Physico-chemical Study of a Unani Antipruritic Formulation: Safoof Kharish

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Abstract

afoof Kharish is a compound formulation of Unani Medicine which acts as *Daf-e-Ufoonat* (antiseptic) and *Jali* (detergent), applied topically to treat *Kharish* and *Hikka* (pruritus). It contains one herbal drug i.e. Kamila (*Mallotus philippinensis*) and three mineral drugs i.e. Gandhak (*Sulphur*), Safaida Kashghari (*Zinc oxide*) and Murdar Sang (*Letharge*). Until now no physico-chemical standards are available to assess the quality of the formulation. Therefore standardization of the finished product was done by evaluating the relevant organoleptic and physicochemical parameters like pH, ash values, extractive values, bulk density, tapped density, compressibility index, hausner's ratio, angle of repose, TLC etc. The physicochemical characteristics obtained from this study will be helpful for quality evaluation and to set the Pharmacopoeial standards for *Safoof Kharish*.

Keywords: *Kamila*, Physicochemical properties, *Safoof Kharish*, Unani Medicine

Introduction

To rescue man from the clutches of disease several system of medicine practised in the world, every system with its own basis, philosophy and therapeutics, but with one common object i.e. alleviation of disease (Said, 1997). During the last decade, use of traditional medicine has expanded globally and has gained popularity. Unani System of Medicine is one among them which was originated in Greece. The great Greek Philosopher & Physician Hippocrates (460 - 377 B.C.) is the founder of Unani Medicine, later Galen, Rhazes and Avicenna enriched the System (Anonymous, 2007). World Health Organisation (WHO) pays attention to herbal medicinal products by bringing out monographs, guidelines on Quality control and on research methodologies in traditional medicine. India has made rules for Good Manufacturing Practices (GMP) on traditional Ayurveda, Unani and Siddha (ASU) products. Standard operating procedures and standards of raw and finished products are mentioned in pharmacopoeias and formularies of ASU (Venkat *et al.*, 2010). Standard of any drug relate to the uniformity in quality. A standard may be reflected by descriptive or numerical values obtained through assessment of quality by a given protocol (Venkat *et al.,* 2010). Standardization of herbal formulations is an essential factor in order to assess the quality, purity, safety and efficacy of drugs based on the concentration of their active principles. Evaluation of physicochemical properties of the formulation is

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essential for the assessment of the quality, that is, ash values determination, such as total ash, water-soluble ash and acid-insoluble ash. The extractive values of the formulation are also performed to ensure the presence of plant actives and their solubility profile.

Safoof are the fine powder forms of medicinal preparations made of plant, animal and mineral origin drugs. These are used internally as well as externally (Chaudhary *et al.,* 2013). *Safoof Kharish* is an externally used fine powder, which acts as *Daf-e-Ufoonat* (antiseptic) and *Jali* (detergent) (Anonymous, 2011) widely used to treat *Kharish* and *Hikka* (pruritus). It is advised to apply locally on the skin after mixing sufficient quantity of powder with jasmine oil. It is a Pharmacopoeal preparation, composed of one herbal drug i.e. Kamila (*Mallotus philippinensis*) and three mineral drugs i.e. Gandhak (*Sulphur*), Safaida Kashghari (*Zinc oxide*) and Murdar Sang (*Letharge*). *Kamila* is used to treat acne, scabies, pruritus and helminthiasis (Anonymous, 2007). *Gandhak* is applied externally in the form of paste for ringworm, scabies and other parasitic diseases (Nadkarni, 2009). *Murdar Sang* is a powerful local astringent, cooling and an insecticide; it is used externally as ointment for baldness, itching, ulcers, acne, eczematous eruptions and other skin diseases (Nadkarni, 2009). As *Safaida* have soothing action on skin therefore is used externally to treat burns and various eye diseases (Kabiruddin, 2007).

Because of the rapid progress of the herbal drug industry in India for the last quarter century, an increasing need is felt to standardize the Unani products. It is necessary to develop the scientific protocols such as SOP and pharmacopoeial standards of Unani formulations. Therefore, in the present study, physicochemical evaluation of the Unani formulation *Safoof Kharish* has been carried out because these evaluations are surprisingly uncharted till date and determination of these parameters are very essential to assure the quality, safety, and efficacy of this formulation.

Materials and Methods

Materials

Kamila (*Mallotus philippinensis*), Gandhak (*Sulphur*), Safaida Kashghari (*Zinc oxide*) and Murdar Sang (*Letharge*) were purchased from the local market of Bangalore. All the reagents and solvents used were of analytical grade.

Preparation of formulation

The formulation was prepared as prescribed in National Formulary of Unani

Medicine (NFUM) part-VI. Twenty grams of each ingredient namely, kamila (*Mallotus philippinensis*), gandhak (*Sulphur*), safaida kashghari (*Zinc oxide*) and murdar sang (*Letharge*) were weighed accurately and made into fine powder separately by passing through sieve no. 120 (Table 1). The powders were mixed in a mortar, again passed through 120 no. sieve and packed in plastic containers (Fig.1).

Determination of pH in 1% solution and 10% solution

An accurately weighed one gram and ten gram powder was dissolved separately in 100 ml distilled water, filtered and pH was measured using a digital pH meter (Anonymous, 1986).

Determination of loss on drying

The percentage loss on drying (%LOD) was determined for *Safoof Kharish* gravimetrically in which 5 g of accurately weighed air-dried material was placed in a previously dried and tared petriplate. The sample was dried in an oven at 100°C–105°C until two consecutive weighing did not differ by more than 5 mg (Afaq *et al.,* 1994).

Bulk density

Bulk Density is the ratio between the given mass of a powder and its bulk volume. 20gm of the powder is dried and filled in a 50 ml measuring cylinder. Carefully level the powder without compacting, and read the unsettled apparent volume to the nearest graduated unit. This is repeated to get average values. The Bulk Density was calculated in g per ml by using the formula (Halith *et al.,* 2009).

$$\text{Bulk Density} = \frac{\text{Mass}}{\text{Bulk Volume}}$$

Tapped density

The tapped density is an increased bulk density attained after mechanically tapping a container containing the powder sample. 20gm of the powder is dried and filled in a 50 ml measuring cylinder. Powder sample was tapped for 500 and 750 taps and the corresponding volumes were noted. The powder was subjected to tapping until the difference between succeeding measurements is less than two ml. The Tapped Density was calculated in g per ml by using the formula (Lachman *et al.,* 1991).

$$\text{Tapped Density} = \frac{\text{Mass}}{\text{Tapped Volume}}$$

Carr's index (or) % compressibility

It indicates powder flow properties. It is expressed in percentage and is calculated by the following formula (Moiz *et al.,* 2011).

Carr's Index (%) = [(Tapped density – Bulk Density) / Tapped Density] × 100

Hausner ratio

Hausner ratio is an indirect index of ease of powder flow. It is calculated by the following formula (Moiz *et al.,* 2011).

Hausner's Ratio = Tapped density / Bulk Density

Angle of repose

The fixed funnel method was employed to measure the angle of repose. A glass funnel was secured with its tip at a given height (h), above the graph paper that is placed on a flat horizontal surface. The powder was carefully pored through the funnel until the apex of the conical pile just touches the tip of the funnel. The radius (r) of the base of the conical pile was measured. The angle of repose (θ) was calculated using the following formula. Values for angle of repose ≤ 30 usually indicate free flowing material and angle ≥ 40 suggested a poor flowing material (Subrahmanyam, 2009).

$$\theta = \tan^{-1} (h/r)$$

Determination of ash values

Total ash

Two grams of the powdered material was accurately weighed and placed in a previously ignited and tared silica crucible. The material was ignited to a temperature of 500–600°C until free from carbon, cooled and weighed and the percentage was calculated in mg/g of powder drug (Anonymous, 2007).

Acid-insoluble ash

Total ash was boiled gently with 25ml of dilute hydrochloric acid for five minutes. 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 in mg/g of powder drug (Anonymous, 2007).

Water soluble Ash

Total ash was boiled with 25 ml of distilled water for five minutes. 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 in mg/g of powder drug (Anonymous, 2007).

Determination of Extractive values

The extractive values were recorded in water, alcohol, petroleum ether and chloroform separately by percolation in soxhlet apparatus with a view to study the distribution of various constituents of *Safoof Kharish*. Accurately weighed five gram of coarsely powdered air-dried material was taken and subjected to separate extraction with each solvent. The extracts were filtered using filter paper (Whatman No.1) and after evaporation of the solvents on water bath, the extractive values were determined with reference to the weight of drug. The procedure was repeated three times and the mean value for each extract was calculated (Jenkins *et al.,* 2008).

Thin layer chromatography

Preparation of extracts for TLC: Two gram of *safoof* was soaked in petroleum ether and chloroform separately for 18 hours, refluxed for ten minutes on water bath and filtered. The filtrates were concentrated on water bath and made up to 5ml in a standard flask separately.

Method of developing for TLC: Pet. ether and Chloroform extracts were applied on precoated silica gel 60 F254 TLC plate (Merck Germany) as absorbent and developed the plate using solvent systems, benzene and chloroform in ratio 2:2 v/v in which 3 drops of acetic acid was added. After developing, the plates were dried and exposed to iodine vapours to visualize the spots (Anonymous, 1992).

Table 1 : Ingredients of *Safoof Kharish*

Unani Name	Botanical/English Name	Quantity
Kamila	*Mallotus philippinensis*	20 gm
Gandhak	*Sulphur*	20 gm
Safaida Kashghari	*Zinc oxide*	20 gm
Murdar Sang	*Letharge*	20 gm

Table 2 : Organoleptic characteristics of *Safoof Kharish*

Colour	Light Pink
Odour	Sulphur like
Texture/State	Fine Powder

Table 3 : Physicochemical tests of *Safoof Kharish*

S. No.	Parameters	1	2	3	Mean±SD
1.	pH(1%)	6.56	6.59	6.67	6.6±0.056
2.	pH(10%)	7.53	7.53	7.59	7.5±0.034
3.	Loss of weight on drying (%)	0.803	0.911	0.945	0.886±0.0741
4.	Bulk density(gm/ml)	0.8695	0.8695	0.8695	0.869±6.63
5.	Tapped density(gm/ml)	1.4285	1.4285	1.4285	1.4285±1.32
6.	Carr's index(%)	39.13	39.13	39.13	39.13±0.00
7.	Hausner's ratio	1.6428	1.6428	1.6428	1.6428±0.00
8.	Total ash(%)	70.63	71.91	70.52	71.02±0.772
9.	Acid insoluble ash(%)	44.89	44.32	43.97	44.39±0.464
10.	Water soluble ash(%)	1.26	1.20	1.31	1.25±0.055
11.	Angle of Repose	51°	50°	50°	50.33±0.577
12.	Extractive Values Aqueous	1.36	1.52	1.30	1.393±0.113
13.	Ethanolic	6.56	6.72	6.43	6.57±0.145
14.	Petroleum Ether	2.28	1.90	1.89	2.02±0.222
	Chloroform	3.9	4.1	4.2	4.06±0.152

Table 4 : TLC of *Safoof Kharish*

Extract	Solvent System	Treatment	No. of Spots	R_f Values
Petroleum Ether	Benzene: Chloroform (2:2) + 3 drops of acetic acid	Exposed to Iodine vapours	2	0.83, 0.45
Chloroform	Benzene:Chloroform (2:2) + 3 drops of acetic acid	Exposed to Iodine vapours	4	0.84, 0.43, 0.26, 0.12

Fig. 1: Finished Product *Safoof Kharish*

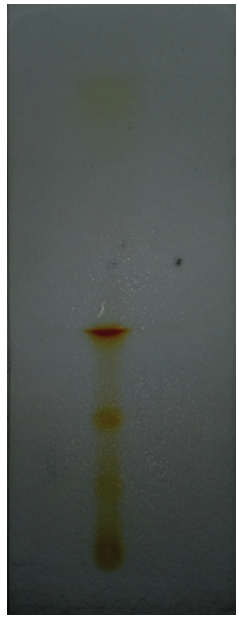

Fig .2 : TLC for Chloroform extract

Fig. 3 : TLC for Petroleum Ether extract

Results and Discussion

Organoleptic characteristics of *safoof kharish* showed light pink colour, sulphur like odour and homogenous fine powder (Table 2). These characteristics might be useful for distinguishing it from its substitutes and adulterants. pH of 1% and 10% solution (w/v) of *safoof kharish* was found to be 6.6±0.056 and 7.5±0.034 respectively. Total ash value of *safoof kharish* was found to be 71.02±0.772%. High content of total ash value may be due to the presence of three mineral origin drugs in formulation, as *kamila* the only organic drug in formulation have total ash value of NMT 6% (Anonymous, 2007). Acid insoluble ash was found to be 44.39±0.464%, it showed that part of total ash which was insoluble in dilute hydrochloric acid. Water soluble ash was found to be 1.25±0.055% (Table 3). Ash values are useful in determining authenticity and purity of drug and in detection of low grade product, excess of earthy matter and exhausted drugs. Loss of weight on drying was found to be 0.886±0.0741%, it tells about the moisture content. The moisture content of the formulation should be minimized in order to prevent decomposition of the formulation either due to chemical change or microbial contamination (Kokate *et al.,* 2010). Extractive values of the formulation in different solvents like water, ethanol, petroleum ether and chloroform was found to be 1.393±0.113%, 6.57±0.145%, 2.02±0.222% and 4.06±0.152% respectively (Table 3). The extracts obtained by exhausting crude drugs are indicative of approximate measure of their chemical constituents (Kokate *et al.,* 2010).The extractive value of the formulation determines the quality as well as purity of the formulation. In the present study extractive values were found to be more in alcohol than other solvents. The bulk density of a powder is the ratio of the mass of an untapped powder sample and its volume including the contribution of the interparticulate void volume. Bulk density of the formulation was found to be 0.8695±6.63 gm/ml (Table 3). The bulk density can be use as a quality control measure, used to check the uniformity of bulk chemicals and in selecting the proper size of a container and packing material (Subrahmanyam, 2009). On the other hand tapped density is an increased bulk density attained after mechanically tapping a container containing the powder sample. Tapped density of the formulation was found to be 1.428±1.32 gm/ml (Table 3). Compressibility index and Hausner ratio are measures of the tendency of a powder to be compressed and also indicate the flow property. Values of Carr's index ≤ 10% indicate excellent flow and > 38% indicate very poor flow property (Table 3). Similarly values of Hausner's ratio 1.00-1.11 indicate excellent flow and > 1.60 indicate very poor flow property (Nuka *et al.,* 2012). Values of Carr's index and Hausner's ratio for *safoof kharish* were found to be 39.13±0.00% and

1.642±0.00 respectively and showed poor flow property of the formulation. Flow characteristics are also measured by angle of repose. Values for angle of repose ≤ 30º indicate a free flowing material and angles ≥ 40° suggest a poorly flowing material. In the present study increased angle of repose i.e. 50°.33'±0.577 was found which showed very poor flow property of the formulation (Table 3). It is due to the small particle size (passed through 120 mesh). The TLC of *Safoof Kharish* (petroleum ether and chloroform extract) was developed in Benzene and Chloroform in ratio 2:2 v/v solvent system in which three drops of acetic acid was added (Fig. 2&3). Plates exposed to iodine vapours showed two spots with R_f values 0.83, 0.45 for petroleum ether extract and four spots with R_f values 0.84, 0.43, 0.26, 0.12 for chloroform extract (Table 4). TLC is one of the important parameter used for detecting adulteration and judging the quality of drugs.

Conclusion

Among the various Unani formulations *Safoof Kharish* is one which is widely use in common practice but still its official physicochemical standards are not present. Therefore an attempt has been made to establish the scientific basis of the formulation. The physicochemical characteristics obtained may be used for quality evaluation and the standardization of the compound formulation *Safoof Kharish*. These explorations will definitely help to set a standard for this traditional medicine. Further analytical and other studies may be conducted to make its complete monograph.

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References

Afaq, S.H., Tajuddin, Siddiqui M.M.H., 1994. Standardization of Herbal Drugs. A.M.U. Press, Aligarh, p. 41.

Anonymous, 1986. Physicochemical standards of Unani formulations, Part I. CCRUM, Ministry of Health & Family Welfare, Govt. of India, New Delhi, p. A50.

Anonymous, 2007. The Unani Pharmacopoeia of India. Part-I, Vol 1. CCRUM, Ministry of Health & Family Welfare, Govt. of India, New Delhi, p. 17, 45, 109.

Anonymous, 2011. National Formulary of Unani Medicine, Part VI. CCRUM, Ministry of Health & Family Welfare, Govt. of India, New Delhi, p. 104.

Anonymous, 1992. Standardization of single drugs of Unani medicine, Part-II. CCRUM, Ministry of Health & Family Welfare, Govt. of India, New Delhi, p. 201.

Chaudhary, S.S., Tariq, M., Zaman, R., Imtiyaz, S., 2013. Solid dosage forms in Unani system of Medicine. *Journal of Pharmaceutical and Scientific Innovation* 2(3):17-22.

Halith, S.M., Abirami, A., Jayaprakash, S., Karthikeyini, C., Pillai, K.K., Firthouse, P.U.M., 2009. Effect of *Ocimum sanctum* and *Azadirachta indica* on the formulation of antidandruff herbal shampoo powder. *Scholar Research Library* 1 (2): 68-76.

Jenkins, G.L., Knevel, A.M., Digangi, F.E., 2008. Quantitative Pharmaceutical Chemistry, 6th ed. CBS Publishers, New Delhi, p.235.

Kabiruddin, M., 2007. Makhzanul Mufardat yani Kitab ul Advia. Idarah Kitab ul Shifa, New Delhi, p. 346.

Kokate, C.K., Purohit, A.P., Gokhale, S.B., 2010. Pharmacognosy. Nirali Prakashan, Pune, 45th ed., p.6.19-6.22.

Lachman, L., Liberman, H.A., Kanig, J.L., 1991. The Theory and Practice of Industrial Pharmacy. Varghese Publishing House, Mumbai, India, 2nd ed, p. 183.

Moiz, M., Srinivas, M.P., Sadanandam, M., 2011. Formulation and evaluation of bilayered tablets of montelukast and levocetrizine dihydrochloride Using natural and synthetic polymers. *International Journal of Drug Delivery* 3 (4): 597-618.

Nadkarni, K.M., 2009. Indian Materia Medica. Popular Prakashan Private Limited, Mumbai, Vol. 2nd, 3rd ed, p.86, 122.

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

Said, M., 1997. Hamdard Pharmacopoeia of Eastern Medicine. Sri Satguru Publications, Delhi, p. 7.

Subrahmanyam, C.V.S., 2009. Text book of Physical Pharmaceutics. Vallabh Prakashan, 2nd ed., Delhi, pp. 216, 222-224

Venkat, P., Sudha, P., Lalitha., 2010. Quality control of Indian traditional medicines. Institute of Ayurveda and Integrative Medicine (I-AIM), Bangalore, pp. 8,15.