Bellier Turbidity Temperature Test (BTTT) as a tool for assessing the purity of cottonseed oil available in markets of India

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Abstract:- In this study an attempt has been made to investigate the applicability of BTTT to cottonseed oils obtained from different parts of India and thereby examine the influence of geographical variations on BTTT. In the present work, the cottonseed oils used for analysis, such as refined cotton seed oil (Rct, Jagdish), refined cottonseed oil(Rct1,Sarthi),refined cottonseed oil (Rct2,Sidhbali) and cottonseed oil (Rct3,Narayan) exhibited BTT in the range of 19.6 to 20.4 ^{0}C. The result have demonstrated the reproducibility through the analyzed data. Hence It is observed that cottonseed oil fulfils BTTT values as per Regulation (Food Products and Standards) 2011 of Food Safety Standards and Act 2006.the standard mean error is in between 0.1-0.12 in case of BTT.

Keywords: Vegetable oil , cottonseed oil ,purity , BTTT.

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1. Introduction and objective

The total production of cottonseed oil (Washed) in India, i.e. both from undecorticated crushing and scientific processing, was estimated to be 851,000 tonnes during the period between November 2008 and October 2009 as compared to 934,000 tonnes between 2007 and 2008. It clearly revealed that cottonseed oil production was lagging behind the estimated potentiality of 1.17 million tonnes during 2008-09 as against 1.29 million tonnes during 2007-08, if all cottonseed available for processing was scientifically processed. Of course, this is due to huge amount of cottonseed is being crushed through traditional methods in India**[4]**.As per an estimate, about 9 million tonnes of cottonseed produced in India could yield approximately 1.5 million tonnes of oil **[3]**. The nutritional value of cottonseed oil is around 9 kcal/g, while the average digestibility is around 97%, which is comparable to that of the soybean, safflower and sunflower oil **[1]**.

As of 2008, oilseed was grown on 26.54 million hectares and produced 28.82 million tonnes, with an average productivity of 1086 kgs/ha. The net domestic availability of edible oil was only 7.2 million tonnes as against the actual consumption of 11.5 million tonnes and the gap of 4.3 million tonnes had to be imported, and this posed a great drain on the Indian foreign exchange. It was projected that by 2020 A.D, India would have to produce 66 million tonnes of oil seeds to meet the oil demand of 21.8 million tonnes. Otherwise, India has to helplessly depend on imports **[2]**.

1.1 Test for Presence of Cottonseed Oil (Halphen's Test): The development of red colour on heating the oil with a solution of sulphur in carbon disulphide indicates the presence of cottonseed oil. The test is also given by Hempseed oil, Kapokseed oil / oils and fats containing cyclopropenoid fatty acids (such as sterculic and malvalic acid). Hydrogenation and deodorization wholly or partially destroy the cromogens and react with diminished intensity. A positive reaction is not given by an oil heated to 250°C or above. The fat of animals fed on

cottonseed meal (butter, lard) or other cottonseed products may give faint positive reaction by this test. Take about 5 ml of the oil or melted fat in a test tube and add to it an equal volume of the sulphur solution (one percent (w/v) solution of sulphur in carbon disulphide and then add an equal volume of amyl alcohol). Mix thoroughly by shaking and heat gently on a water bath (70º to 80ºC) for a few minutes with occasional shaking until the carbon disulphide has boiled off and the sample stops foaming. Place the tube in an oil bath or a saturated brine-bath maintained at 110-115ºC and hold for 2.5 hours. A red colour at the end of this period indicates the presence of cottonseed oil. The test is sensitive to the extent of 0.5 % cottonseed oil in other oils .As per Food safety and standards (prohibition and restriction on sale) Regulations 2011, sale of certain admixtures prohibited. As per 2.1.1(5), a mixture of two or more edible oils as an edible oil ,a maximum tolerance of 10 red units in one cm cell on Lovibond tintometer scale is permitted when the oil is tested for halphen test without dilution. Halphen test is one of the qualitative identification test for cottonseed oil**[5].**

The quality of fats and oils is dictated by several physical such as texture, density, pacific gravity, colour, refractive index etc and chemical parameters such as acid value, iodine value, saponification value, unsaponifiable matter BTT etc are dependent on the source of oil; geographic, climatic, and agronomic variables of growth. Thus one must assess quantitatively the influence of these variables on characteristics of oils and fats; in present case on characteristics of cottonseed oil, **Bellier Turbidity Temperature Test** (BTTT) (acetic acid method), based on insolubility of Arachidic acid (1.13%) is used as a qualitative method for identification of pure cottonseed oil. Sometimes it is observed that cottonseed oil fulfils all specifications of refined oil but fails to pass BTTT and halphen test is positive. The addition of cottonseed oil in some edible oils such as cottonseed, soyabean, sunflower, cottonseed and olive oils are admixtures of edible oils, if halphen test is more than 15 red units on lovobond tintometer reading as per food safety rules 2006 hence BTTT values and other quality parameters are violating the Food safety and standard act 2006 with standard values of respective edible oils. In this present work, Purity of cottonseed oil can be conforming through BTT values of 19 to 21.0^{0}C as per the food product standards and food additives regulation 2011.
The Bellier figure or the temperature at which turbidity appears in a specified and neutralised oil sample under specified conditions was first proposed by Bellier and modified by several workers including Franz and Adler. According to Ever in 1912, the addition of sufficient acetic acid used instated of 1% hydrochloric acid succeeding modifications in the BTT. This had been adopted by several workers and gives satisfactory results for sufficient to judge the purity of respective edible oil and their admixture of oils **[10]**.

The objective of the present studies was to investigate the applicability of BTTT to cottonseed oils obtained from different parts of India and thereby examine the influence of geographical variations on Bellier Turbidity Temperature Test (BTTT) as a tool for assessing the purity of cottonseed oil available in markets of India. Assessing the quality and compared the assessed value with existing standards of BTTT for the respective oils as per Food safety and standards (food products and additives) Regulation 2011.

1.2 Literature review

There are varieties of cooking oil and fat available today and the claims made by them are, at best, confusing. On one side are the traditional ghee, cottonseed oil, coconut oil, and gingelly

oil. Then, there are the used-for-decades vanaspati and cottonseed oil, as well as the relatively newer kinds of vegetable oil ranging from cottonseed, sunflower, safflower, corn, canola, soybean, and palm to various blends. In particular, cottonseed oil performs better than other oil as it lasts a long time and stores well by withstanding higher temperature for food items due to its high antioxidant content. For instance, chips and snacks fried in cottonseed oil may maintain a longer shelf life. It is a good option for preparing healthier foods **[5]**.

The solubility of oils in various solvents is a constant, depending on the nature of the glycerides composing the oil. Fryer and Weston found that a mixture of equal volume of 92% ethyl alcohol and pure amyl alcohol used as a solvent for turbidity. In Valenta test, acetic acid was used as a solvent, the results are affected by the presence of moisture in the oil and free fatty acid which lower the turbidity temperature, increasing the solubility of the oils, which raises the turbidity temperature **[10]**.

The modified BTT test has been used by Ever for judging the purity of oils and has been found simple, rapid and fairly accurate for routine analysis as compared to the results obtained by Valenta test. Moreover, it can be conveniently used in the analysis of soap and commercial fatty acids and also for determining the % of two mixed oils. Others workers have also successfully used the same test for determining adulteration of cottonseed oil in some edible oils and also suggested its analytical importance. Besides the turbidity temperatures obtained with fatty acids by the method of fryer and Weston are different from those for the respective oils, depending on the difference in the solubility of the glycerides of the oil and its fatty acids in the same solvent **[7]**.

Table-1 Shows BTT standards/values for some edible vegetable oils under 2.2: Fats, oils and Fat emulsions as per FSSA 2006[8]

Sr.No	Item No	Vegetable oil	BTT limits
1	2.2.1.2	**Cotton seed oil**	**19.0 -21.0^{0}C**
2	**2.2.1.3**	Groundnut oil	39.0-41.0^{0}C
3	2.2.1.6	Rape seed oil /Mustard oil (toria oil)	23.0-27.5 ^{0}C
4	2.2.1.7	Rape seed oil or Mustard oil-Low erucic acid	Not more than 19.0^{0}C
5	2.2.1.8	Virgin olive oil	17.0^{0}C Max
		Refined olive oil	17.0^{0}C Max
6	2.2.1.10	Safflower seed oil (barrey ka tel)	Not more than 16.0^{0}C
7	2.2.1.12	Til oil (Gingelly/sesame oil)	Not more than 22.0^{0}C
8	2.2.1.13	Niger seed oil (sargiya ka tel)	25.0-29.0^{0}C
9	2.2.1.17	Almond oil	Not more than 60.0^{0}C

Source FSSA2006

1.3. Material and Experimental procedures

1.3.1 Materials

All the chemicals and reagents were analytical grade and used as received. Four cottonseed oils of different brands such as refined cotton seed oil (Rct, Jagdish), refined cottonseed oil(Rct1, Sarthi),refined cottonseed oil (Rct2, Sidhbali) and refined cottonseed oil (Rct3, Narayan) were gathered from super market of different places of India. All these oils were in different forms of packaging while some in poly packs (HDPE), others were in tetra packs, plastic cans and pet bottles of 1 litres and 5 litres. Since these four cottonseed oils were easily available for procurement. Most of the brands have mentioned nutritional values, green vegetarian logo, best before 6 months and 12months, free from argemone on their packs. These different cottonseed oils are used in the investigations on BTTT in this research study. These different cottonseed oils are used in the investigations on BTTT in this research study.

3.2 Experimental procedures

1.3.2.1 Determination of Bellier turbidity temperature acetic acid Method

Pipette out one ml of the filtered sample of oil in a flat-bottom 100 ml round flask, add 5ml of 1.5 N alcoholic potash heating over a boiling water bath using an air condenser After complete saponification cooling, neutralised by adding carefully dilute acetic acid and then add an extra amount of 0.4 ml of accurately measured dilute acetic acid using phenolphthalein indicator. Add 50 ml of 70% alcohol and mixed well. Heat and allow the flask to cool in air with frequent shaking. Note the temperature by using calibrated thermometer at which the first distinct turbidity appears which is the turbidity temperature. This turbidity temperature is confirmed by a little further cooling which results in deposition of the precipitate. Dissolve the precipitate by heating the contents to 50°C over water bath, again cool as desiccated above and make a triplicate determination of the turbidity temperature [6**,8,9]**.

Table 2: BTTT of different cottonseed oils with accuracy on BTT

Sr. No	Name of oil	Brand name	Code	BTTT*	SD	CV	SEM
1	Refined cottonseed oil	Jagdish	Rct	19.8	0.2	1.01	0.12
2	Refined cottonseed oil	Sarthi	Rct1	20.4	0.17	0.85	0.1
3	Refined cottonseed oil	Sidhbali	Rct2	19.6	0.17	0.87	0.1
4	Refined cottonseed oil	Narayan	Rct3	20.2	0.2	1.0	0.12

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* **Each value is averages of three measurements, SD-standard deviation, CV-coefficient of variance, SEM-Standard mean error**

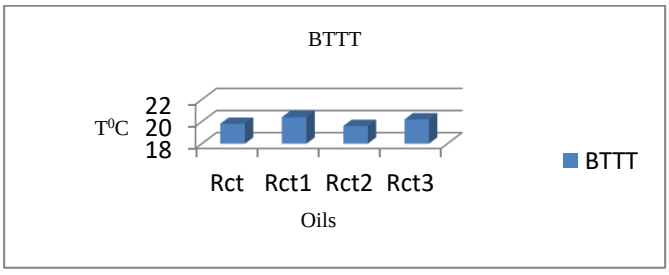

Fig.1 shows the BTTT values for different cottonseed oil

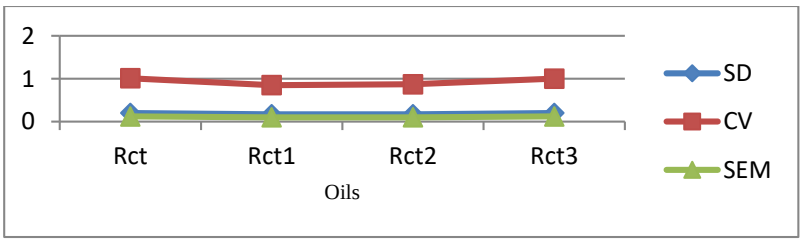

Fig.2 shows the statistical values for different cottonseed oil

1.4 Statistical analysis:

The data obtained from the experimental measurements and accuracy of BTTT for different brands of cottonseed oils have been analyzed and the Statistical parameter like standard deviation, coefficient of variance and standard mean error were calculated for both the parameters. All the experiment was carried out in triplicate and the results are presented as the mean SD, CV and SEM. Descriptive Statistics of different cottonseed varieties from different parts of India as shown in figure1 and 2.

1.5 Result and discussion

BTT values prescribed for the certain vegetable oils comes under the mandatory food laws in some countries but due to development towards hybridization in oil seeds, reconsideration in laws is required. **Table-1** Shows BTT standards/values for some edible vegetable oils under 2.2: Fats, oils and Fat emulsions as per FSSA 2006**[8]**.The results obtained for the BTTT and statistical accuracy for the cottonseed oils obtained from different places of India are shown in **Table2, Fig 1 and Fig 2**. The data obtained for Rct (19.8),Rct1(20.4),Rct2(19.6)and Rct3(20.2) are exhibited BTT in the range of 19.4 to 21°C.As all the reported BTTT values are average of three readings, the results have demonstrated the reproducibility of the analysis data. Thus the present investigations prove with due certainty the applicability of BTTT to all cottonseed oils. **Table 2** shows the accuracy, the standard deviation and coefficient is in the range of 0.17 -0.2 and 0.85-1.01.

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1.6 Future prospects

Wherever required, BTTT analysis Quantitative test should essential and can be easily supplemented with GC and HPLC analysis, which provide the quantitative data on presence of high molecular weight fatty acids in cottonseed oils. Hence BTTT depends on the presence of arachidic acid and other higher acids in cottonseed oil.

1.7 Conclusion

The BTTT method is cheaper, easier, requires little laboratory infrastructure and recognised as a convenient qualitative tool for identification of different variety of oils. In this study BTTT is applied on cottonseed oils and found that BTTT can be easily used as qualitative tool for identification of purity of cottonseed oil from different places of India. The present investigations prove with due certainty about applicability of BTTT to all cottonseed oils. This study also confirms prove reliability, reproducibility and diverse applicability of BTTT.

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