ORIGINAL ARTICLE

Physico-chemical standardization of Habbe Shifa: A polyherbal Unani formulation with modern techniques

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Background: Habbe Shifa (HS) is an important pharmacopoeial Unani formulation, which is used in the treatment of *Humma* (fever), *Iya* (fatigue), *Tashannuje rewi* (pulmonary spasm) and *Zeequn Nafas* (asthma) and opium deaddiction. **Aim:** The physico-chemical standards of HS were established in the present study. **Materials and Methods:** HS was prepared with ingredients of particle size 150 µm (100 mesh sieve), 5% w/w Gum Acacia mucilage was used as binder, dried at a temperature 90°C for 120 min and finally evaluated for different physico-chemical parameters to develop standards for HS. **Results and Conclusion:** Physico-chemical standards of HS were observed as characteristic brown colour, spherical shape, hard in texture, odourless and bitter in taste; average weight 242.95 ± 1.53 mg; diameter 7.33 ± 0.16 mm; hardness 3.5 ± 0.00 kg/cm; friability 0.02 ± 0.003%; pH value in 1% and 10% aqueous solution 6.22 ± 0.06 and 5.39 ± 0.008 respectively; percentage loss of weight on drying at 105°C 6.63 ± 0.12%; total ash, acid insoluble ash and water soluble ash 5.33 ± 0.16, 0.95 ± 0.05 and 1 ± 0.00% respectively and total alkaloid 0.65 ± 0.01% and R_f values in the thin layer chromatography in ethanolic extract in hexane: Acetone (7.6:2.4) solvent system were 0.25, 0.78 and in hexane: Diethyl ether (4:6) solvent system were 0.58, 0.89. The results obtained for the various physico-chemical tests of lab sample of HS may be taken as standard parameter for future reference and help in setting up regulatory limit to assure the quality of Unani medicine.

Key words: Habbe Shifa, physico-chemical, standardization

INTRODUCTION

Medicinal plants have been known for millennia and are highly esteemed all over the world as a rich source of therapeutic agents for the prevention of diseases and ailments.[1] In spite of the great advances observed in modern medicine in recent decades, plants still make an important contribution to health-care. Now a day's herbal drug are gaining popularity in the world market due to the side-effects produce by synthetic drugs. India has a unique position in the world, where a number of recognized indigenous systems of medicine viz., Unani, Ayurveda, Siddha, Homeopathy, Yoga and Naturopathy are being utilized for the health-care of people. Herbal drugs are generally used in Unani, Ayurveda and Siddha and all these drugs have a very good therapeutic value/efficacy, but these medicines are widely criticized only due to lack of standardization and poor quality presentation. Development of standard operating procedures and establishment of physico-chemical standards of all products are two important tools to assure the quality of herbal drugs. Three attributes are described for standardization and quality assurance purposes, i.e., authenticity, purity and assay. Authenticity relates to proving that the material is true. Purity pertains to evaluating that there are no adulterants present in plant material. Assay part of standardization is chemical and biological profiling, which could assess the chemical effects and curative get established.[2]

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Composition of herbal drugs is quite variable because of various factors such as misidentification of plants, contamination, substitution and adulteration of plants, incorrect preparations and/or dosage, etc., Other factors such as temperature, light exposure, water availability, nutrients, period and time of collection, method of collecting, drying, packing, storage and transportation of raw material, age and part of the plant collected, etc., can greatly affect the quality, composition and consequently the therapeutic value of herbal medicines.[3] Thus, proper standardization and quality control of raw material and the herbal preparations should be permanently carried out.

Habb (Pill) is one of the earliest dosage forms and was invented by ancient Unani physicians. Habbe Shifa (HS)

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is an important pharmacopoeial Unani formulation, which is widely used in the treatment of *Humma* (fever), *Iya* (fatigue), *Tashannuje rewi* (pulmonary spasm) and *Zeequn Nafas* (asthma) and opium deaddiction.[4-6] The analgesic, anti-convulsant and anti-pyretic activity of HS has been reported by Tajuddin *et al*. and aqueous and alcoholic extract of HS was found to produce significant analgesic, anti-convulsant and anti-pyretic activity,[7] but there is no data available on physico-chemical standardization of HS. Therefore, in the present study, physico-chemical standardization of a polyherbal Unani formulation HS was carried out.

MATERIALS AND METHODS

Ingredients of HS

The ingredients of HS as per National Formulary of Unani Medicine (NFUM) are given in table [Table 1].[5]

Procurement of Raw Drugs

The ingredients of HS were procured from the registered crude drug dealer, Bangalore and after checking the identity and purity, pills were prepared according to the method given below. Voucher specimens have been kept in our museum for future references.

Preparation of Powders

All crude drugs were grounded with the help of an electric grinder and passed through sieve no. 100 to get powders of less than or equal to150 µm of dimensions for preparing of HS.[8,9]

Preparation of Loabe Samaghe Arabi (Gum Acacia mucilage

Around 5% w/w Loabe Samaghe Arabi GAM was used as a binder for preparation of HS GAM was prepared as per the method given by Pharmacopoeia of India.[10]

Preparation of Huboob (Pills)

HS were prepared manually according to instructions prescribed in NFUM and dried at 90°C for 120 min in a hot air oven.[5]

Physico-Chemical Studies

The physico-chemical studies were carried out on HS prepared in the laboratory of Department of Ilmul Saidla, National Institute of Unani Medicine, Bangalore.

Organoleptic Properties

Organoleptic properties of pills such as appearance, colour, smell and taste were noted.

Weight Variation of Pill

A total of 20 pills were randomly selected and individually weighed, their average weight were determined and comparing the individual pills weight to the average. The deviation from the average weight in each case was calculated and expressed as a percentage. The pills meet the test if no more than 2 pills are outside the percentage limit of 7.5%.[8,11]

Table 1: Ingredients of Habbe Shifa

Ingredient	Botanical name	Quantity
Tukhme dhatura	*Datura stramonium*	6 parts
Rewandchini	*Rheum emodi*	4 parts
Zanjabeel or Sonth	*Zingiber officinale*	2 parts
Samaghe Arabi	*Acacia arabica*	2 parts

Diameter of Pill

Uniformity of diameter was performed by picking three pills randomly and the diameter was measured individually by using a vernier caliper and expressed in mm.[12]

Hardness of Pill

Hardness of pills was evaluated by Monsanto hardness tester. Hardness was performed on three tablets in all instances and the average values were recorded.[11]

Friability Test

Friability of the pills was determined using Roche's Friability test apparatus also called Friabilator (Labinda Tab Friability Tester). The friability (f) is calculated by the formula:

$$f = \left(1 - \frac{W}{W_0}\right) \times 100$$

Where, W is the weight of the pills before the test and W_0 is the weight of the pills after the test. The procedure was repeated 3 times and the mean value was calculated.[11,13]

Determination of pH (1% and 10% Solution)

An accurately weighed 1 g and 10 g of powdered drug was dissolved in accurately measured 100 ml of distilled water, filtered and pH was measured with a pH meter.[14]

Determination of Moisture Content

The moisture content of the drug was determined by Toluene Distillation method and average value was noted.[15]

Loss of Weight on Drying

Five gram of the drug was taken, spread uniformly and thinly in a shallow petri dish and it was heated at a regulated temperature of 105 ± 1°C, cooled in a desiccator and weighed. The process was repeated many times until two consecutive weights were constant. The percentage loss in weight was calculated with respect to initial weight.[14-16]

Ash Values Determination

Total ash

A sample of 2 g of air dried powdered drug was incinerated in a silica dish at a temperature not exceeding 450°C until free from Carbon, cooled and weighed and the percentage was calculated with reference to air dried drug.[15-17]

Acid insoluble ash

The total ash was boiled with 25 ml of dilute hydrochloric acid for 5 min. The insoluble matter was collected on an ash less filter paper washed with hot water and ignited at a temperature not exceeding 450°C and weighed after cooling. The percentage of acid insoluble ash was calculated with reference to the air dried drug.[15-17]

Water soluble ash

The total ash was boiled with 25 ml of distilled water for 5 min. The insoluble matter was collected on an ash less filter paper, washed with hot water and ignited. The weight of insoluble ash was subtracted from the weight of the total ash, giving the weight of the water soluble ash. The percentage of water soluble ash was calculated with reference to air dried drug.[15-17]

Determination of Water and Alcohol Soluble Matter

Cold maceration

Accurately weighed 4 g of the drug was placed in a glass stoppered conical flask. Macerate with 100 ml of water for 6 h shaking frequently, and then wait for 18 h. Shake well and filter rapidly through dry filter paper. A total of 25 ml of the filtrate was transfer to a previously weigh and tarred flat-bottomed dish and evaporate to dryness on a water bath. Dry at 105°C for 6 h, cool in a desiccator for 30 min and weigh without delay. The percentage of water soluble matter was calculated with reference to the amount of drug taken. The percentage of alcohol soluble content was determined as above by using alcohol in place of water.[16]

Determination of Extractive Values

Successive extractive value

The extractive values of HS in different solvents viz. petroleum ether, chloroform, alcohol and water were carried out by percolation in soxhlet's apparatus. The heat was applied for 6 h at a temperature of 40-60°C for petroleum ether, 60°C for chloroform, 78°C for alcohol and 100°C for water. Powdered drug was taken and subjected to successive extraction with each solvent. The extracts were filtered using filter paper and after evaporation of the solvents on water bath, the values were determined with reference to the weight of air dried drug (% w/w).[18]

Non-successive extractive value

The extractive values of HS in different solvents viz. petroleum ether, chloroform, alcohol and water were carried out separately by percolation in soxhlet's apparatus as in successive extraction.[18]

Estimation of Total Alkaloids

Total alkaloid was determined by using Harborne method (1973). 5 g of the sample was taken into a 250 ml beaker and add 200 ml of 10% acetic acid in Ethanol, cover and allow to stand for 4 h. Filter the whole material and concentrate the extract on a water bath to one-quarter of the original volume. Add concentrated ammonium hydroxide drop wise to the extract until the precipitation was complete. The whole solution was allowed to settle and the precipitates were collect and wash with dilute ammonium hydroxide and then filtered. The residue is the alkaloid, which was dried and weighed.[19]

Chromatographic Studies (thin layer chromatography)

TLC was carried out on TLC. Precoated aluminium plates, silica gel 60 F 254 (layer thickness 0.25 mm) for alcoholic extract of HS. The R_F values of the spots were calculated by the following formula[17] [Table 2, Figures 1 and 2].

$$R_F \text{ value} = \frac{\text{Distance traveled by the spot}}{\text{Distance traveled by the solvent}}$$

RESULTS

The results of organoleptic properties, weight variation of the pills, diameter of the pills, hardness of the pills, friability test of the pills, pH values, moisture content, loss of weight on drying, ash values, water and alcohol soluble matter, successive and non-successive extractive values, estimation of total alkaloids and TLC are given in Tables 2 and 3. The data is based on multiple observations.

DISCUSSION AND CONCLUSION

Physico-chemical standardization is a pre-requisite in quality control of Unani drugs in both single as well as compound formulations. The efficacy of a drug mainly depends upon its physical and chemical properties; therefore, the determination of physico-chemical characters for the authenticity of a drug is necessary. Physico-chemical study is also important because it helps in characterization of constituents or groups of constituents that frequently lead to establish the structure-activity relationship and the likely mechanism of action of the drug. Phytochemical constituents

Table 2: TLC of Habbe Shifa

Extract	Solvent system	No. of spots	R_f value
Ethanol	Hexane: Acetone (7.6:2.4)	2	0.25, 0.78
Ethanol	Hexane: Diethyl ether (4:6)	2	0.58, 0.89

TLC – Thin layer chromatography

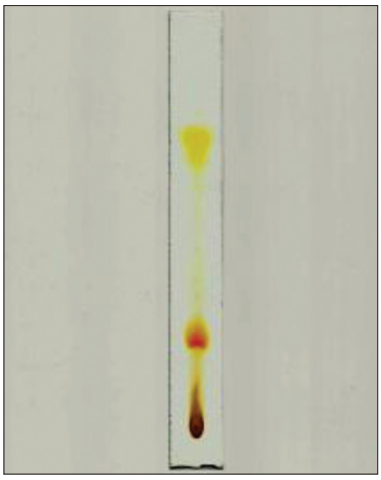

Figure 1: Thin layer chromatography of ethanolic extract of Habbe Shifa (hexane: Acetone)

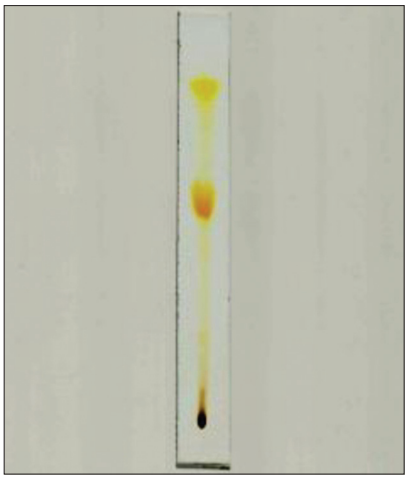

Figure 2: Thin layer chromatography of ethanolic extract of Habbe Shifa (hexane: Diethyl ether)

Table 3: Physico-chemical parameters of Habbe Shifa

Parameters	Results (mean±SEM)
Organoleptic description	
Appearance	Pill
Colour	Brown
Smell	Odourless
Texture	Hard
Taste	Bitter
Average weight (mg)	242.95±1.5
Diameter (mm)	7.33±0.16
Hardness (kg/cm)	3.5±0.00
Friability (%)	0.02±0.003
pH values	
1% solution	6.22±0.06
10% solution	5.39±0.008
Moisture content by toluene distillation method (%)	6.63±0.12
Loss of weight on drying at 105°C (%)	9.33±0.33
Ash values (%)	
Total ash	5.33±0.16
Acid insoluble ash	0.95±0.05
Water soluble ash	1±0.00
Water and alcohol soluble matter	
Water soluble matter	25.56±0.38
Alcohol soluble matter	9.16±0.08
Successive extractive values (%)	
Petroleum ether	7.43±0.23
Chloroform	5.83±0.32
Alcohol	5.70±0.32
Water	14.96±0.98
Non-successive extractive values (%)	
Petroleum ether	7.16±0.11
Alcohol	13.86±1.36
Water	27.03±1.19
Total alkaloid (%)	0.65±0.01

present in the drug vary, not only from plant to plant but also among different samples of same species, depending upon various atmospheric factors, storage and drying conditions. A little deviation from the normal in terms of quality and quantity of the constituents may alter the effect of the drug. Apart from the degradation in the quality of the drugs that occurs due to above conditions, adulteration also contributes a lot to variability. Organoleptic properties are important parameter for rapid identification and consumer acceptance. HS has characteristic brown colour, spherical shape, hard in texture, odourless and bitter in taste the weight of the tablet being made is routinely measured to help ensure that a tablet contains the proper amount of drug. The % variation of the lab samples was within the prescribed pharmacopoeial limits of ± 7.5%. The mean weight value was found to be 242.95 ± 1.53 mg. The diameter of a pill can vary without any change in its weight; hence, the uniformity of diameter of the circular hand-made pills was also measured. The mean value of the diameter was found to be 7.33 ± 0.16 mm. Hardness is a measure of resistance of a solid dosage form to mechanical deforming. The resistance of the tablet or pill to shipping, abrasion or breakage under conditions of storage, transportation and handling before usage depends on its hardness.[8] Hardness was found to be 3.5 ± 0.00 kg/cm. Friability is another important measure of tablet's strength. For Friability a loss of less than 1% is considered acceptable by industrial standards. All pills were found well within the range. The mean percentage was found to be 0.02 ± 0.003%. pH of the pills were determined and was found to be acidic. The values were 6.22 ± 0.06 in 1% aqueous solution and 5.39 ± 0.008 in 10% aqueous solution. Excessive moisture content becomes an ideal medium for the growth of the different types of bacteria as well as fungi. They subsequently spoil the purity of the drug.[16] The percentage of moisture content and loss of weight on drying were found to be 6.63 ± 0.12% and 9.33 ± 0.33%. Determination of the ash value provides a criterion for judging the identity and purity of the drug. The percentage of total ash, acid insoluble ash and water soluble ash were found to be 5.33 ± 0.16, 0.95 ± 0.05 and 1 ± 0.00%. The amount of the extract that the

drugs yield in a solvent is often an approximate measure of the amount of a certain constituent that the drug contains. Therefore, for establishing the standard of any drug the extractive values play a major role.[20] Water soluble matter and alcohol soluble matter were found to be 25.56 ± 0.38% and 9.16 ± 0.08% respectively; successive extractive values in petroleum ether, chloroform, alcohol and water were found to be 7.43 ± 0.23%, 5.83 ± 0.32%, 5.70 ± 0.32% and 14.96 ± 0.98% respectively; non-successive extractive values in petroleum ether, alcohol and water 7.16 ± 0.11%, 13.86 ± 1.36% and 27.03 ± 1.19% respectively.

The assay of alkaloidal drugs and preparations is generally performed for purposes of standardization, proof of purity, commercial evaluation or pharmacological purposes.[20] Total alkaloidal estimation was found to be 0.65 ± 0.01%. TLC is one of the important parameter used for detecting the adulteration and for judging the quality of the drugs.

In conclusion, it can be stated that the results obtained from the various physico-chemical parameters and TLC profiles together may be used for quality evaluation and the standardization of the compound formulation HS. Thus, the data generated in this analysis may be helpful in setting up regulatory limit, to assure the quality of Unani medicine.

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