

## STANDARDIZATION OF TILA TAILA WITH SPECIAL REFERENCE TO PHYSICO-CHEMICAL PARAMETERS AND HPTLC

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### ABSTRACT

Tila is one of the best drugs of sasyaja origin. Tila has been known in India from the ancient times. Sesame oil, otherwise also referred to as gingelly oil, is one of the major sources of edible oil in India and is culturally associated from the Vedic period. Sesame oil is used as a solvent, oleaginous vehicle for drugs, and skin softener. Chlorosesamone obtained from roots of sesame has antifungal activity. *Sesamum indicum* (sesame) seed oil and related cosmetic ingredients are derived from *Sesamum indicum*. *Sesamum indicum* (sesame) seed oil, *Sesamum indicum* (sesame) oil unsaponifiables, and hydrogenated sesame seed oil function as conditioning agents. In Ayurveda Classics Tila taila is best among the Tailas for the purpose of Snehana Karma. Standardization of Tila taila through physico-chemical parameters and HPTLC, Refractive index, Determination of acid value, Determination of Saponification value, Determination of unsaponifiable matter, Iodine value, Weight, Specific gravity assessed.

**KEYWORDS:** Tila taila, Physico-chemical parameters, HPTLC.

### INTRODUCTION

Tila is one of the best drugs of sasyaja origin. Tila has been known in India from the ancient times. Sesame oil, otherwise also referred to as gingelly oil, is one of the major sources of edible oil in India and is culturally associated from the Vedic period<sup>1</sup>. The Sanskrit word for oil is taila and is derived from the Sanskrit word for sesame i.e. Tila. It is called “sesame” internationally. It is considered to be oldest oil seed crop known to man. Tila seed is an important source of edible oil and is also widely used as a spice. Sesame seed is considered to be the oldest oilseed crop

known, domesticated well over 5000 years ago. Sesame is very rough-tolerant. It has been called a survivor crop, with an ability to grow where most crops fail. Sesame has one of the highest oil contents of any seed. With a rich nutty flavour, it is a common ingredient in cuisines across the world. *Sesamum indicum* (Sesame) is an ancient spice, one of the first recorded plants used for its seeds<sup>2</sup>. It has been used for thousands of years and is still an oil seed of worldwide significance. Non-culinary uses include its application as an ingredient in soap, cosmetics, lubricants and medicines. In

southern India it is used to anoint the body and hair. Sesame oil is used as a solvent, oleaginous vehicle for drugs, and skin softener. Chlorosessamone obtained from roots of sesame has antifungal activity.

*Sesamum indicum* (sesame) seed oil and related cosmetic ingredients are derived from *Sesamum indicum*. *Sesamum indicum* (sesame) seed oil, *Sesamum indicum* (sesame) oil unsaponifiables, and hydrogenated sesame seed oil function as conditioning agents. In Ayurveda Classics Tila taila is best among the tailas for the purpose of Snehana Karma<sup>3</sup>. Sodium sesameseedate functions as a cleansing agent, emulsifying agent, and a nonaqueous viscosity increasing agent<sup>4</sup>. These ingredients are neither skin irritants, sensitizers, teratogens, nor carcinogens at exposures that would result from cosmetic use. Both animal and human data relevant to the cosmetic use of these ingredients were reviewed. The Committed Information Rate Expert Panel concluded that these ingredients are safe in the present practices of use and concentration as described in this safety assessment.<sup>5</sup>

## MATERIALS AND METHODS

Good quality of Krishna Tila (*Sesamum indicum*) weighing of 1 kilogram was purchased from market and, Cleaned and 600 ml Tila taila was extracted in Bhairaveshwara milling center at Hassan.

### Refractive index

Place a drop of water on the prism and adjust the drive knob in such a way that the boundary line intersects the separatrix exactly at the center. Note the reading. Distilled water has a refractive index of 1.3325 at 25 degree C. The difference

between the reading and 1.3325 gives the error of the instrument. If the reading is less than 1.3325, the error is minus (-) then the correction is (+) if the reading is more, the error is plus (+) and the correction is minus (-). Refractive index of oil is determined using 1 drop of the sample. The correction if any should be applied to the measured reading to get the accurate refractive index. Refractive index of the test samples were measured at 28 degree C.

### Determination of acid value

Weighed 10g of sample in a conical flask. Added 50 ml of acid free alcohol-ether mixture (25+25ml) previously neutralized by the addition of 1 ml of phenolphthalein solution and titrated against 0.1N potassium hydroxide solution. End point was the appearance of pale pink colour which persists for 15sec. repeated the experiment twice to get concordant values.

$$\text{Acid value} = \frac{56.1 \times \text{Titer} \times \text{Strength of Potassium hydroxide}}{\text{Weight of the Oil/ fat}}$$

### Determination of Saponification value

About 2g of the substance was weighed in tared 250ml round bottom flask. 25ml of the alcoholic solution of KOH was added and a reflux condenser was attached. Kept it for boiling on water bath for 1 hr, the contents of the flask was rotated frequently. The flask was cooled and 1 ml phenolphthalein solution was added and excess of alkali titrated with 0.5N HCL. The number of ml (a) required was noted. The experiment was repeated with the same quantities of reagents in the same manner omitting the substance. The number of ml required (b) was noted. The experiment was repeated twice to get concordant values.

$$\text{Saponification value} = \frac{56.1 \times (b-a) \times \text{Strength of hydrochloric acid}}{\text{Weight of the sample taken}}$$

### Determination of unsaponifiable matter

Weighed 5g of the substance into the flask. Added 50ml alcoholic KOH into the sample. Boiled gently but steadily under reflux condenser for one hour. The condenser was washed with 10ml of ethyl alcohol and the mixture was collected and transferred to a separating funnel. The transfer was completed by washing the sample with ethyl alcohol and cold water. Altogether, 50ml of water was added to the separating funnel followed by an addition of 50 ml petroleum ether. The stopper was inserted and shaken vigorously for 1 minute and allowed it to settle until both the layers were clear. The lower layer containing the soap solution was transferred to another separating funnel and repeated the ether extracts were collected in a separating funnel. The combined extracts were washed in the funnel 3 times with 25ml of water until the water no longer turns pink on addition of a few drops of Phenolphthalein indicator solution. The ether layer was transferred to a tarred flask containing few pieces of pumice stone and evaporated to dryness on a water bath. Placed the flask in an air oven at 85 degree C for about 1 hour to remove the last traces of ether. A few ml of Acetone was added and evaporated to dryness on a

water bath. Cooled in desiccators to remove last traces of moisture and then weighed.

### Iodine value

The sample was accurately weighed in a dry iodine flask. Dissolved with 10ml of CCl<sub>4</sub>, 20 ml of iodine mono-chloride solution was added. Stopper was inserted, which was previously moistened with solution of potassium iodide and flask was kept in a dark place at a temperature of about 17° C for 30 minute 15ml of potassium iodide and 100ml of water was added and shaken well. This was titrated with 0.1N sodium thio-sulphate, starch was used as indicator. The number of ml of 0.1N sodium thiosulphate required (a) was noted. The experiment was repeated with the same quantities of reagents in the same manner omitting the substance. The experiment was repeated twice to get concordant values.

### Weight per ml

It is obtained by dividing the weight of sample, by 10, contained in the 10ml relative density bottle.

### Specific gravity

Carefully fill the specific gravity bottle with test liquid, insert the stopper and remove the surplus liquid. Note the weight and repeat the procedure using distilled water in the place of sample.

$$\text{Specific gravity} = \frac{\text{Weight of the sample held in the specific gravity bottle}}{\text{Weight of water held in specific gravity bottle}}$$

## RESULTS

**Table.No.1:** Analytical parameters of Tila taila

Sl.no.	Parameter	Value
01	Refractive index	1.47229
02	Unsaponifiable matter	3.99
03	Saponification value	169.5
04	Acid value	3.4
05	Specific gravity	0.917

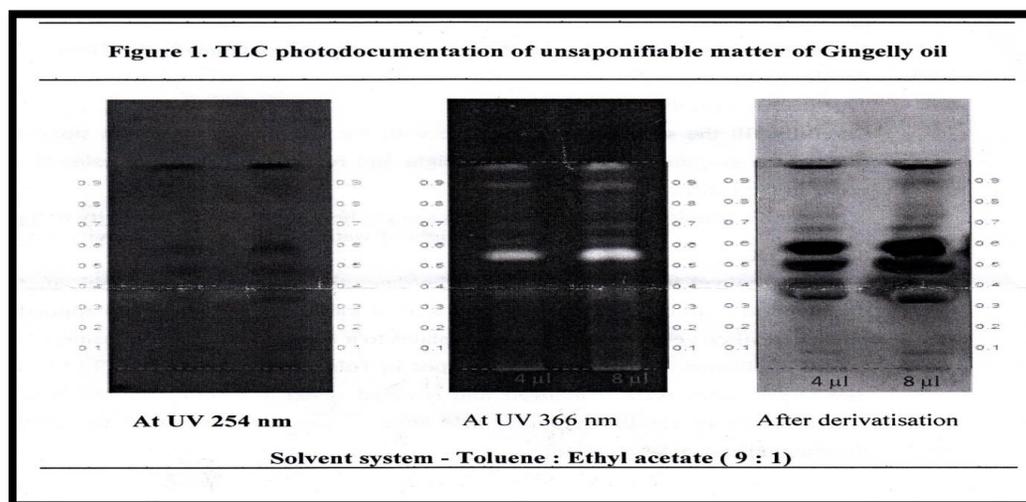
06	Weight per ml	0.916
07	Iodine value	6.84

**Table No.2:** Standards as per Ayurvedic pharmacopeia of India<sup>6</sup>

Contents	Ayurvedic pharmacopeia of India
Foreign matter	Not more than 2 per cent, Appendix 2.2.2.
Total Ash	Not more than 9 per cent, Appendix 2.2.3.
Acid-insoluble ash	Acid-insoluble ash Not more than 1.5 per cent, Appendix 2.2.4
Alcohol	Extractive Not less than 20 per cent, Appendix 2.2.6.
Soluble Water	Soluble extractive Not less than 4 per cent, Appendix 2.2.7.
Fixed Oil	Not less than 35 per cent, Appendix 2.2.8

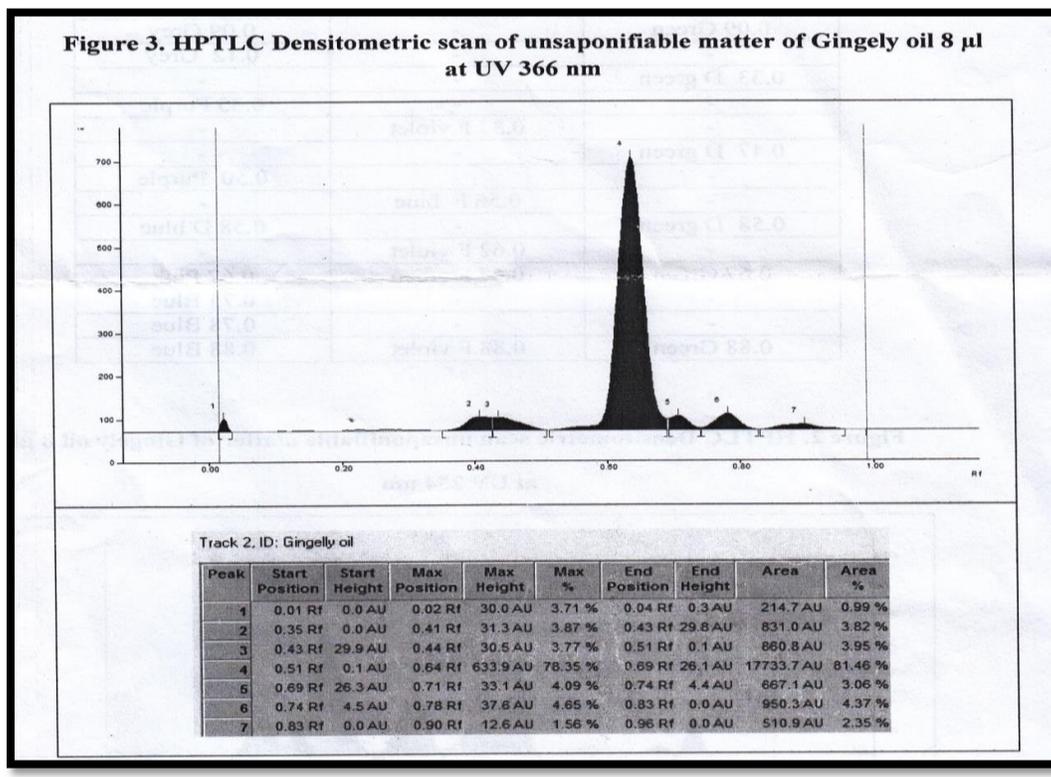
**Table No.3:** TLC results

T.L.C. of Alcoholic extract <sup>7</sup>	
Silica gel 'G' plate using Toluene	Ethylacetate (9 : 1)
UV (366 nm) three fluorescent zones	0.57, 0.64 (both light blue) and 0.72 (blue)
Iodine vapour	Five spots appear at Rf. 0.08, 0.57, 0.64, 0.72 and 0.94 (all yellow).
Vanillin-Sulphuric acid reagent and heating the plate for ten minutes at 110o C	Seven spots appear at Rf. 0.08, 0.57, 0.64, 0.72 (all violet), 0.76, 0.84 (both light violet) and 0.94 (violet).



## HPTLC

Sample obtained in the procedure for the determination of unsaponifiable matter is dissolved in 5ml of chloroform. 4 and 8 $\mu$ L of the above samples were applied on a precoated silica gel F254 on aluminum plates to a band width of 8 mm using Linomat 5 TLC applicator. The plate was developed in Toluene – Ethyl acetate (9:1) and the developed plates were visualized and scanned under UV 254 and 366 nm, after derivatisation in vanillin-sulphuric acid spray reagent.  $R_f$ , colour of the spots and densitometric scan were recorded



## DISCUSSION

Before considering any trials, drug must be standardized by comparing with available various authentic research bodies such as API & PSAF then only valuable results can be obtained with proper quality of drug. Hence Efficacy of the drug can be proved.

## CONCLUSION

The sample has been standardized as per testing protocol for oils. Result of standardization parameters shown in table 1. TLC photo documentation,  $R_f$  values and

HPTLC densitometric scan have been documented.

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