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SIGNIFICANCE OF BRIX READING IN DETERMINATION OF QUALITY OF ORAL SYRUP AND SEMISOLID FORMULATIONS

Rajashree Rane[1*], Divya Hattangadi[2], Prajakta Jadhav[3], Sachin Kundalwal[4], Chetna Chotalia[5], Ashish Suthar[6]

[1*]Group Leader, [2]Junior Research Scientist, [3]Research Associate, [4]Senior Research Scientist, [5]Deputy General Manager, [6]General Manager

Phytomedicine Division, Piramal Phytocare Ltd., 1, Nirlon Complex, Off Western Express Highway, Goregaon (E), Mumbai 400063, Maharashtra, India.

***Correspondence for Author: Rajashree Rane**

Group Leader, Phytomedicine Division, Piramal Phytocare Ltd., 1, Nirlon Complex, Off Western Express Highway, Goregaon (E), Mumbai 400063, Maharashtra, India.

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ABSTRACT

The study is aimed to determine the application of Brix Reading to control the quality of oral herbal syrup and semisolid formulations, by focusing on other relevant parameters such as TS (Total solids), weight per ml in syrup and LOD (Loss On Drying) at 105^0C in semisolid formulations. Also tried to show the interrelations of these parameters by analyzing few lots of ten different brands of herbal syrup formulations and semisolid formulations such as three types of Citrospreads and four different flavored jam samples. In sugar base syrup formulations brix reading was found in the range of 50 degree to 70 degree brix. Which was observed to be almost matching with total solids in %w/w of corresponding sample and in sugar-free syrup sample, it was found to be around 40 in one set and around 55 degree brix in rectified set, as in the formula sugar is replaced by sucralose and 70% sorbitol. Quantity of xanthan gum is adjusted to maintain the density of the syrup. In semisolid formulations like citrospreads and jam samples the sum total of degree brix and Loss on Drying (LOD) at 105^0C was almost 100. This way one can make use of these parameters as quality determining factors to maintain the batch to batch consistency of different liquid and semisolid formulations.

KEYWORDS: Brix reading, Total Solids, Weight per ml, LOD.

INTRODUCTION

Drugs or nutraceutical formulations exist in different forms. Various formulations are available as per the compositions. It is very important to check the efficacy and bioavailability of the drug for getting better medicinal effect. This can be achieved by estimating the analytical marker or biomarker present in the formulation. However, it is equally important to maintain the physical status of the formulations like syrup and semisolid forms by evaluating batch to batch consistency of these samples. Sample of desired consistency will assure the palatability and will indirectly support the assigned shelf life.

Professor A. F. W. Brix was a 19th Century German chemist (b.1798, d.1890). He was the first to measure the density of plant juices by floating a hydrometer in them. BRIX is a measure of the percent solids (TSS) in a given weight of plant juice---nothing more---and nothing less[1].

Brix is often expressed another way: Brix equals the percentage of sucrose. For, indeed, the brix is actually a summation of the pounds of sucrose, fructose, vitamins, minerals, amino acids, proteins, hormones, and other solids in one hundred pounds of any particular plant juice.

BRIX varies directly with plant QUALITY. For instance, a poor, sour tasting grape from worn out land can test 8 or less brix. On the other hand, a full flavored, delicious grape, grown on rich, fertile soil can test 24 or better brix.

In syrup formulations, specific gravity or weight per ml of the liquid as well as total solids and degree brix determine the density or thickness of the syrup which in turn decides the consistency of the product.

Degree brix is the ratio of total soluble solids to water in solution. Higher brix means[2] higher nutrient density (assumption), better taste (widely acknowledged), resistance to rotting, resistance to disease, resistance to frost, that is higher quality.

Dissolved solids affect both density and refractive index or brix reading of a liquid. Thus when the concentration of sugar in water is increased, the density and refractive

index or brix reading increases. In this way density and brix reading can be used to determine solute concentration in a binary system.

Food products can contain many types of sugar and other soluble components such as acids, salts, color and flavor agents. However, the Brix scale is used regardless of the composition of the aqueous phase. Strictly speaking, for non-sucrose based products, measurements should perhaps be expressed as 'apparent Brix'[3] or 'sucrose equivalents'.

The fruit jam will have a definite amount of total soluble solids. Soluble solids can be determined by means of brix hydrometer, which measures the specific gravity. Degree brix can be recorded on Abbe's refractometer. Degree brix is a measure of soluble solids only in the case of pure sucrose solution. Generally fruit juice contain more sugar than any other soluble constituents. Hence degree brix provides useful guide of soluble solids or sugar content. Soluble solids other than sucrose do not affect specific gravity. Sahni et al[4] have reported TSS in pineapple, apple, plum, orange and mango fruit as 9.00, 12.00, 16.00, 10.00 and 20.00 respectively. It is quite evident that for a fruit juice or pulp which has higher percentage of moisture, the brix reading will be lower. The brix reading indirectly indicates TSS as well as amount of sugar or the sweetness of final product. In order to obtain the correlation between percentage moisture content in the form of loss on drying and degree brix, a statistical treatment was given and the regression equation was obtained as ^{0}Brix = 80.2 – 0.342 LOD with correlation coefficient (-0.559). 31.3% variation in moisture content is explained by degree brix.

Various categories of sugar based herbal syrups, sugar free herbal syrup samples were prepared as per the specified formulae and subjected to analysis. Also semisolid formulations like Citrospread and jams with different flavours were taken for the study.

MATERIALS AND METHODS

Weight per ml, brix reading, and total solids in % w/v were practically estimated in all syrup formulations under study, by applying stated pharmacopoeial methods. Similarly, semisolid formuations like citrospreads and jam samples were analysed for Loss On Drying at 105^{0}C (LOD) and brix reading.

Samples: Three lots of total nine different types of sugar based samples namely Appetite stimulant syrup, Bronchodilator cough syrup, Anti-stress syrup, Anti-pyretic syrup, Immunomodulator syrup, Memory enhancing syrup, Hepatoprotective, syrup, Anti-allergic cough syrup and Anti spasmodic cough syrup along with three lots of two different categories of sugar free cough syrup samples were chosen for this study. Amongst semisolid fomulations three types of Citrospread samples and four different flavored jam samples like pineapple, mango, strawberry and mixed fruit jams were subjected to quantitative evaluation.

Chemicals and Reagents: All the chemicals and reagents used in different processes were procured from M/s Merck India and M/s Qualigens. Reagents were standardized as per the standard pharmacopoeia.

Equipments and Instruments: All the glassware used were well calibrated and were procured from M/s Borosil Glass Works Ltd. Instruments used were weighing balance (M/s Schimadzu Corporation), Electric oven and Water bath (M/s Pathak Electrical Works Ltd.).

The most important, easy to operate instrument used here was "Abbe's Refractometer from M/s Rajdhani.

METHODS

Weight per ml

A thoroughly cleaned and dried calibrated[5] pycnometer was filled with freshly boiled and cooled water at 25°C and its content was weighed. (The density of water is 0.99602 g at 25°C). The capacity of pycnometer (normally it is 25 ml) was calculated. Gently the pycnometer was filled with homogenous syrup sample, allowed to attain the temperature (25°C) and it was weighed with the content. The process was repeated three times and the average value was recorded. The weight/ml was calculated by applying the formula as,

Weight / ml (g/ml) = (B – A) / wt/ml factor of pycnometer.

Where, A:- Weight of the pycnometer in g

B:- Weight of the pycnometer + syrup sample in g

Total Solids

5ml of syrup sample was accurately pipetted out from the homogenous syrup sample, with the help of previously calibrated pipette, in a dry previously weighed petridish and it was evaporated on a water bath at 100°C temperature for about half an hour. Sample in the petridish was dried in an oven at 105^{0}C temperature for about 6 to 7 hours. Then it was allowed to attain the room temperature in a desiccator and weighed till constant weight.

"Total Solids" in Percentage weight /volume was calculated by applying the following formula:

Total Solids %w/v = (C – A) X 100 / (B)
Where, A :- weight of empty petridish in g.
B :- Aliquot of sample pipetted out in ml
C :- Weight of petridish + syrup sample (After drying)

Loss On Drying at 105^{0}C

Semisolid form of citrospread and jam samples were thoroughly mixed. About 2 to 3g homogenous uniform sample was accurately weighed in a previously cleaned and dried loss on drying (LOD) bottle, The sample was allowed to dry in an oven at 105^{0}C for about 3 to 4 hours, till the constant weight. The weight at every step was recorded, and loss on drying was calculated by applying the formula as,

Loss On Drying at 105°C %w/w) = (B – C) X 100 / (B – A)
Where,
A:- Weight of empty LOD bottle in g
B:- Weight of LOD bottle with sample before drying in g
C :- Weight of LOD bottle with sample after drying to constant weight in g.

Brix Reading

A drop of the homogenous syrup / semisolid sample formulation was gently loaded on the prism of the previously calibrated[6] suitable refractometer. The reading on the brix scale was recorded at ambient temperature as mentioned in the pharmacopoeia by appropriately adjusting the light rays with the help of the mirror situated at the bottom floor. The process was repeated for minimum three times and the average reading was recorded as the final "brix reading."

Application of "Brix reading" and its relation with total soluble solids is explained thoroughly in fruit juices[7], jams, jellies and so on.

RESULTS AND DISCUSSION

Weight per ml, total solids in % w/v and brix reading values in all herbal syrup samples were practically evaluated and findings are recorded in Table 1. Also total solids in %w/w for each syrup sample was calculated by considering the weight per ml value of corresponding syrup sample.
Total solids in %w/v (By considering corresponding Brix reading) can be calculated as…
Total solid in %w/v = Brix Reading (B) x weight per ml (A)
Total solids in %w/w can be calculated by applying corresponding weight per ml as….
Total solids in %w/w = Total solids in %w/v (C) / weight per ml (A)
Where, (A) → Weight per ml in g/ml
(B) → Brix reading in degree brix
(C) → Total solids in %w/v

Table 1: Weight per ml, total solids and degree brix in oral herbal syrup formulations

Sr. No.	Product	Lot Number	Weight per ml	Brix reading (degree brix)	Total solids (%w/v)		Total solids (%w/w)	
	Syrup Formulations		(A)	(B)	Practical findings (C)	WRT Brix Rdg By calculation (B) x (A)	WRT weight per ml. by calculation (C) / (A)	Practical Brix reading
1	Appetite stimulant syrup	APR1	1.276	65.00	82.092	82.94	64.33	65.00
		APR2	1.274	64.00	81.092	81.54	63.65	64.00
		APR3	1.275	64.00	81.100	81.60	63.61	64.00
2	Anti-allergic cough syrup	BRO1	1.256	56.00	68.810	70.33	54.78	56.00
		BRO2	1.239	53.00	63.490	65.66	51.24	53.00
		BRO3	1.256	55.00	69.380	69.08	55.24	55.00
3A	Anti-allergic SF cough syrup - category 1	BRS1	1.121	38.00	33.240	42.59	29.65	38.00
		BRS2	1.125	40.00	38.570	45.00	34.28	40.00
		BRS3	1.123	40.00	34.250	44.92	30.50	40.00
3B	Anti-allergic SF - cough syrup (category 2)	BRSE1	1.204	48.00	54.550	57.79	45.31	48.00
		BRSE2	1.195	49.00	54.844	58.55	45.89	49.00
		BRSE3	1.190	48.50	56.64	57.71	47.59	48.50
4	Anti-stress syrup	DOR1	1.307	66.00	84.970	86.26	65.01	66.00
		DOR2	1.307	65.50	85.370	85.61	65.32	65.50
		DOR3	1.322	68.00	90.520	89.90	68.47	68.00
5	Anti- pyretic syrup	FEV1	1.274	60.00	78.000	76.44	61.22	60.00
		FEV2	1.270	59.00	72.650	74.93	57.20	59.00
		FEV3	1.264	58.00	70.670	73.31	55.90	58.00
6	Immunomodulator syrup	IMN1	1.231	55.00	67.718	67.70	55.01	55.00
		IMN2	1.237	54.00	64.984	66.79	52.53	54.00

		IMN3	1.243	54.00	68.550	67.12	55.15	54.00
7	Memory enhancing syrup	INT1	1.246	60.00	73.074	74.76	58.65	60.00
		INT2	1.217	56.00	67.016	68.15	55.07	56.00
		INT3	1.260	58.00	73.500	73.08	58.33	58.00
8	Phyleclip syrup	PHY1	1.254	60.00	72.876	75.24	58.11	60.00
		PHY2	1.232	57.00	66.370	70.22	53.87	57.00
		PHY3	1.253	57.00	74.952	71.42	59.82	57.00
9	Anti -allergic syrup	TUS1	1.243	56.00	68.662	69.61	55.24	56.00
		TUS2	1.257	57.00	70.840	71.65	56.36	57.00
		TUS3	1.248	57.00	70.710	71.14	56.66	57.00
10	Anti-spasmodic syrup	KHA1	1.262	61.00	74.860	76.98	59.32	61.00
		KHA2	1.267	60.00	73.610	76.02	58.09	60.00
		KHA3	1.218	52.00	60.642	63.33	49.79	52.00

Practical value of Total solids in %w/v can be converted to %w/w by applying weight per ml of corresponding syrup sample. From the recorded findings it is clearly seen that the syrup sample containing dissolved sugar, shows the maximum resemblances with corresponding brix reading value and the value of total solids in %w/w. However, in sugar free syrup samples with other artificial sweeteners, the results of brix reading and the value of total solids in %w/w vary considerably, which can be further adjusted by increasing the concentration of sweeteners and xanthan gum, as shown in the category 2.

Resemblances in the brix reading and total solids in %w/w in sugar based syrup formulation is well illustrated in figure 1.

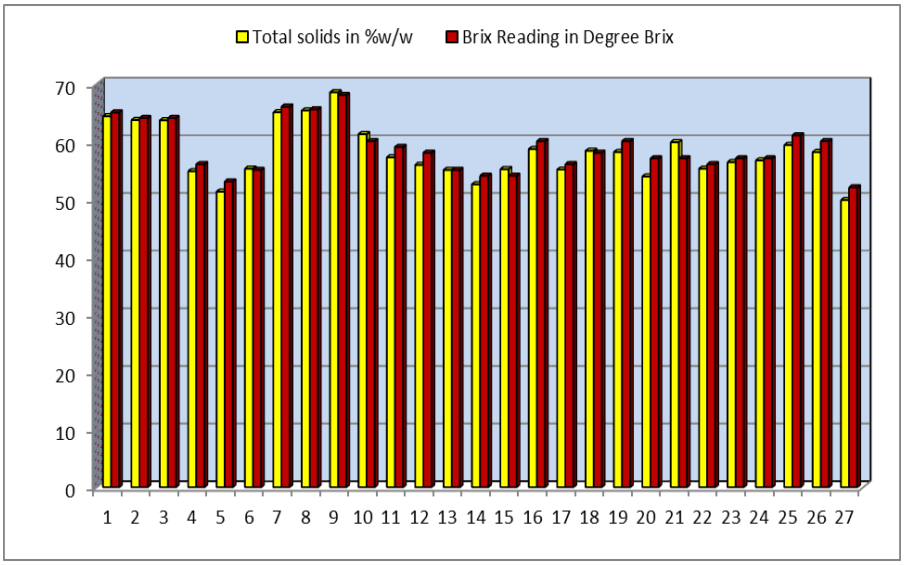

Fig.1: Brix reading and Total solids in %w/w in sugar based syrup formulations.

Difference in brix reading and total solids is observed in Anti allergic sugar free (SF) cough syrup where instead of sugar, honey, sucralose, 70% sorbitol and xanthan gum are added. Whereas, in second category of sugar free syrup sample, amount of 70% sorbitol and xanthan gum is increased to maintain the consistency of syrup. Effect of the same can be clearly seen in figure 2A and 2B respectively. Samples of category 2 depicted in Figure 2B have increased amount of xanthan gum compared to category 1 reflected in figure 2A, for adjusting the consistency equivalent to sugar base syrup samples.

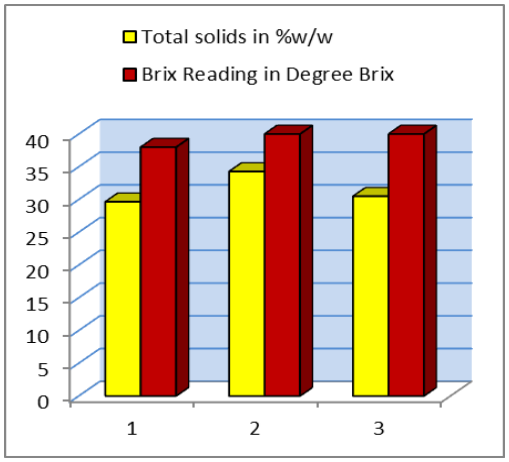

Fig. (2A)

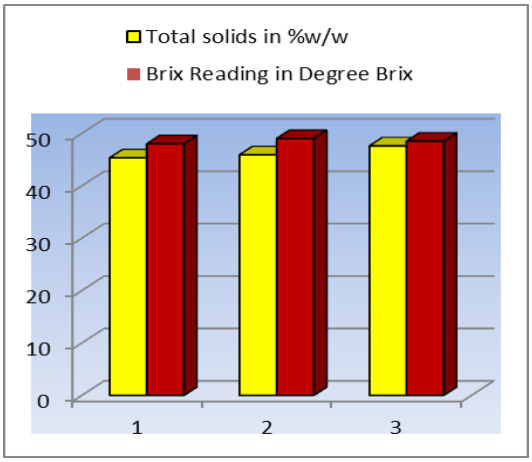

Fig. (2B)

This was observed from the data recorded in table 1, that total solids in sugar based syrups varies from 50%w/w to 70%w/w as per the formula and concentration of sugar in base preparation, Accordingly the value of brix reading also changes in corresponding syrup samples.

In case of Anti-allergic sugar free (SF) cough syrup, brix reading in category 1 is found to be about 40 degree, in which actual practical finding for total solids in %w/w is found to be around 30. This does not resemble to the theoritically expected value. However, in category 2, quantity of increased amount of 70% sorbitol and xanthan gum in the syrup formulation make the consistency equivalent to that of sugar based syrup. Hence brix reading is reached upto 50 degree, and practical finding of total solids seems to be aroud 55%w/w.

Table 2, shows the relationship between moisture content in the form of loss on drying (LOD) at 105^0C and average brix readings observed in different lots of semisolid sugar based formulations such as Citrospreads fortified with individual herb and different flavored jam samples.

Table 2: Loss on drying and brix reading in sugar based semisolid formulations.

Sr. No.	Product	Lot Number	Average Brix reading (degree brix)	Average value of Loss On drying (%w/w)	Average Residue on drying (%w/w) (100-LOD)
1	Citrospread (Balya)	CIT1	69.50	30.67	69.33
2	Citrospread (Hridya)	CIT2	71.00	29.77	70.23
3	Citrospread (Medhya)	CIT3	69.50	31.17	68.83
4	Jam sample (Pinapple Flavor)	JPAF	72.00	27.17	72.83
5	Jam sample (Mango Flavor)	JMAF	71.00	26.17	73.83
6	Jam sample (Strawberry Flavor)	JSTF	70.00	25.97	74.03
7	Jam sample (Mixed fruit Flavor)	JMIF	70.50	27.58	72.42

Average brix values are found in the range of 69 degree to 72 degree, which are inversely proportional to the moisture content in the form of loss on drying (LOD) at 105^0C. This is in conformity with the findings of Adsule[8], where he has given the inverse relations between moisture and total soluble solids. Brix value and moisture content in all semisolid formulations are depicted in the figure 3.

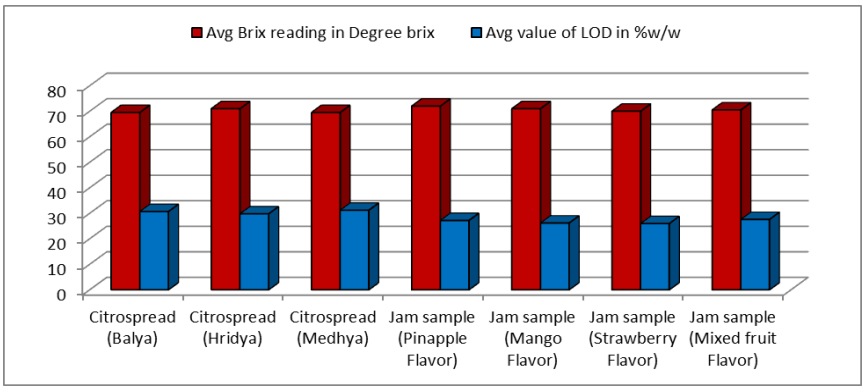

Fig. 3: Relationship of Average brix reading with LOD in semisolid formulations

Average value of residue on drying was calculated by deducting the corresponding practical value of loss on drying in %w/w from 100. Average brix reading and average residue on drying is found to be almost similar in respective samples. This is well illustrated in the figure 4.

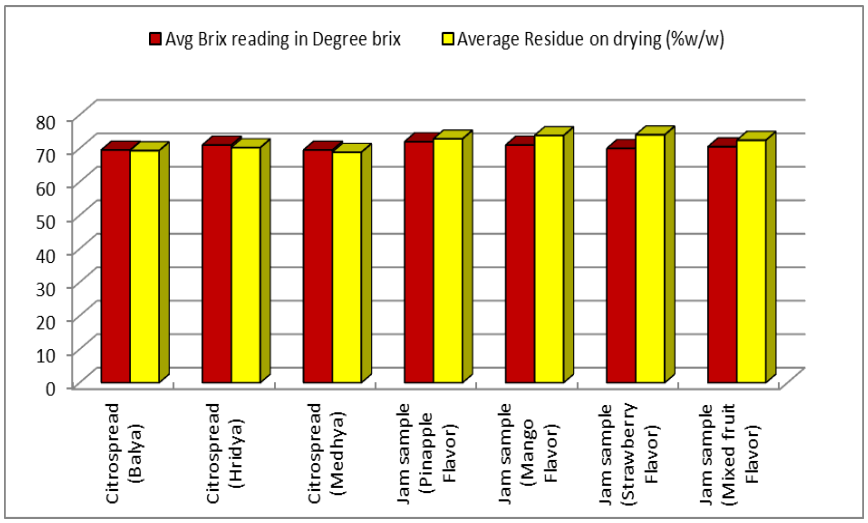

Fig. 4: Average brix reading and average residue on drying (%w/w) in semisolid formulations

Barry et al[9] used the TS meter, a temperature compensated hand refractometer of recent development for determination of total solids of human sera and other body fluids. A correlation was made between the refractometer reading for total serum solids and total serum protein.

The brix measurement can be read off the scale, showing levels of sugar, minerals, proteins and vitamins in the plant's juices, commonly referred to as dissolved solids. In conjunction with the presence of weeds and insects, brix levels can be used to help assess crop health.

David[10] explains that healthy plants, registering a high brix level, indicate superior resistance to pests and diseases, which ultimately means less time and expense need be spent on pest control measures.

CONCLUSION

In sugar based syrup formulations degree brix reading matches with total solids in %w/w, whereas in sugar free syrup formulations, it shows remarkable deviation with respect to the theoretical value. If we adjust the concentration of sweeteners and increase the concentration of excipients like sorbitol, xanthan gum, we can get the sample of same density as of sugar based syrup.

Moisture contents and Brix readings are inversely proportional in case of semisolid formulations, which again indicate the similarities between brix reading and residue on drying that is total solids in %w/w. Interdependencies of these parameters can be used for determining the texture, consistency and physical quality of syrup and semisolid formulations. It can add the value to quality norms.

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