

# Identification of cottonseed oil available in markets of India using Bellier Turbidity Temperature Test (BTTT)

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**Abstract:-** 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 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, Samara), pure cottonseed oil(Pct, Tirupati), refined cottonseed oil (Rct1, Tirupati), cottonseed oil(Ct, Rani) and pure cottonseed oil(Pct1, Glick's) exhibited BTT in the range of 19.4 to 21.0 °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 0.06-0.12 in case of BTT.

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

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## 1. Introduction and objective

Cottonseed contains hull and kernel. The hull produces fibre and linters. The kernel contains oil, protein, carbohydrate and other constituent such as vitamins, minerals, lecithin, sterols etc. Cottonseed oil is extracted from cottonseed kernel. With three times as much unsaturated as saturated fatty acid, cottonseed oil is considered by health organisations to be a healthful vegetable oil acceptable for modern diets. Cottonseed oil does not have to be as fully hydrogenated for many purposes as some of the more polyunsaturated oils. Refined and deodorized Cottonseed oil is one of the purest food products available. Processed cottonseed oil is the fifth leading vegetable oil in the world and the seeds are a vital source of protein and calories in the cattle and dairy industries. Fats and oils are a combination of fatty acids, both saturated (C14:0, 16:0, etc.) and unsaturated (C 18:1, 18:2, 18:3). Cottonseed oil also termed as "Heart Oil" is among the most unsaturated edible oils. It has a 2:1 ratio of polyunsaturated to saturated fatty acids and generally consists of 73% unsaturated fatty acids including 18% monounsaturated (oleic) and 55% polyunsaturated (linoleic) and 27% saturated (palmitic and stearic) fatty acids. Cottonseed oil is cholesterol free, as are all oils extracted from plants. An additional benefit from Cottonseed Oil is the high level of antioxidants tocopherol [1].

**Table 1 : Cottonseed oil and food manufacturing applications**

Sr.no	Use	Remark
1	Deep frying	Cottonseed oil is an excellent flavour carrier and enhances rather than masks the fresh, natural flavours of foods.
2	Baking	Cottonseed oil is noted for its ability to form the beta prime crystal, which helps promote the desired consistent texture and smooth, creamy appearance in shortenings, toppings and spreads. Blending of fully hydrogenated cottonseed

		oil with a partially hydrogenated base stock creates a shortening with a solid fat index that achieves an optimum plasticity for baked goods. Margarines, icings and whipped toppings
3	Salad dressings	Excellent choice for salad dressings owing to its neutral flavour profile and the fact that it can be winterised- a necessity for mayonnaise and commercial flavoured dressings. Dressings made with cottonseed oil resist oxidation well.

**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 chromogens 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 (70° to 80°C) 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 110-115°C 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, specific 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. Some edible oils such as cottonseed, soyabean, sunflower and olive oils are adulterated with cottonseed oil up to 5% because the sensitivity of Halphen test is up to 5% 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°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[7].

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 BTTT as tool for identification of cottonseed oil. 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.

### 1.3 Literature review

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 [7].

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 [3].

**Table-2 Shows BTT standards/values for some edible vegetable oils under 2.2: Fats, oils and Fat emulsions as per FSSA 2006[4]**

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

Source FSSA 2006

### 1.3. Material and Experimental Methods

#### 1.3.1 Materials

All the chemicals and reagents were analytical grade and used as received. Five cottonseed oils of different brands such as refined cotton seed oil (Rct, Samara), pure cottonseed oil (Pct, Tirupati), refined cottonseed oil (Rct1, Tirupati), cottonseed oil (Ct, Rani) and pure cottonseed oil (Pct1, Glick's) 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.

### 1.3.2 Experimental Methods

#### 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 [4,5,6].

**Table 3: 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	Samara	Rct	19.4	0.2	1.03	0.12
2	cottonseed oil	Tirupati	Ct	20.8	0.17	0.83	0.1
3	Pure cottonseed oil	Tirupati	Pct	21.0	0.1	0.48	0.06
4	cottonseed oil	Rani	Ct2	20.6	0.17	0.81	0.1
5	Pure cottonseed oil	Glick's	Pct1	20.9	0.1	0.48	0.06

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

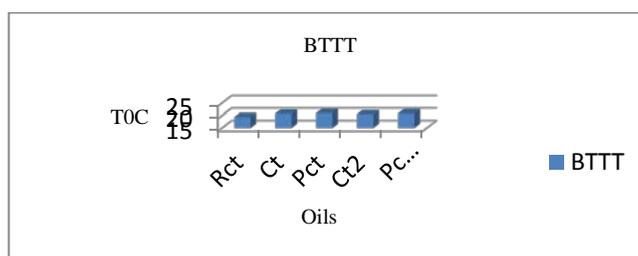


Fig.1 shows the BTTT values for different cottonseed oil

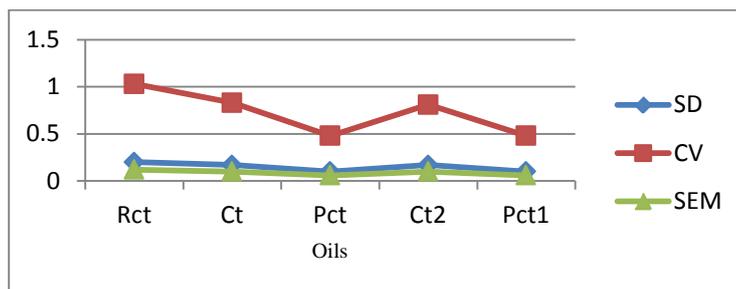


Fig.2 shows the Statistical values for different cottonseed oil

#### 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

Table1 shows the cottonseed oil and food manufacturing applications. Table-2 Shows BTT standards/values for some edible vegetable oils under 2.2: Fats, oils and Fat emulsions as per FSSA 2006[4].The results obtained for the BTTT and statistical accuracy for the cottonseed oils obtained from different places of India are shown in **Table3, Fig 1 and Fig 2**. The data obtained for Rct (19.4),Pct(20.8),Rct1(21.0) ,Ct(20.6) and Pct1(20.9) 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 3** shows the accuracy, the standard deviation and coefficient is in the range of 0.1 -0.2 and 0.48-1.02.

#### 1.6 Recommendation

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.

#### 1.7 Conclusion

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 oils from different places of India. The present investigations prove with due certainty about applicability of BTTT to all four cottonseed oils. This study also confirms prove reliability, reproducibility and diverse applicability of BTTT. BTTT should be quantitative 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.

## 2. References:-

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