Physico-chemical Standardization of Safoofe Deedan – A Unani Anthelmintic Powder

Waris Ali,
***Hamiduddin,**
Abdullah Tauheed
and
R. Zaman

Department of Ilmul Saidla,
National Institute of Unani Medicine,
Kottigepalaya, Magadi Main Road,
Bengaluru-560091

Abstract

Present study has been designed to study *Safoofe Deedan* on certain physicochemical parameters in order to determine its quality standards. The method mentioned in National Formulary of Unani Medicine (NFUM) Part II was followed for the preparation of *Safoofe Deedan*. Physicochemical parameters such as organoleptic properties, powder characterization, extractive value, ash value, loss of weight on drying at 105°C pH value and TLC were investigated.

Physicochemical and powder characterization standards were set in. Non successive extractive values were found to be 4.02±0.1129, 6.13±0.200, 15.42±0.3645 and 11.00±0.44090 in petroleum ether, chloroform, ethyl alcohol and water, respectively. Total ash, water soluble ash and acid insoluble ash were determined to be 5.80±0.0288, 2.21±0.1424 and 1.39±0.0781. Loss of weight on drying was 2.033±0.03712 and the pH at 1% and 10% solution was recorded to be 6.74±0.04842 and 6.04±0.0318, respectively. R_f value was calculated from TLC profile which has been shown in Table 6. These findings may be used to determine the quality of *Safoofe Deedan*.

Keywords: Physicochemical, Standardization, Unani, *Safoofe Deedan*, Anthelmintic.

Introduction

Safoofe Deedan is an important drug of Unani medicine. It has been discussed to possess anti-helmintic effect and useful in all three types of intestinal worms, as it either kills them or facilitate their removal from the gut (Anonymous, 2011; Kantoori, 1889). Safoof (Powder) as a dosage form has certain advantages as it has been attributed to have flexibility of compounding, chemical stability, rapid dispersion of ingredients etc. However, there are certain disadvantages that have been associated with powdered drugs (Connor *et al*., 2005). These include unpleasant tasting, hygroscopic and deliquescent nature and shorter shelf life etc. Therefore, the powdered drugs should be standardized more carefully because their kinetic and dynamic profile may alter quickly because of their peculiar nature. Despite the fact that *Safoofe Deedan* is a pharmacopoeial preparation and is in use since decades, it has not been standardized on physicochemical parameters. Therefore, present study was undertaken to set its physicochemical standard so as to establish its quality. The pharmacological activity and dose response relationship can only be ascertained only if the quality of the drug is ensured.

*Author for correspondence

Materials and Methods

Collection of Drugs and its identification

The ingredients of *Safoofe Deedan* viz. Afsanteen Roomi, Baobarang, Turbud Safaid, Gule Surkh and Sate Ajwain (Table 1) were procured from the Unani Pharmacy of NIUM and A.B. General Store, Avenue Road, Bangalore, and identified by the experts of Unani Medicine and Botany.

Method of preparation of *Safoofe Deedan*

The method mentioned in National Formulary of Unani Medicine (NFUM) Part II was followed for the preparation of *Safoof Deedan* (Anonymous, 2007). All the ingredients except *Sate Ajwain* were first dried in shade and then powdered separately in a mixer grinder and passed through 80 no. sieve. *Sate Ajwain* was powdered manually by *kharal* and passed through 80 no. mesh. All these powdered drugs were mixed together in a mixer grinder and properly stored in air tight container (Fig. 1).

Table 1: Ingredients of *Safoofe Deedan*

S.No.	Unani Name	Botanical Name	Quantity
1.	Afsanteen Roomi	*Artemisia absinthium Linn.*	200 g.
2.	Baobarang	*Embelia ribes Burm f.*	200 g.
3.	Turbud Safai	*Ipomoea turpethum* R.Br	200 g. Hollow Root
4.	Gule Surkh	*Rosa damascena* Mill	200 g. petals
5.	Sate Ajwain	*Trachyspermum ammi* (L.) Sprague	10 g.

Fig. 1: *Safoofe Deedan*

Physiochemical parameters

Organoleptic properties: Appearance, Colour, Smell, Taste were evaluated (Anonymous, 2006).

Powder characterization

Bulk density: The volume of the packing was determined by taking a known weight of powder of *Safoofe Deedan* and carefully poured into a long measuring cylinder then the volume corresponding to top level of the sample in the cylinder was noted and the bulk density was calculated using specific formula. (Bulk Density = Mass / Bulk Volume (Anonymous, 2014)).

Tapped density: Powder of *Safoofe Deedan* was carefully poured into a long measuring cylinder and subjected to 500, 750 and 1250 tapping's until constant tapped volume was not obtained, the volume corresponding to top level of the sample was noted and bulk density was calculated by dividing the mass by tapped volume (Anonymous, 2014).

Compressibility index: This method is also used to evaluate the flowability of the powder sample and the rate at which it packs down. For Carr's index same process was fallowed as in Tapped density and was calculated by the following equation (Anonymous, 2014; Manjula *et al.,* 2012).

$$\text{Carr's index (\%)} = \frac{(\text{Unsettled apparent volume} - \text{Final tapped volume}) \times 100}{\text{Unsettled apparent volume}}$$

Hausner's ratio: It is well known that particle size influences flowability. The fine particles below 100 ìm tend to be more cohesive and therefore less free-flowing, whereas larger denser particles tend to be free flowing. Hence the Hausner's ratio and compressibility index are both measure to evaluate the flowability of the powder substances. Hausner's ratio is related to inter particle friction and as such can be used to predict the powder flow properties. For Hausner's ratio same process was followed as in Tapped density and it was calculated by the following equation (Anonymous, 2014; Manjula *et al.,* 2012).

$$\text{Hausner's ratio} = Vo/Vf$$

Vo = Unsettled apparent volume V_f = final tapped volume

Angle of repose: Angle of repose was calculated by fixed funnel and free standing conc. method. On a flat horizontal surface a funnel was clamped with its tip 2 cm above a graph paper. The powders were poured through the funnel carefully until the cone formed by powder just reached the tip of the funnel. The mean diameter of the powder cones were noted and angle of repose was calculated by using following formula (Manjula *et al.,* 2012 and Musa *et al*., 2011).

Tan Ø =2h / D [h = Height of powder (from graph paper to tip of funnel), D = Mean diameter of the powder]

Non successive extractive value: The powder was extracted by Soxhlet apparatus separately in different solvents (petroleum ether, chloroform, ethyl alcohol and water). 10 g. powdered drug was taken and subjected to separate extraction with each solvent. The extracts were filtered using filter paper (Whatman No. 1) and evaporated on water bath. Extractive values were determined with reference to total drug taken (w/w) (Agrawal and Paridhavi, 2007)

Ash value

Total ash: 5 g. accurately weighted powdered drug was taken in a tarred silica dish and incinerated in Muffle furnace (Optics technology Sr.no. 3163) at a temperature not exceeding 450°C until free from carbon. It was then cooled and weighted and the percentage was calculated with reference to ground drug (Anonymous, YNM).

Acid insoluble ash: Ash obtained from above method was boiled for 5 minutes with 25 ml of dilute hydrochloric acid. Insoluble matter was collected on an ashless filter paper (Whatman 41) and washed with hot water, and ignited in Muffle furnace at a temperature not exceeding 450°C to constant weight. Residue was allowed to cool in desiccator for 30 minutes and weighed without delay. The percentage of acid insoluble ash was calculated with reference to the air dried drug (Anonymous, 2009).

Water soluble ash: Ash obtained from above method was boiled for 5 minutes with 25 ml of water. Insoluble matter was collected on an ashless filter paper and washed with hot water. It was then ignited for 15 minutes at a temperature not exceeding 450°C in Muffle furness and weighed. Weight of insoluble matter was subtracted from weight of ash, difference in weight represented water soluble ash. The percentage of water soluble ash was calculated with reference to air dried drug (Anonymous, YNM).

Loss of weight on drying at 105°C: In tarred evaporating dish, about 10 g. of drug was taken and dried in oven (Labline mod. no. HO 6.7) at 105°C for 5 hours and weighed. Drying and weighing was continued at one hour interval until difference between two successive weighing corresponded to not more than 0.25%. Two consecutive weighing after drying for 30 minutes and cooling for 30 minutes in a desiccator, show not more than 0.01 g difference, until constant weight was reached. The % loss of weight was calculated with reference to original weight of the drug (Anonymous, 2009).

pH value

pH value of 1% solution: Accurately weighed 1 g. of *Safoofe Deedan* powder was taken and dissolved in accurately measured 100 ml of water, pH of filtrate was measured with standard glass electrode (Anonymous, 2006).

pH value of 10% solution: Accurately weighed 1 g. of powder of *Safoofe Deedan* was dissolved in accurately measured 10 ml of water, pH of filtrate was determined with pH meter (Eutech instrument Sr.no. 1544421) (Anonymous, 2006).

Thin layer chromatography: TLC pre-coated plates of silica gel 60 F 254 (layer thickness 0.25 mm) on aluminium sheets was used. TLC test was carried out on these pre coated plates for pet. ether, chloroform and ethanol extract of *Safoofe Deedan*. Two different mobile phases used were Chloroform: Methanol (9:1), Toluene: Ethyle acetate: Formic acid (5:4:1) for each extract. The plates were examined under U V light (254nm) to detect the spots. After detecting spots Rf value was calculated by the following formula (Anonymous, 2009)**:**

Rf value = Distance travelled by the spot / Distance travelled by mobile phase

Results

Organoleptic properties: Appearance: Fine powder, Colour: Brown, Smell: Pleasant, Taste: Bitter Powder characterization: The mean values of Bulk Density, Tapped Density, Compressibility index and Hausner´s ratio of powder of *Safoofe Deedan* were found to be 0.2604±0.0015, 0.4550±0.0015, 23.873±0.081 and 1.308±0.0041, respectively (Table 2). Angle of repose was found to be 39.69±0.356 (Table 2).

The mean percentage of the non successive extractive values was found to be 4.02±0.1129, 6.13±0.200, 15.42±0.3645 and 11.00±0.44090 in petroleum ether, chloroform, ethyl alcohol and water respectively (Table 3). The mean percentage of the values of total ash, water soluble ash and acid insoluble ash were found

Table 2: Powder characterization

Sr.No.	Parameters	Mean± SEM Value
1.	Bulk Density (gm/ml)	0.2604±0.0015
2.	Tapped Density (gm/ml)	0.4550±0.0015
3.	Compressibility index (%)	23.873±0.081
4.	Hausner´s ratio	1.308±0.0041
5.	Angle of repose	39.69±0.356

to be 5.80±0.0288, 2.21±0.1424 and 1.39±0.0781 respectively (Table 4). The mean % age value of Loss of weight on drying was found to be 2.033±0.03712 (Table 3). The mean value of pH was determined at 1% and 10% solution and was found to be 6.74±0.04842 and 6.04±0.0318, respectively (Table 4).

TLC Study

Mobile phase: Benzene: Chloroform (4:1) 5 spots in chloroform (Rf values 0.033, 0.050, 0.080, 0.118,0.542); 5 spots in petroleum ether (Rf values 0.050, 0.067, 0.107, 0.135, 0.559); and 3 spots in ethanol (Rf values 0.118, 0.237, 0.542) (Table 5) (Fig. 2)

Table 3: Extractive values

Sr.No.	Solvents	Non-successive Extractive values (%) (Mean± SEM)
1.	Petroleum ether	4.02±0.1129
2.	Chloroform	6.13±0.200
3.	Ethyl alcohol	15.42±0.3645
4.	Water	11.00±0.44090

Table 4: Physicochemical Parameters

Sr.No.	Physicochemical Parameters	Mean± SEM
1.	Total ash (%)	5.80±0.0288
2.	Water soluble ash (%)	2.21±0.1424
3.	Acid insoluble ash (%)	1.39±0.0781
4.	Loss of weight on drying (105^{o}) (%)	2.033±0.03712
5.	pH value at 1% 10%	 6.74±0.04842 6.04±0.0318

Table 5: TLC Mobile phase = Benzene: Chloroform (4:1)

Sr.No	Extract	Treatment	No. of spot	Rf value
1.	Chloroform	Iodine vapours	5	0.033, 0.050, 0.080, 0.118, 0.542
2.	Petroleum ether	Iodine vapours	5	0.050, 0.067, 0.107, 0.135, 0.559
3.	Ethanol	Iodine vapours	3	0.118, 0.237, 0.542

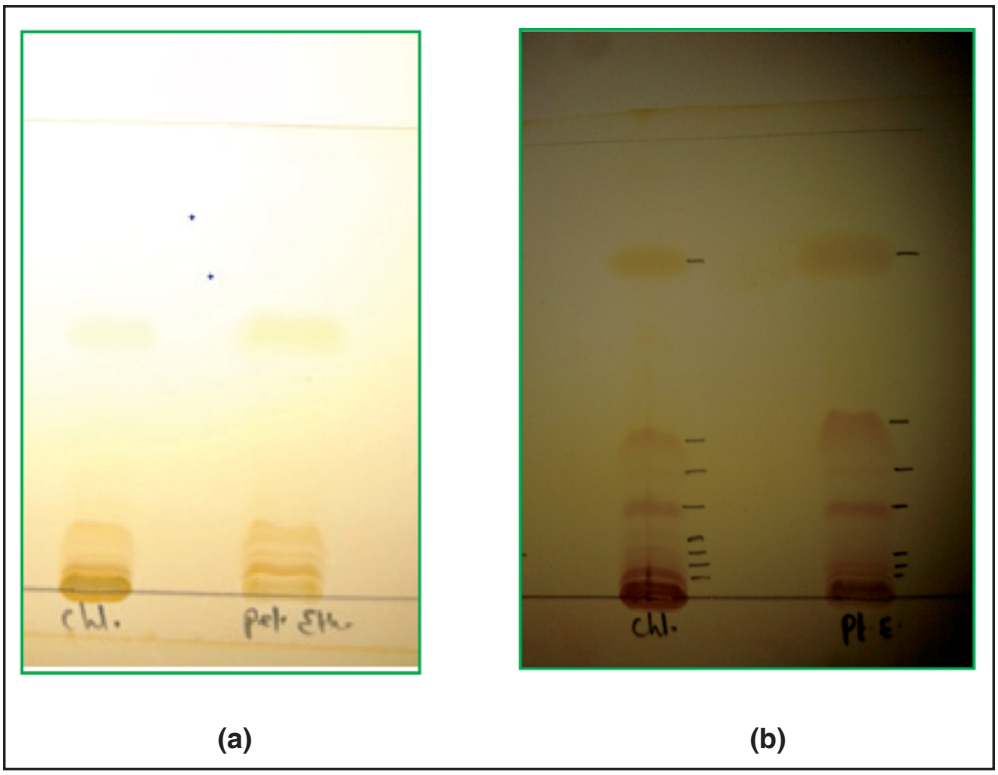

Fig. 2: (a) TLC for chloroform and pet. ether extract in mobile phase Benzene: Chloroform (4:1),
(b) TLC for chloroform and pet. ether extract in mobile phase Toluene: ethyle acetate (9:1)

Mobile phase- Toluene : Ethyl acetate (9:1) 8 spots in chloroform Rf vlues 0.050, 0.066, 0.10, 0.133, 0.20, 0.266, 0.333, 0.733; 7 spots in petroleum ether Rf values 0.050, 0.080, 0.10, 0.183, 0.283, 0.383, 0.733 and 5 spots in ethanol Rf values 0.050, 0.116, 0.150, 0.30, 0.35 (Table 6) (Fig. 3).

Table 6: TLC Mobile phase = Toluene: Ethyl acetate (9:1)

Sr.No.	Extract	Treatment	No. of spot	Rf value
1.	Chloroform	Iodine vapours	8	0.050, 0.066, 0.10, 0.133, 0.20, 0.266, 0.333, 0.733
2.	Petroleum ether	Iodine vapours	7	0.050, 0.080, 0.10, 0.183, 0.283, 0.383, 0.735
3.	Ethanol	Iodine vapours	5	0.050, 0.116, 0.150, 0.30, 0.35

Discussion

Organoleptic features such as appearance, colour, smell and taste plays an important role in quick identification of the drug and these characteristics are peculiar with each drug and provide a qualitative index of identity and quality. The features as observed in respect of *Safoofe Deedan* will also be helpful to determine its identity and quality. Extractive value of a drug in specific solvent is an index of purity of a drug and plays an important role to find out adulteration, if any. The amount of drug soluble in a particular solvent is an index of its purity (Jenkins *et al.,* 2008). Ash value is a significant parameter for finding of adulteration and impurities. Loss of weight on drying indicates the amount of water and volatile substances present in a particular drug. A drug becomes ideal medium for growth of different types of bacteria and fungi if it has moisture. These bacteria and fungi affect the purity, quality and efficacy of drug. pH determines the absorbability of oral dosage forms as with increase and decrease in pH level the ability of drug to get absorbed is altered (Goodman and Gilman, 2001). Altered number of spots and Rf value in a particular mobile phase is an index of purity and quality of a drug and plays an important role to find out adulteration in the drug. The data generated in respect of physicochemical standardization such as bulk density, tapped density, compressibility index and Hausner´s ratio, angle of repose, loss of weight on drying, pH, total ash, water soluble and acid insoluble, extractive values, TLC may be used as standard for future reference to ensure the quality standards of *Safoofe Deedan*.

References

Agrawal, S.S. and Paridhavi, M., 2007. Herbal Drug Technology Part II. Hyderabad: Universities Press; p. 326.

Anonymous, 2009. The Unani Pharmacopeia of India, Part II. Vol.1st. New Delhi: Ministry of H & FW, Govt. of India, Dept. of AYUSH, Govt. of India; p. 146-150

Anonymous, 2006. Physiochemical Standardization of Unani Formulation, Part VI. CCRUM, Ministry of H & FW, Govt. of India, New Delhi, p.142-145.

Anonymous, 2011. National Formulary of Unani Medicine (Urdu Edition). Vol. VIth. CCRUM, Ministry of H & FW, Govt. of India, New Delhi, pp. 98, 102

Anonymous, 2014. Bulk Density*, TB, HR, CI, AR.* Available from URL: http://www.pharmacopeia. cn/v29240/usp29nf24s0_c616.html [Cited on 2014 Feb 13]

Anonymous, YNM. Protocol for Testing of Ayurvedic, Siddha & Unani Medicines. PLIM, Ghaziabad, Dept. of AYUSH, Govt. of India, p. 49

Connor, R.E., Schwartz, J.B., Felton LA., 2005. Powders In: Beringer P, *et al*. Remington: The science and practice of pharmacy. Vol. I, 21st Edition. B.I. Publication, London, p.716, 711,712, 916-918, 986.

Goodam and Gilman, 2001. The Pharmacological Basis of Therapeutics. 10th ed. Mc Graw –Hill, Medical Public Division, USA, p. 5.

Jenkins, G.L., Knevel, A.M., Digangi, F.E., 2008. Quantitative Pharmaceutical Chemistry. 6th ed. CBS Publishers and Distributors, New Delhi, pp. 229,225.

Kantoori, G.H., 1889. (Urdu translation), Majusi A., (d. 994 A.D.). Kamilus Sana. Vol. 1. Matba Munshi Naval Kishore, Lucknow, pp.515-516

Manjula, S., Shashidhara, S., Anita S., Shilpa S., 2012. Design Development and Evoluation of Herbal Tablets Containing *Andrographis paniculata* and *Phyllanthus amarus*. Pharma Science Monitor An *International Journal of Pharmaceutical Science* 3(4): 2352-2362.

Musa A., Adamu B.I., Teriyila S.A. and Musa I., 2011. Use of Hydrophobic Fumed Silica and Selected Binders in the Tablet Formulation of a Deliquescent Crude Plant Extract: *Vernonia galamensis* (Asteraceae). *Journal of Pharmaceutical and Biomedical sciences* 6(13): 2.

Nuka R., Potu A.R., Nandan N.R., 2012. Formulation development and invitro evaluation of ramipri micropellets. *World Journal of Pharmaceutical Research* 2(1): 76-87