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PHARMACOPOEIAL AND MICROSCOPICAL STANDARDIZATION OF AN IMPORTANT UNANI FORMULATION-QURS-E-ISQEEL

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ABSTRACT

The people worldwide are adopting herbal medicines as alternative health care system. Their reliance on herbal medicines has led to remarkable growth of tradition system of medicine. The preparation of herbal formulation is usually based on traditional methods in accordance with the procedure described in classical literature which sometimes lacks scientific validation. It is utmost important to scientifically standardize the method of preparation and physico-chemical parameters to ensure their authenticity and therapeutic effects. Therefore, a herbal Unani formulation Qurs-e-Isqeel widely known for its action as diuretic and antidote and therapeutically used in poisoning, fracture, ascites and dysuria. The drug was studied through various standardization parameters such as organoleptic evaluation (colour, Odor and taste), physico-chemical evaluations (moisture content, total ash, acid insoluble ash, pH values, water and ethanol soluble extractive, volatile oil) and microscopy. The evaluations of WHO parameters such as heavy metals, aflatoxins, pesticide residue and microbial contamination were also carried out to ascertain the quality of drug. The data revealed the present work will help in developing pharmacopoeial standards for maintaining the quality and batch to batch consistency of Qurs-e-Isqeel.

KEYWORDS: SOP, Microscopy, Pharmacopoeial parameters, Isqeel, Quality control.

INTRODUCTION

Around the world, there has always been a demand for using traditional medicines as an alternative and complementary medicines have always existed. World health organization (WHO) also addresses and boost up the exploration of traditional drugs by virtue of their easy availability and affordability.[1] The government of India has formed structure to regulate quality, safety, efficacy practice and documentation of herbal medicines (National policy of Indian System of Medicine and Homoeopathy-2002).[2]

Qurs-e-Isqeel is a herbal Unani formulation categorized as Qurs under Aqras section listed in the national formulary of Unani medicine, Part-1.[3] Qurs can be prepared by grinding the crude drugs with the fine powder. Then this fine powder was mixed with a suitable solvent to make lubdi mass. Then flat, circular and uniformly shaped tablets are prepared from this lubdi mass weigh from one tenth of gram to one gram. The finished products dried and stored in a dried container. Qurs should neither be very hard nor very soft.[4]

Qurs-e-Isqeel is an important Unani formulation reputed for its Dafae sumoom (antidote) and Mudirr-e-boul (diuretic) action and therapeutically used in Tasammum (poisioning), Kasr-e-Izaam (fracture), Usr-e-Tanaffus (dyspnoea) and Istisqa (ascites).[5,6] Qurs-e-Isqeel is composed of two herbal ingredients which individually has magnificent medicinal properties. The main ingredients Isqeel (jangli piyaaz) is eminent for its action as anti-inflammatory, ulcerative, deobstruent, eye tonic, stomachic, diuretic, purgative and sexual tonic and therapeutically used in epilepsy, melancholia, migraine, tremor, amnesia, neuralgia, cataract, halitosis, asthma, dyspnoea, haematemesis, ascites, dysuria, kidney and bladder stone, hysteria, leprosy and pruritus.[7]

The present research work aims to develop quality parameters and evaluate the data to laid down pharmacopoeial standard of Qurs-e-Isqeel. The conventional parameters such as organoleptic parameters, microscopy and physico-chemical evaluation were carried out. The WHO quality control parameters such as heavy metals, aflatoxins, pesticidal residue and microbial contamination were also analysed in order to determine the quality of the formulation.[8]

MATERIALS AND METHODS

Preparation of drugs

All the ingredients were procured from local raw drug dealer and were identified botanically using pharamcognostical methods. The ingredients were further validated by comparing with the monographs available in UPI, API and IP.[9]

All the ingredients were taken of pharmacopoeial quality (Table 1). The ingredients were cleaned and dried under shade to remove the moisture if any. The ingredients no. 1 *(Isqeel)* was covered with any flour paste and roasted in an oven. After roasting it was crushed and flour coating was removed. This roasted Isqeel was grinded and mixed with equal amount of ingredient no. 2 *(Arad-e-Karsana).* The coarse powder was further grinded in a mixer grinder to get the fine form. The fine powder was mixed thoroughly and sieved through mesh no. 80. *Sharab-e-Musallas* was added to the mixture and again mixed thoroughly to obtain the *lubdi* (mass). The Qurs were prepared from the *lubdi* (mass) and dried under shade. The prepared Qurs were stored in tightly closed glass container free from moisture and kept in a cool and dry place.[10]

Microscopy

5g of the powdered drug was taken and stirred gently with hot water in a beaker. The supernatant was discarded and the residue was washed with distilled water. A little residue was stained with iodine solution and mounted in 50% glycerine. Some of the residue was heated in chloral hydrate solution and mounted in 50% glycerine and a little residue was boiled in 2% potassium hydroxide solution, washed with distilled water and mounted in 50% glycerine.[11,12]

Physico-chemical Analysis

The physico-chemical parameters of Qurs-e-Isqeel such as removal of foreign matters, moisture contents, extractive values (solubility in water, ethanol and hexane), ash values (total ash and acid insoluble ash), pH values (1% and 10% aqueous solution) and volatile oil estimation were analyzed by standard methods.[13,14]

Quality control Analysis

Quality control parameters like microbial load, heavy metals, aflatoxins and pesticidal residues for the samples of the drug were undertaken and analysed. The microbial load estimation was carried out as per the guidelines. Heavy metal analysis was done by atomic

absorption spectrophotometer. Analysis of aflatoxins was performed by HPLC method. Pesticide residues were analysed using GC-MS (Thermo) instrument.[15,16,17,18,19and 20]

RESULTS AND DISCUSSION

Observations

Qurs-e-Isqeel is a greenish brown tablet with slightly unpleasant odour and bitter taste. The drug did not show any filth, fungus or objectionable matter while the sample was spread in a petri dish (Fig. 1). On examination under the microscope, following cells/tissues/cell contents were observed.

Microscopic observation

Numerous identifying characteristics of ingredients of the drug were observed under microscope in various mounts (Fig. 2). Abundant of acicular calcium oxalate crystals, mucilage cells and spiral or annular vessels with lignified walls found under the microscope which are noticeable identifying microscopic characters of *Jangli Piyaz*. Beside this various mount show the presence of other characters of *Jangli Piyaz* such as parenchyma cells, few fibres with tapering ends, mucilage cells with acicular calcium oxalate crystals. Pseudo-compound starch granules confirm the presence of *Arad-e-Karsana*. Fragments of cotyledon filled with starch grains of *Arad-e-Karsana* are also seen in some mounts.

Physico-chemical Analysis

The physico-chemical data of the drug Qurs-e-Isqeel are shown in Table 2. The extractive values show that the solubility of phytoconstituents of the drug was more in water (7.85-8.34 %) and less amount of phytoconstituents are soluble in alcohol (1.04-1.28 %) and Hexane (0.44-0.51%). The moisture content in drug was low as the loss in weight on drying at 105 ^{o}C occurred below 10%. Quantitative standards revealed the presence of negligible amount of siliceous matter in the sample because the total ash content was found to be (2.12-2.244 %) and acid insoluble ash was found to be (0.27-0.35 %). The water-soluble ash value is low (0.31-0.75 %). The aqueous extract of the drug was almost neutral as pH of aqueous solution falls in the range of 6.14-6.56. The volatile oil is in traces only.

Quality control parameters

Microbial load

Estimation of microbial growth is very important parameter in traditional medicines. It indicates whether the drug contains disease causing and spoilage microorganisms in

permissible limits. The assessment is done for evaluating the total bacterial count, total fungal count, count of bacteria belonging the *Enterobacteriaceae* family, count of pathogens like *E. coli*, to *Staphylococcus aureus*, *Salmonella* spp. and *Pseudomonas aeruginosa*. The results of microbial load are shown in Table 3 which indicate that the drug is safe for internal use.

Aflatoxins

The results of aflatoxins analysis of the drug are given in Table 4. Aflatoxins are toxic metabolites produced by a variety of molds such as *Aspergillus flavus*, A. *parasiticus* and *A. nomius*. The results do not show the presence of any of the aflatoxins contents (B1, B2, G1, and G2) in Qurs-e-Isqeel.

Pesticide residue

The results of pesticide residues are given in table 5. Harvest of herbal material without the use of pesticides is very difficult due to several factors. But as per WHO guidelines, the major concern is whether the drug contains pesticide residue in permissible limits or not. In order to estimate the pesticide residue, the drug was analyzed on GC-MS. The results indicated that the drug is free of pesticide residues and safe for use.

Heavy metal analysis

The results of Heavy metal estimation are given in Table 6. Heavy metals are hazardous to human health and may cause many fatal diseases. A heavy metal has relatively high density or atomic weight and is toxic or poisonous even at low concentrations. The heavy metal content in Qurs-e-Isqeel was found to be below detection limit which indicated that the drug was free from heavy metal contamination.

Table 1: Formulation composition.

S.No.	Ingredients	Botanical Name/English Name	Part used	Form
1.	Isqeel (Jangli Piyaz)	*Drimia indica* (Roxb.) Jessop	Bulb	Powder
2.	Arad-e-Karsana	*Pisum sativum* L.	Pea flour	Powder
3.	Sharab-e-Musallas	Brandy	As such	Liquid

Table 2: Physico-chemical parameters.

S. No.	Parameters	Results (%)
1.	Water soluble extractive (%)	7.85-8.34
2.	Alcohol soluble extractive (%)	1.04-1.28
3.	Hexane soluble extractive (%)	0.44-0.51
4.	Loss in wt. on drying at 105^0C	8.81-9.78
5.	Total ash (%)	2.12-2.24

6.	Acid Insoluble ash (%)	0.27-0.35
7.	pH of 1% aqueous Soln.	6.14
8.	pH of 10% aqueous Soln.	6.56
9.	Volatile oil	Traces

Table 3: Microbial load.

S. No.	Parameter Analysed	Results	Permissible limit as per WHO
1.	Total Bacterial count	<1 x10^2 CFU/gm	10^5 CFU/gm
2.	*Enterobacteriaceae*	Absent	Nil
3.	*Salmonella spp.*	Absent	Nil
4.	*Escherichia coli*	Absent	Nil
5.	*Staphylococcus aureus*	Absent	Nil
6.	*Pseudomonas aeruginosa*	Absent	Nil
7.	Total fungal count	<1 CFU/gm	10^3 CFU/gm

Table 4: Aflatoxins level.

S. No.	Parameter Analyzed	Results	Permissible limit as per WHO
1.	B1	Not detected	< 2ppb
2.	B1+B2+G1+G2	Not detected	< 5ppb

Table 5: Pesticide residue.

S. No.	Parameter Analyzed	Results	Permissible limit as per WHO (mg/kg)
1.	Alachor	BLQ	0.02
2.	Aldrin	BLQ	0.05
3.	Azinphos –methyl	BLQ	1.0
4.	Chlordane (cis & trans)	BLQ	0.05
5.	Chlorfenvinphos	BLQ	0.5
6.	Chlorpyrifos	0.030	0.2
7.	Chlorpyrifos-methyl	BLQ	0.1
8.	Cypermethrin	BLQ	1.0
9.	DDT	BLQ	1.0
10.	Deltamethrin	BLQ	0.5
11.	Diazinon	BLQ	0.5
12.	Dichlorvos	BLQ	1.0
13.	Dimethoate	BLQ	0.1
14.	Dieldrin	BLQ	0.03
15.	Endosulphan	BLQ	3.0
16.	Endrin	BLQ	0.05
17.	Ethion	BLQ	2.0
18.	Fenitrothion	BLQ	0.5
19.	Fenvalerate	BLQ	1.5
20.	Heptachlor	BLQ	0.05
21.	Hexacholobenzene	BLQ	0.06
22.	Lindane (gamma HCH)	BLQ	0.6

23.	Malathion	BLQ	1.0
24.	Parathion	BLQ	0.5
25.	Parathion-methyl	BLQ	0.2
26.	Permethrin	BLQ	1.0
27.	Phosalone	BLQ	0.1
28.	Primiphos methyl	BLQ	0.1

BLQ-Below limit of quantification

Table 6: Heavy Metals.

S. No.	Parameter Analyzed	Results	Permissible limit as per WHO (ppm)
1.	Lead	Not detected	10.00
2.	Aluminium	Not detected	0.30
3.	Arsenic	Not detected	3.00
4.	Mercury	Not detected	01.00

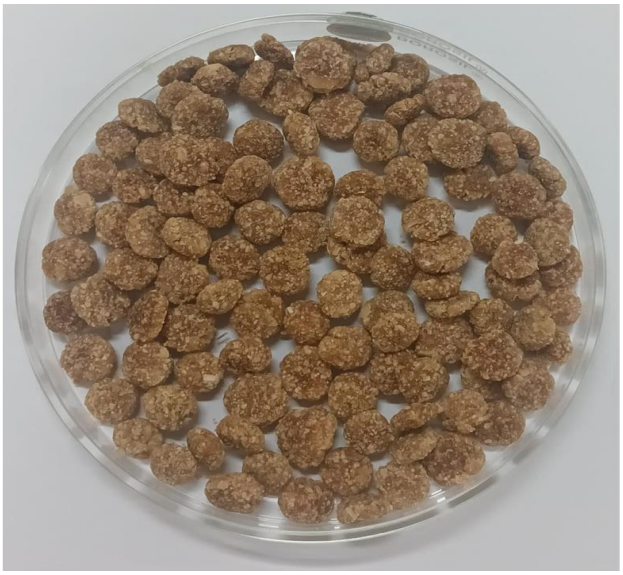
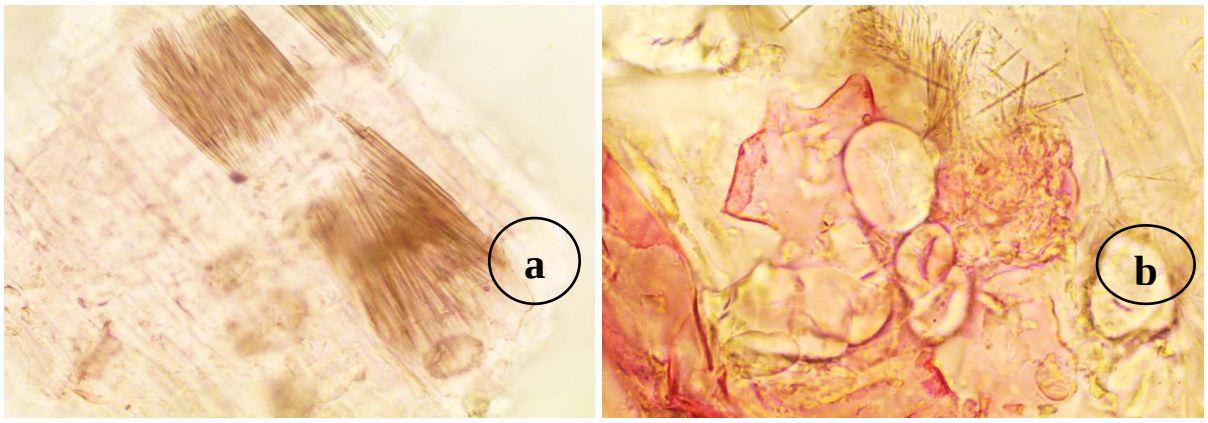


Figure 1: Qurs-E-Isqeel.



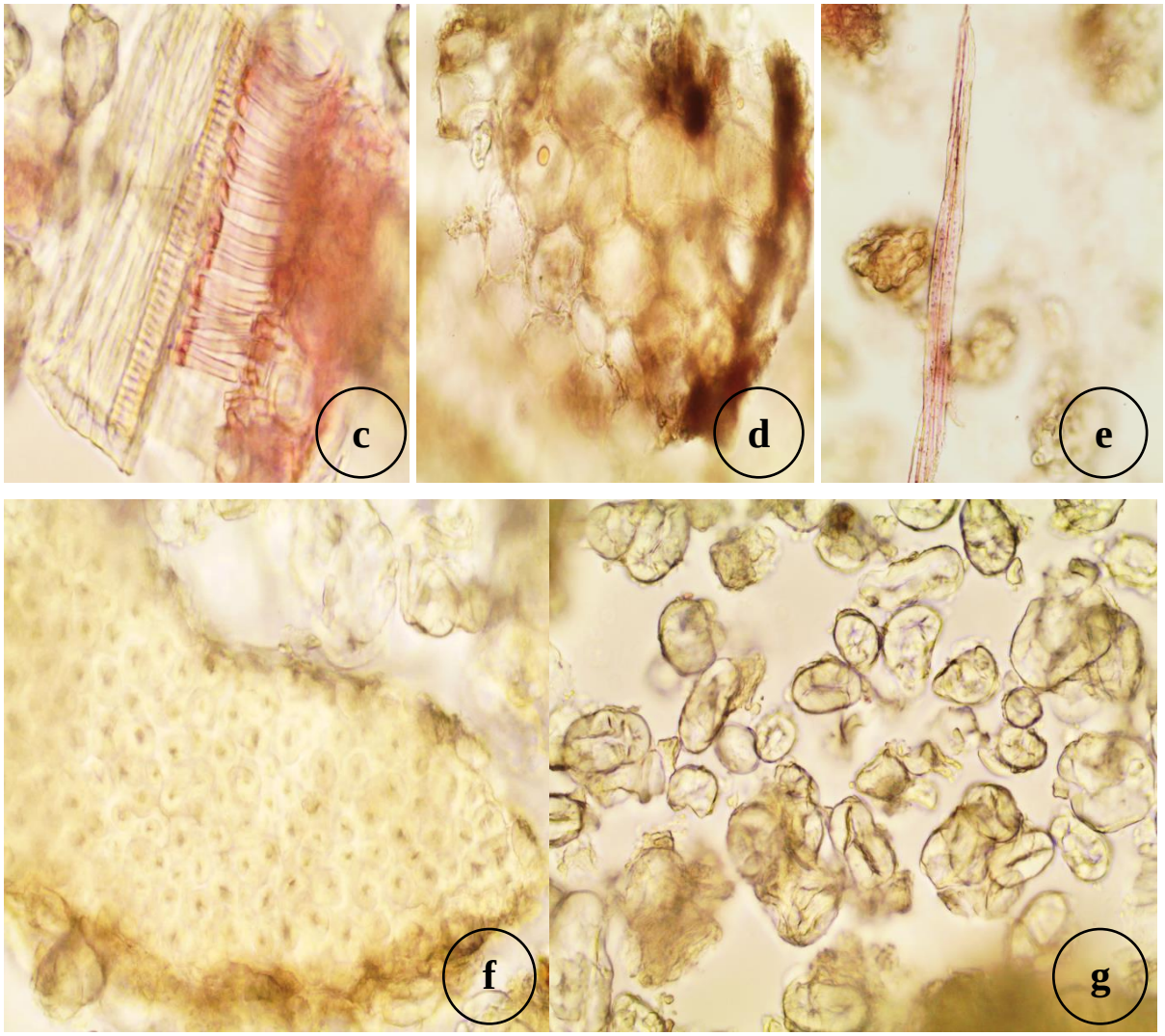


Figure 2: Microscopic examination of compound formulation *Qurs-e-Isqeel*:- (a) Acicular calcium oxalate crystals, **b)** mucilage cells with acicular calcium oxalate crystals, **c)** spiral or annular vessels with lignified walls, **d)** parenchyma cells, **e)** fibres with tapering ends **(a to e - *Jangli Piyaz*); f)** fragment of cotyledons, **g)** pseudo-compound starch granules **(f & g - *Arad-e-Karsana*)**.

CONCLUSION

It can be concluded that organoleptic parameters are not much reliable in identification of herbal drugs as the ingredients are usually powdered and mixed together for preparation of compound formulations. The present study therefore holds high significance as the microscopic features and various physico-chemical parameters provide criteria for easy identification of the formulation Qurs-e-Isqeel and quality control analysis ensures the authenticity, quality and efficacy of the medicine.

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