40.7-17.8 log RS/A	Near or Equal	16	Results
2.	Miller	41.1-8.7 log RS/A	Near or Equal	14	Results
3.	Bruijin	39.9-19.6 log RS/A	Near or Equal	20	Results
4.	Saska	42.4-12.3 log RS/A	Near or Equal	10	Results

If all these four equations are evaluated in one equation, a new formula is formed: - **40.86 – 14.60 log RS/A**

Discussion:

Now applying this new formula at all thirty molasses exhaustion tests following results are obtained.

Table No. 3

Factory No.	RS/A Ratio	True Purity	New Equation	Final Molasses TPD
			Target Purity	TPD=True Purity-Target Purity
1	0.99	36.79	40.95	-4.16
2	0.92	35.98	41.41	-5.43
3	0.65	44.36	43.57	0.79
4	0.79	42.87	42.38	0.49
5	0.82	40.97	42.12	-1.14
6	0.65	43.23	43.57	-0.34
7	0.84	45.45	42.01	3.44
8	0.76	42.73	42.62	0.11
9	0.69	43.18	43.19	-0.02
10	0.75	43.93	42.72	1.21
11	0.73	43.37	42.87	0.50
12	0.66	43.11	43.48	-0.37
13	0.97	43.65	41.06	2.58
14	0.88	39.52	41.65	-2.13
15	1.03	37.20	40.65	-3.46
16	0.96	41.54	41.16	0.38
17	0.98	40.43	41.02	-0.59
18	0.72	41.86	42.91	-1.05
19	0.70	44.01	43.13	0.88
20	0.71	43.45	43.04	0.41
21	0.90	41.55	41.56	-0.01
22	0.77	42.66	42.55	0.11
23	0.80	42.55	42.28	0.28
24	0.86	36.36	41.85	-5.49
25	0.67	43.35	43.45	-0.10
26	0.75	41.98	42.66	-0.68
27	0.83	45.51	42.06	3.44
28	0.89	39.50	41.59	-2.09
29	0.75	44.19	42.69	1.49
30	0.77	42.66	42.54	0.12

Results:

Minus sign of TPD shown in Table No. 3, that True Purity of final molasses is less than Target Purity which is an indication of good performance. In case of plus sign, effort should be made to reduce actual purity of final molasses.

Taking into consideration the existing condition of exhaustion of final molasses in our country, the new target purity formula gave **best fit** results to quantify molasses exhaustion.

Parameters to control final molasses purity

To achieve lowest final molasses purity following important points should be kept in mind.

a) Maximum removal of impurities from mixed juice.
b) Addition of minimum quantities of non sugars in juice as lime salts.
c) Prevention of inversion of sucrose, distraction of reducing sugars and deterioration of non sugar components.
d) C graining should be done at 68-70^0 purity with full exhausted hard grain. Dropping Purity of C Strike should range between 47-49^0 with maximum achievable brix 100-104$^{o.}$
e) Cooling of C-massecuite up to 40-44^{o}C and then reheating up to 50-52^{o}C gives better results.
f) As a rule of thumb feeding molasses Brix (A. heavy. B. heavy. C. light)should maintain at 75^0 with 75^{o}C temperature in C.Massecuite.
g) Four massecuite boiling (A.A1.B.C)scheme is suitable to control final Molasses purity.

Acknowledgement:

Grateful acknowledgement is extended to Mr. Muhammad Hashim Rajar, Dy. Managing Director and General Manager for his support and keen interest to make this paper a reality.

References:

1. Hubert Olbrich. 1963,The Molasses, Berlin, Germany.
2. Khan Hayat-ur-Rahim. 2000. Fabrication Practices of Boiling House of a sugar Factory**.** Karachi**.** Pakistan. 62-63
3. Naqvi, H.A. 2013. Studies in Cane Sugar Industries. Fine Book Printers, Lahore, Pakistan page.177-179
4. Rein. P.W. 2007. Cane Sugar Engineering. Verlag Dr. Albert Bartens KG, Berlin, Germany page 455-465.
5. Tahir A.A. Assessment of Hidden Sugar Losses in Cane Sugar Factory, 47th Annual Convention of PSST 2013, Rawalpindi.