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PREPARATION OF SIDDHA HERBAL DISTILLATE FROM TRADITIONAL STILL AND GLASS STILL; A COMPARATIVE STUDY

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ABSTRACT

Background: The quality or yield of a *Theeneer* (Herbal distillate*)* is mainly dependent on the nature of the raw drugs and the type of distillation apparatus used for extraction. *Siddha* classical texts describes so many traditional or modified varieties of stills for selective distillation. **Aim and Objectives:** This preliminary study is aimed to compare the quality differences of classical *Siddha* distillate *Sanjeevi Theeneer* prepared in both Traditional Still and Glass apparatus through GC-MS and to identify which make of distillate meets the standard traditional parameters as mentioned in the *Siddha* literature. **Methods:** The distillate is prepared in two different stills (Traditional apparatus -ST (t) and Glass made- ST (g)) for quality comparison and GC-MS studies. **Results:** There were considerable differences between the two samples in terms of traditional quality (Color, aroma & taste) and yield. ST (t) distillates meet the standards of parameters than ST (g). GCMS reported the presence of major

compounds like Isothymol in both the samples with varying peak percentage. 6,7-Dimethyl-3,5,8,8a-tetrahydro-1H-2-benzopyran being the active compound of ST(g). Terpineol, beta terpineols and linoleic acid were the other active compounds of ST (t). **Conclusion:** In supportive with the study and analysis we conclude that distillates prepared from Traditional stills are far better in quality, therapeutically potent than those prepared from glass stills.

KEYWORDS: Siddha medicine, Theeneer, Sanjeevi Theeneer, Traditional quality parameters, GCMS.

I. INTRODUCTION

Theeneer[1,2] is the distilled essence, which contains the volatile constituents or water-soluble constituents of the drugs used in the preparation in a medium of water. They are colloidal suspensions (hydrosol) of essential oils as well as water-soluble components obtained by steam distillation from herbs. By distilling in a special apparatus, the medicine can be collected in an aqueous state.

Siddha system, which was developed under the principles of nature, uses vast variety of material resources. The profounders of *Siddha* medicine called *Siddhars* had an in-depth knowledge in every aspect of natural resources and how it can be transformed into healing elements of medicine. Understanding the body nature and the sufferings it catches is as tough as understanding the elements of nature.[3] For the effective application these natural elements has to be directly or indirectly modified, processed into a form acceptable or assimilable by the body constituents. Distillation is one such art of extracting the valuable essences of a material either of plant origin, animal origin, salt, mineral origin etc.[4,5]

The fundamentals are same for the processing of each material into a distillate but there is considerable variation in the quality and yield of the product in relation with the materials used in it, the methods adopted for its processing and the kind of apparatus used. For distillation, ancient practices of *Siddha* medicine advices the usage of special made apparatus termed as *Valai iyanthram*[6] (Distillation still). So many varieties of stills[4] has been mentioned, commonly used Munn Valai iyanthram (traditional mud apparatus), to rarely used Uloga valai (metal apparatus), Spadika valai or Kannadi Valai (glass apparatus), and Peenkana Valai (Porcelain made apparatus). This was in usage in different periods of Dravidian history.

Each still has its own peculiarity in selective distillation. There will be variations in distillates with each apparatus used which is studied by using two samples one prepared from the Traditional apparatus (ST(t) and the another in a Glass still (ST(g). For the study, *Sanjeevi Theeneer* a distillate with reference to classical *Siddha* texts was selected. As with experience and Literary references, *Theeneer* should have the peculiar color, taste, odor and medicinal property of that of the raw drugs used and such are superior in balancing *Mukutram*[5] *(Three dhosha humors). Theeneer* that do not satisfy the above features were considered as inferior quality distillates. Yield may be accounted as the quantity of the distillate collected which should be more or slightly less than 75% of the total quantity of the medium used. GC-MS (Gas Chromatography Mass Spectrometry) screening can trace out active components of each sample, its peak percentage and the difference in component identity.

II. AIM AND OBJECTIVES

A. Aim

Comparative Study of classical *Siddha* distillate *Sanjeevi Theeneer* prepared in both Traditional Still and Glass apparatus by GC-MS.

B. Primay objective

To identify which make of distillate meets the quality parameters as mentioned in the *Siddha* literature and to assess the bioactive compounds of the two different samples by using GC-MS analysis.

C. Secondary objective

To study the factors determining the quality of distillates.

III. MATERIALS AND METHODS

For the comparative sample studies, we have selected the classical distillate- *Sanjeevi Theeneer* mentioned in the *Siddha* classical texts *Chikitsa ratnadeepam* and *Siddha* Formulary of India.[7]

A. Ingredient Details

There are 12 Ingredients, 1.*Chukku* (dried rhizomes of Zingiber officinale). 2. *Milagu* (Piper nigrum). 3. *Thippili* (Piper longum). 4. *Kadukkai thodu* (dried epicarp of Terminalia chebula). 5. Nellikkai *vatral* (dried fruits of Phyllanthus emblica). 6. *Tantrikkai* (dried epicarp of Terminalia bellerica) 7.*Korai kizhangu* (Cyprus rotundus). 8. Kodiveli (Plumbago zeylanica).

9. *Omam* (Trachyspermum ammi). 10. *Vaividangam* (Embelia ribes). 11. Panam *karkandu* (Palm candy-Borassus flabelifer). 12. Purified *Aya podi* (Ferrum).

B. Raw Drug Collection and Authentication

The raw drugs were purchased from a reputed raw drug store located at *Nagercoil* and *Chennai*. Each raw drug has been identified and authenticated from the Branch of Botany, National Institute of *Siddha* (NISMB2472016), and mineral sample from Department of Geology, Madras University.

C. Method of Preparation of Samples

Each of the raw drugs were cleaned, purified, powdered and are mixed in the prescribed ratio as per the methods mentioned in the *Siddha* classical texts The powder mixture were added in pure water, stirred and sealed well in a vessel for a period of 7 days. This counts the period of fermentation. On the 8th day the mixture along with water were charged in two different stills for separate distillation.

D. Preparation of Distillate Samples

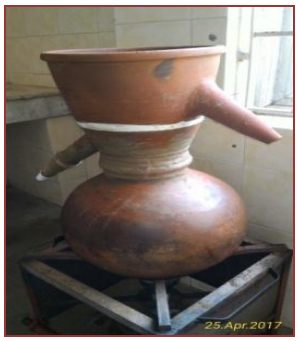
D.1 ST (t)

This mixture (drug with water) were charged in a Traditional distillation apparatus *(valai yanthram*[6]) which consist of a lower Mud pot (*kalayam*[8] *or* mutkalam[9]) and an upper still (*munn valai*[4] (clay made). The lid or junction between the two apparatus or vessel were sealed properly with a *Seelai thuni (*a cloth ribbon of uniform length and width (5cm*1metre, 3nos) smeared with *Seelai munn*[10] (Clay soil) to prevent leakage of vapors. The plastering was allowed to dry completely, then after heating is slowly started to boil the contents. Continuous water current was maintained in the upper vessel with a provision to drain the heated water that was removed frequently. The condensed steam was collected as distillates through the outlet (*keezh kuzhai*[2]) provided in the upper still and were preserved for studies (Fig: 1).

D.2 ST (g)

This mixture (drug with water) were charged in a glass still which consist of a flat bottomed round beaker (1 liter Capacity), Cylindrical condenser with coils (glass made) with provision of water inlet, outlet and steam outlet in one side connected to the beaker via 'U' bend and distillate outlet connected to the collecting bottle. The whole apparatus rests in a heating

mantle and temperature set in different intervals up to the maximum 100 ^{0}C.The distillate collected were preserved for studies (Fig: 1).



1. Valai yanthram (ST (t) **2. Glass Still (Traditional Still)**

Fig. 1: Stills used in distillate preparation.

E. *Gas Chromatography- Mass Spectroscopy Analysis (GC-MS)*[11,12]

The Quantitative analysis of bioactive compounds present in the two different samples were carried out with GC-MS.

IV. RESULTS AND DISCUSSION

There will be difference in the nature and percentage value of the original chemical compounds during the various stages of distillation. The most possible known mechanisms will be the loss or alteration during various purification procedures that is specific to the raw drug or general (including washing, excess drying or sun drying), bio degradation due to pH variation while fermentation, inter drug molecular interactions- synergist or antagonist (in compound formulations), heat interaction (due to the nature, thickness and quality of the apparatus), heat degradation (during boiling, evaporation and re-distillation). As the process up to the fermentation is similar to both the distillates, the factors of heat interaction and heat degradation can be coined to explain the differences in quality of the distillates. (Basic difference of Traditional and Glass stills and Samples ST (t) and ST (g) (Table: 1).

Table 1: Basic Comparison between Traditional and Glass Stills.

S. No.	Features	Glass Stills	Traditional Apparatus
1	**Make of the Apparatus**	**Glass**	**Clay Soil**
2	**Parts of the Apparatus (full set)**	**a**. Glass beaker (Borosil flat bottomed (1no). **b.** 'U'Bend (2 nos) **c**. Condenser with Steam inlet, Distillate outlet, water inlet &	a. Lower Vessel b. Upper Vessel with condenser part, Water inlet and outlets, Distillate outlet. c. Collecting Vessel or bottle.

		outlet. **d**. Collecting Bottle (1no). **e**. Heating Mesh.	
3	**Working on**	Electricity	Conventional Fuels
4	**Maximum heat control**	100 ^{0} C	> 100 ^{0} C (approx)
5	**Heat Loss**	Minimal	Considerable
6	**Steam Loss**	Minimal	Depends on apparatus quality and sealing.
7	**Safety Issues**	Suspectable to breakage during process or inexperienced handling. Safety issues are concerned.	No issues of safety
8	**Purpose & Limitations**	For distillation of water, alcohol, herbs. *Not suited for Salt distillations or super concentrated distillates.	For distillation of wide range of materials of herbal, herbo-mineral compounds. *Best for salt distillations and concentrated distillates. *Best for Manufacturing Traditional therapeutic distillates.

Glass stills offer purity of the compounds, yield and is suited for commercial purpose. The quality of the distillate as when considering the traditional quality parameters like color, odor, aroma is dependent on aromatic and pungent compounds extracted from herbs and spices. This may help in comparison of same distillates prepared by different methods and in assessing the effective extraction by similar methods. Thus by comparing on these parameters, traditional made distillate have priority over glass still distillate. The typical lemon yellow color, pleasant aroma and pungent taste were more reported in ST (t) **(**Table 3).

IV a. GCMS REPORTS

There is much difference in the compounds screened in both samples. GCMS analysis of sample ST (t) revealed the presence of 7 compounds (Fig. 2 and Table. 2, 3). Sample ST (g) Shown 8 compounds on GCMS screening (Fig. 3 and Table. 2, 3).

An identical Major compound, spotted in both the samples of ST (t) and ST (g). It had similar intensity peak with retention time varying from 6.332 to 6.335 and the compound responsible for this peak is Isothymol.[13] It occupies the peak area of 72.97% in ST (t) sample as compared with ST (g) 48.30% indicating its maximum extraction through traditional still. Other predominant compounds of ST (t) may be considered as biologically active.

Thus by comparing the Extend of component extraction (peak %) and most of biologically active constituents spotted through GCMS, distillate prepared from traditional still was far superior than distillate made in glass still (Table.3).

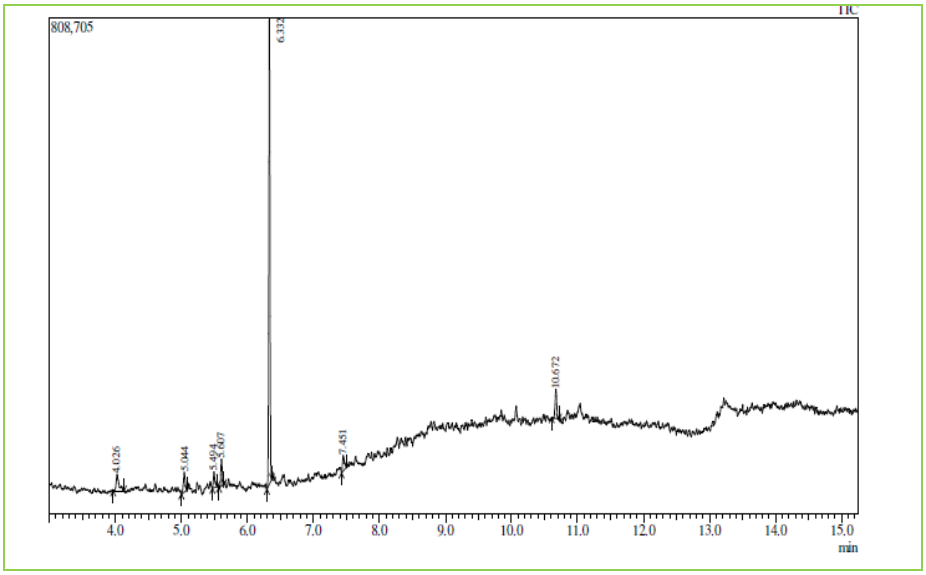


Fig. 2. GC-MS chromatogram of ST (g).

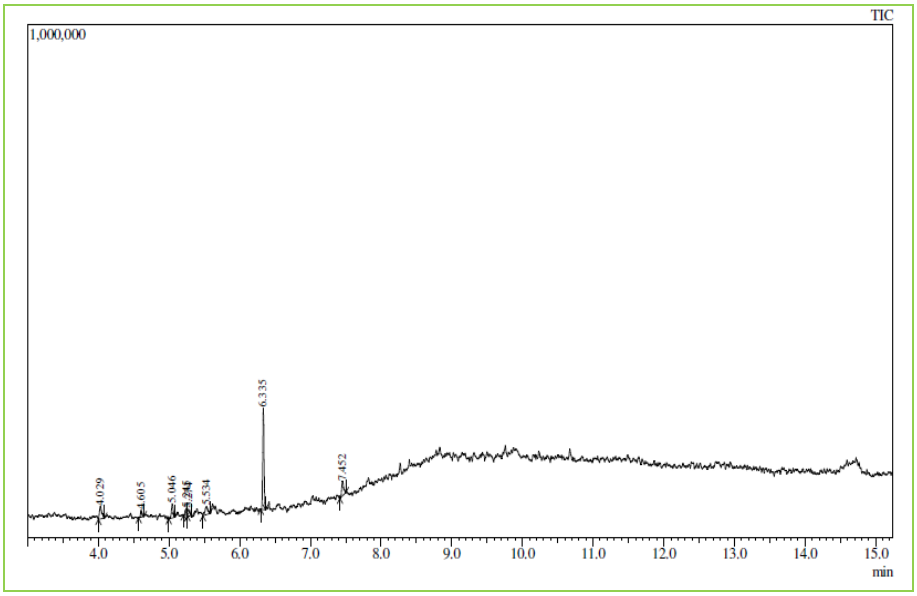
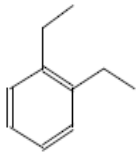
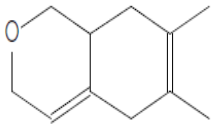
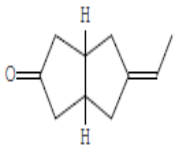
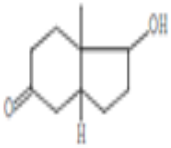
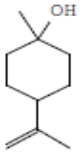
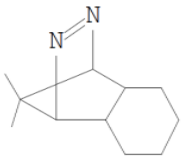
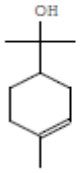
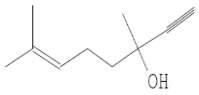
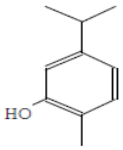
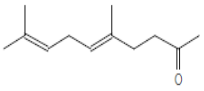
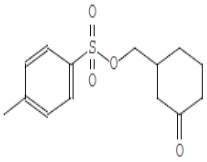
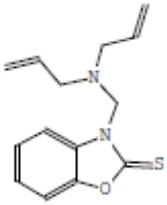
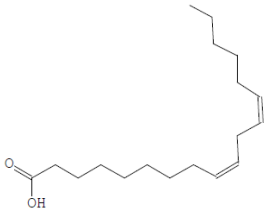
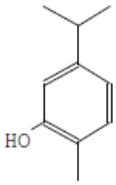
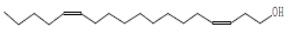


Fig. 3: GC-MS chromatogram of ST (t).

Table 2: GC-MS reports of ST(t) and ST(g) distillate.

	Bio active compounds from ST (t) distillate		Bio active compounds from ST (g) distillate
1	*1,2-Diethylbenzene*  Peak No: 1 | % Peak Area: 5.71 *RT: 4.026 +MW: 134 | ^PIR: 2	1	*6,7-Dimethyl-3, 5,8,8atetrahydro-1H-2 benzopyran*  % Peak No: 1 | % Peak Area: 10.87 *RT: 4.029 +MW: 224 | ^PIR: 2
2	*Bicyclo [3.3.0] octan-3-one, 7 ethylidene-*  Peak No: 2 | % Peak Area: 4.60 *RT: 5.044 +MW:150 | ^PIR: 4	2	*Z,Z-8,10-Hexadecadien-1-ol*  Peak No: 2 | % Peak Area: 3.45 *RT: 4.605 +MW: 238 | ^PIR: 8
3	*Beta.-Terpineol*  Peak No: 3 | % Peak Area: 3.17 *RT: 5.494 +MW: 154 | ^PIR: 7	3	*1,4-Methanophthalazine*  Peak No: 3 | % Peak Area: 9.06 *RT: 5.046 +MW: 178 | ^PIR: 4
4	*Terpineol*  Peak No: 4 | % Peak Area: 4.40 *RT: 5.607 +MW: 154 | ^PIR: 5	4	*Octen-1-yn-3-ol*  Peak No: 4 | % Peak Area: 4.18 *RT: 5.245 +MW: 152 | ^PIR: 7
5	*Isothymol* 	5	*5,8-Decadien-2-one*

	Peak No: 5 | % Peak Area: **72.97** *RT: **6.332** +MW: 150 | ^PIR: 1		 Peak No: 5 | % Peak Area: 4.96 *RT: 5.274 +MW: 180 | ^PIR: 6
6	*p-toluene sulfonate*  Peak No: 6 | % Peak Area: 3.74 *RT: 7.451 +MW: 282 | ^PIR: 6	6	*Benzoxazol*  Peak No: 6 | % Peak Area: 8.44 *RT: 5.534 +MW: 260 | ^PIR: 5
7	*cis-Linoleic acid*  Peak No: 7 | % Peak Area: 5.40 *RT: 10.672 +MW: 280 | ^PIR: 3	7	*Isothymol*  Peak No: 7 | % Peak Area: 48.30 *RT: 6.335 +MW: 150 | ^PIR:1
		8	*Z,Z-3,13-Octadecadien-1-ol*  Peak No: 8 | % Peak Area: 10.73 *RT: 7.452 +MW: 266 | ^PIR: 3

Tabel 3: Comparison of ST(t) distillate over ST(g) distillate.

A	Traditional Parameters	Traditional Distillate (ST(t)	Glass Still Distillate (ST(g)
a	**Nature & Appearance**	Good clarity, good Purity	Good Clarity and purity
	1.Color	Light lemon yellow	Colorless
	2.Aroma	Pleasant Aroma	Mildly aromatic
	3.Taste	Pleasant taste, slightly pungent	No pleasant Taste, Slightly pungent
b	**Yield**	Less than Average	Good (>75%)
c	**Effect on Long storage**		
	Nature & Appearance	Clarity +, Sediments +	No change in clarity, no sediments
	1.Color	Color Fading+	No change in color
	2.Aroma	Mildly aromatic	Aroma absent
	3.Taste	Slight pleasant taste, very less pungent	No pleasant taste, very less pungent
B	**GCMS Analysis**		
	1.	*Isothymol (**72.97%**)*	*Isothymol (**48.30%**)*
	2.	*1,2-Diethylbenzene*	*6,7-Dimethyl-3,5,8,8a-tetrahydro-1H-2-benzopyran*
	3.	*Bicyclo[3.3.0]octan-3-one, 7-ethylidene-*	*Z,Z-8,10-Hexadecadien-1-ol*
	4.	*Beta.-Terpineol*	*1, 4 Methano phthalazine*
	5.	*Terpineol*	*Octen-1-yn-3-ol*
	6.	*p-toluene sulfonate*	*5,8-Decadien-2-one*
	7.	*cis-Linoleic acid*	*Benzoxazol*
	8.	----------	*Z,Z-3,13-Octadecadien-1-ol*

V. CONCLUSION

Choosing the ideal apparatus for distillation is the priority for obtaining a high-class distillate. Traditional stills made from good quality clay can sustain good heat and can be tuned to higher temperatures, apart from it, the porous nature allow entry of cool air to facilitate steam generation and this is why they are way ahead in obtaining maximum extraction of organic compounds. Traditional stills are non reactive to any herbs, chemicals or salts and thus it can be used for distilling Mineral compounds. The Color, aroma and taste of the distillate is more retained in a traditional still and therefore distillates made from traditional stills are more therapeutically active than glass stills. Upgrading the quality of Traditional distillation apparatus can overcome its limitations in terms of yield or wastage. Whether the compounds are retained or lost during the manufacture of distillates still needs a progressive research and

review. By properly setting the standards of distillate preparation and through its progressive analysis of each sample over a number of time one can ascertain the major active compounds of high percentage from the distillate for further pharmacological evaluation.

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