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Development of Quality Standards of Majoon-e-Aradkhurma-A Polyherbal Unani Formulation

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ABSTRACT

Traditional healing through herbs have been the experienced of many countries since ages, as they were generally whispered to be non toxic natural products. Contemporary medicine is more concern for the cure of diseases but remnants indifferent to health conservation. There is an urgent need to combine the best elements of traditional medicine and modern medicine to improve the health care system of human kind. For the reason that of the rapid progress of herbal drug an increasing need is felt to standardize the herbal products. It is needed to develop the scientific protocols such as SOP and pharmacopoeial standards of the poly herbal drug Majoon-a- Aaradkhurma. Majoon-e-aradkhurma is traditionally used to controls spermatorrhoea and nocturnal emissions and it increases the density of semen and sperm count. The pharmacognostical evaluation comprises of phytochemical screening, physical constants such as ash values, extractive values, heavy metal, pesticide residue and aflatoxin analysis. Study revealed that heavy metals such as lead, cadmium, mercury and arsenic were not detected in the drug. Pesticide residues and aflatoxin were also absent in the drug. The data evolved in the present work help to fix the scientific standards for Majoon-a- Aaradkhurma.

Keywords: Majoon-e-Aradkhurma, Extractive values, Ash values, Heavy metal, Pesticide, Aflatoxin

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INTRODUCTION

Traditional healing through herbs have been the practiced of many countries since ages, as they were generally believed to be non toxic natural products, according to world health organization, the usage of herbal drugs exceed 2-3 times than synthetic drugs[1]. The unani medicines are used frequently in many traditional systems throughout the globe, there acceptably in modern medicine and in developed world is remarkably low, largely due to the lack of standardization[2]. Moreover, Unani and Ayurvedic products are exported and marketed in various developed countries of the world under the name of food supplement not the drugs due to quality and safety point of view[3]. Because raw material of these formulations is collected from wild sources and varies in constituents and efficacy due to geographical diversity. Improper collection and storage conditions lead to contamination of microorganism and heavy metals[4]. It is necessary to develop the scientific protocols such as Standard Operational Procedure (SOPs) and Pharmacopoeial standards of the poly herbal drugs. Standardization is the prime need of time because standardization establishes quality and identity profile that can be used for the purpose of safety monitoring and overall quality assurance of unani medicines[5]. In the present investigation an attempt has been made for the Pharmacopoeial standardization and evaluation of Majoon-e-aradkhurma. The pharmacognostical evaluation comprises of physical constants such as ash values, extractive values, heavy metal, and pesticide residue and aflatoxin analysis. The data obtained in present study will serve as valuable tool for identification, authentication and detection of adulterants, standardization and quality control of this unani formulation.

MATERIALS AND METHODS:

All the chemicals and reagents used were of analytical grade, purchased from Sigma chemical co. (St Louis, MQ, USA) and Merck (Darmstadt, Germany). The ingredients as described in National Formulary of Unani Medicine, Part I (Table – 1) were procured from local market of Delhi, India.

Preparation of Majoon:

All the ingredients were powdered in an electric grinder and sieved in No 80 mesh. The concentrated solution (qiwam) of sugar was prepared according to the standard unani method[6]. Powdered ingredients were added in the qiwam (sugary base) and mixed well. The powder of fried Pista (Pistachio) Badam (Almond), Akhroot (Walnut) and paste of Khajoor (Dates) were also added. Citric acid and benzoic acid were added as preservatives. All ingredients of majoon and qiwam were mixed properly by stirring and finally a semisolid syrupy mixture was obtained.

Table1. Ingredient of Majoon-e- Aaradkhurma[6]:

S.No	Unani name	Botanical name	Part used
1.	Ilachi khurd	*Elettaria cardamomum*	Seed
2.	Pepal kalan	*Piper longum*	Fruit
3.	Khajoor	*Phoenix sylvestris*	Fruit
4.	Darcheni	*Cinnamomum zeylanicum*	Bark
5.	Zanjibeel	*Zingiber officinale*	Rhizome
6.	Singhada khushk	*Trapa bispinosa*	Fruit
7.	Qaranfal	*Syzygium aromaticum*	Bud
8.	Khurma	*Phoenix dactylifera*	Fruit
9.	Gond keekar	*Acacia Arabica*	Gum
10.	Maghz badam sheeri	*Prunus amygdalus*	Fruit
11.	Maghz pista	*Pistacia vera*	Fruit
12.	Maghz akhrot	*Juglans regia*	Fruit
13.	Maghz narjiil	*Cocos nucifera*	Fruit
14.	Shaker safaid	*Sugar*	Quiwam
16.	Sat leemu	*Citrus aurantifolia*	Preservative

Physicochemical Standardization:

The formulation was standardized according to WHO guidelines and other pharmacopoeias procedures physicochemical standardization which includes total ash value, acid insoluble ash value and water soluble ash value, pH value, aflatoxins, heavy metals and pesticides residues in different extracts were analyzed as per the standard methods[1,7-10].

Determination of individual extractive values (Cold extraction):

The accurately weighed amount (10 gm) of majoon was extracted with different solvents (Petroleum ether, chloroform and methanol) separately in a conical flask at a room temperature. The extracts were evaporated to dryness and their constant extractive values were recorded[7-10]. Comparative accounts of extractive values determined and presented in Figure 1.

Determination of individual extractive values (Hot extraction):

Weighed amount of majoon (10 gm) was extracted with petroleum ether, chloroform and methanol separately in a Soxhlet apparatus[7-10]. The observations are presented in Figure 1.

Determination of successive extractive values:

The accurately weighed quantity (10 gm) of majoon was subjected to successive extraction in a Soxhlet apparatus with different solvents like petroleum ether, chloroform and methanol. The extracts were evaporated to dryness and their constant extractive values were recorded[7-10].

Determination of total ash values:

The majoon (2 gm) was placed in a suitable tarred crucible of silica previously ignited and weighed. The formulation was spread in to an even layer and weighed accurately. The material was incinerated by gradually increasing the heat, not exceeding 450°C until free from carbon,

cooled in a desiccators, weighed and percentage ash was calculated by taking in account the difference of empty weight of crucible and that of crucible with total ash[7-10].

Determination of acid insoluble ash values:

The obtained ash was boiled with 25 ml dilute HCl (6N) for 5 minutes. The insoluble matter collected on an ash less filter paper, washed with hot water and ignited in crucible at a temperature not exceeding 450^{o}C to a constant weight. The aired materials were then assayed for their acid-insoluble ash content[7-10].

Determination of water soluble ash values:

The ash obtained was dissolved in distilled water and the insoluble part was collected on an ash less filter paper and ignited at 450^{o}C to constant weight. By subtracting the weight of insoluble part from that of the ash, the weight of soluble part of ash was obtained. The materials were then assayed for their water soluble ash content[7-10].

Loss on Drying (LOD):

The formulation sample (10 gm) without preliminary drying was placed on a tarred evaporating dish and dried at 105ºC for 6 hours and weighed. The drying was continued until two successive reading matches each other or the difference between two successive weighing was not more than 0.25%. Constant weight was reached when two consecutive weighing after drying for 30 minutes in a desiccators, showed not more than 0.01 gm difference[7-10].

Determination of pH:

pH 1% solution:

Dissolved an accurately weighed 1 gm of the drug in accurately measured 100 ml of distilled water, filtered and checked pH of the filtrate with a standardized glass electrode.

pH 10% solution:

Dissolved an accurately weighed 10 gm of the drug in accurately measured 100 ml of distilled water, filtered and checked pH of the filtrate with a standardized glass electrode.

Phytochemical screening:

The phytochemical evaluation of drug was carried out as per the method described. Previously weighed amount of Majoon-e-aradkhurma (10 gm) was extracted in a Soxhlet apparatus with petroleum ether, chloroform, and methanol respectively. The extracts were evaporated to dryness under vacuum in rotary evaporator. These extract were used for the analysis of different phyto-constituents *viz*. alkaloids, carbohydrates, phenolics, flavonoids, proteins, amino acids, saponins, mucilage and resins etc.

Heavy metal residues:

Residues of heavy metals (Cd, Pb, As and Hg) in the majoon were determined according to the American Organization of Analytical Chemists (AOAC) official method of analysis[11].

Pesticide residues:

Pesticides (organochlorines, organophosphates and pyrethroids) residues in the extracts were determined according to AOAC guidelines[11].

Aflatoxins analysis:

Aflatoxins were analysised in the different plant extracts by HPLC method as described by Scott (AOAC method 980.20-ITEM-I) [12].

RESULTS AND DISCUSSION:

Extractive value:

Estimation of extractive values determines the amount of the active constituents in a given amount of drug when extracted with solvent. It is employed for that material for which no chemical and biological assay method exit. The extractions of any crude drug with a particular solvent yield a solution containing different phytoconstituents. The compositions of these phytoconstetuents depend upon the nature of the drug and solvent used. The use of a single solvent can be the means of providing preliminary information on the quality of particular drug. Extractive value also give the information regarding the quality of the drug (whether drug is exhausted or not). Methanol followed by petroleum ether proved to be highly effective for high cold extractive values. The maximum hot extractive values noticed in methanol extract. The maximum successive extractive values recorded in methanol (Figure 1).

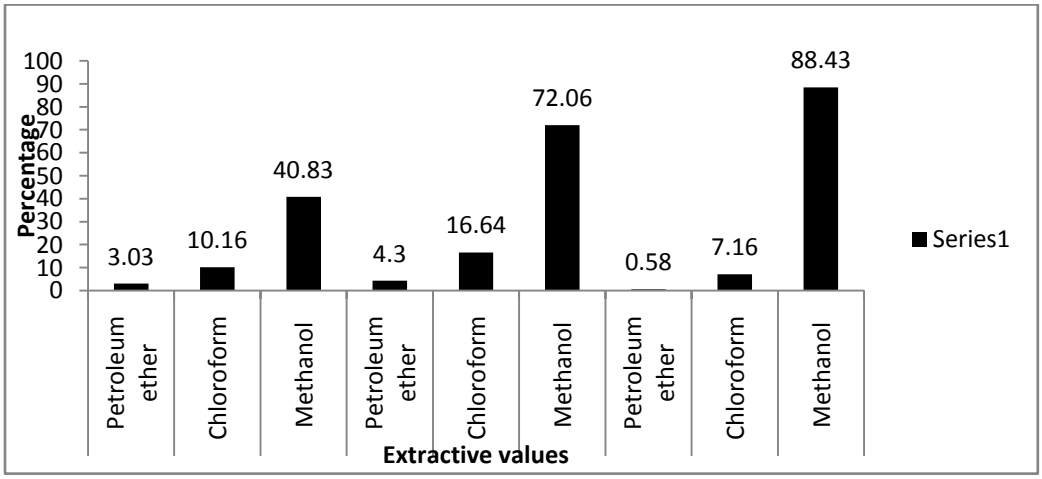

Figure 1. Percentage of different extractive values

Determination of ash values, loss on drying and pH:

The ash value of any organic material is composed of their non volatile inorganic components.

Control incineration of crud drugs result in ash residue consisting of an inorganic material (metallic salt and silica). This value varies within fairly wide limits and is therefore, an important parameter for the purpose for evaluation of crude drugs. Unwanted parts of drug, some time posses a character that will raise the ash value. A high value is indicative of contamination, substitution, adulterations or carelessness in preparing the crude drug for marketing. The total ash values, water soluble ash, acid insoluble ash, loss on drying (LOD), pH values and moisture content were determined. The results noticed were; loss on drying (9.37 ± 0.15%), total ash (1.455 ± 0.25%), water soluble ash (0.77 ± 0.12%), acid insoluble ash (0.52 ± 0.34%), pH value 1% solution (4.3 ± 0.01), pH value 10%solution (7.2 ± 0.02) and moisture content (5.09 ± 1.07) respectively (Table-2).

Table 2. Results of physicochemical evaluation of Majoon-e- aradkhurma

Parameters	% w/w (Mean± SD)
Loss on drying	9.37 ± 0.15
Total ash	1.455 ± 0.25
Water soluble ash	0.77 ± 0.12
Acid insoluble ash	0.52 ± 0.34
Moisture content	5.09 ± 1.07
pH value 1% solution	4.3 ± 0.01
pH value 10%solution	7.2 ± 0.02

Table 3. Phytochemical screening of different extract of Majoon-e- Aradkhurma

Constituents	Hexane	Acetone	Chloroform	Benzene	Petr.ether	Methanol
Alkaloids	_	+	_	_	_	+
Carbohydrates	_	_	_	_	_	+
Phenolic compounds	_	_	+	_	+	+
Flavonoid	_	_	_	_	+	+
Glycoside	_	+	_	_	+	+
Tannin	_	_	+	_	_	+
Proteins and amino- acids	_	_	_	_	_	_
Saponins	_	_	_	+	_	+
Sterol	_	_	+	_	_	+
Resins	+	_	+	_	+	+
Lipids / Fats	+	_	+	_	+	+

(-: Absent, +: Present)

Phytochemical screening:

Phytochemical evaluation of the extracts may provide the information regarding various types of phytoconstituents present. Presence or absence of particular types of phyto constituents in the sample of the interest may be helpful, partly in the development of metabolomics and analytical profile and in the differentiation of contravention plants. The extracts were subjected to

preliminary chemical tests to detect the presence and absence of various phytoconstituents. Alkaloids, carbohydrates, phenolic compounds, flavonoid, glycoside, tannin, proteins and amino acids were present in different extracts (Table-3).

Table 4: Determination of Pesticide residues of Majoon-e-Aaradkhurma

S. No	Pesticide	Test method	Results	MDL
1	α -BHC	AOAC970.52/EPA525.5	Not detected	0.01mg/kg
2	β-BHC	AOAC970.52/EPA525.5	Not detected	0.01mg/kg
3	γ-BHC(Lindanee)	AOAC970.52/EPA525.5	Not detected	0.01mg/kg
4	δ-BHC	AOAC970.52/EPA525.5	Not detected	0.01mg/kg
5	Heptachlor	AOAC970.52/EPA525.5	Not detected	0.01mg/kg
6	Heptachlor_Epoxide	AOAC970.52/EPA525.5	Not detected	0.01mg/kg
7	α -Chlordane	AOAC970.52/EPA525.5	Not detected	0.01mg/kg
8	α -Endoulfan	AOAC970.52/EPA525.5	Not detected	0.01mg/kg
9	β -Chlordane	AOAC970.52/EPA525.5	Not detected	0.01mg/kg
10	Endrin	AOAC970.52/EPA525.5	Not detected	0.01mg/kg
11	Total DDE	AOAC970.52/EPA525.5	Not detected	0.01mg/kg
12	Total DDD	AOAC970.52/EPA525.5	Not detected	0.01mg/kg
13	Total DDT	AOAC970.52/EPA525.5	Not detected	0.01mg/kg
14	β -Endoulfan	AOAC970.52/EPA525.5	Not detected	0.01mg/kg
15	Endrin_Aldehyde	AOAC970.52/EPA525.5	Not detected	0.01mg/kg
16	Endoulfan_sulfate	AOAC970.52/EPA525.5	Not detected	0.01mg/kg
17	Aldrin	AOAC970.52/EPA525.5	Not detected	0.01mg/kg
18	Endrin_Ketone	AOAC970.52/EPA525.5	Not detected	0.01mg/kg
19	Methoxychlor	AOAC970.52/EPA525.5	Not detected	0.01mg/kg
20	Dieldrin	AOAC970.52/EPA525.5	Not detected	0.01mg/kg
21	Alachlor	AOAC970.52/EPA525.5	Not detected	0.01mg/kg
22	Butachlor	AOAC970.52/EPA525.5	Not detected	0.01mg/kg
23	Monochlorphos	AOAC970.52/EPA525.5	Not detected	0.01mg/kg
24	Phorate	AOAC970.52/EPA525.5	Not detected	0.01mg/kg
25	Mevinphos	AOAC970.52/EPA525.5	Not detected	0.01mg/kg
26	Dimethoate	AOAC970.52/EPA525.5	Not detected	0.01mg/kg
27	Malathion	AOAC970.52/EPA525.5	Not detected	0.01mg/kg
28	Methyl-parathion	AOAC970.52/EPA525.5	Not detected	0.01mg/kg
29	Chlorpyrifos	AOAC970.52/EPA525.5	Not detected	0.01mg/kg
30	Ethion	AOAC970.52/EPA525.5	Not detected	0.01mg/kg
31	Atrazine	AOAC970.52/EPA525.5	Not detected	0.01mg/kg
32	Simazine	AOAC970.52/EPA525.5	Not detected	0.01mg/kg
33	Diazinone	AOAC970.52/EPA525.5	Not detected	0.01mg/kg
34	Phosphamidon	AOAC970.52/EPA525.5	Not detected	0.01mg/kg
35	Fenitrothion	AOAC970.52/EPA525.5	Not detected	0.01mg/kg
36	Fenthion	AOAC970.52/EPA525.5	Not detected	0.01mg/kg
37	Phosalone	AOAC970.52/EPA525.5	Not detected	0.01mg/kg
38	Quinaphos	AOAC970.52/EPA525.5	Not detected	0.01mg/kg
39	Coumaphos	AOAC970.52/EPA525.5	Not detected	0.01mg/kg
40	Parathion	AOAC970.52/EPA525.5	Not detected	0.01mg/kg

MDL- maximum detection limit

Determination of pesticides, heavy metals and aflatoxins:

The chemical constituent in plants including metal ions, are particularly responsible for medicinal and nutritional properties and as well as toxicity. Men, animals and plants through air, water and food take up these metals from the environment. Medicinal plants which are the raw material for many of the unani formulations and popular nutrients supplements are sold all over the country. Effect of toxic metals (Cd, Cr, Pb, Ni, etc) on human health and their interaction with essential trace elements may produce serious consequences. Environmental impact of heavy metals such as Cd, Pb, Hg and As, as well as their health effects has been the source of the major concern. The amount of pesticide, heavy metals and aflatoxin were determined in majoon. The results noticed were; absence of pesticide more ever neither heavy metals nor aflatoxin were detected in the majoon sample. The results and observation are presented in Table 4-6.

Table .5: Aflatoxin residues of Majoon-e- Aaradkhurma

S. No	Test parameter	Test method	Results	MDL
1	AflatoxinB1	AOAC 990.33	Not detected	1.0µg/kg
2	AflatoxinB2	AOAC 990.33	Not detected	1.0µg/kg
3	AflatoxinG1	AOAC 990.33	Not detected	1.0µg/kg
4	AflatoxinG2	AOAC 990.33	Not detected	1.0µg/kg

MDL- maximum detection limit

Table.6: Heavy metal residues of Majoon-e- aarad khurma

S. No	Test parameter	Instrument used	Results	MDL
1	Cadmium (Cd)	ICP-OES	Not detected	0.01mg/kg
2	Lead (Pb)	ICP-OES	Not detected	0.01mg/kg
3	Arsenic (As)	ICP-OES	Not detected	0.01mg/kg
4	Mercury (Hg)	ICP-OES	Not detected	0.01mg/kg

MDL- maximum detection limit

CONCLUSION:

Hence the drug under study was subjected to physicochemical analysis, which is helpful in establishing the standard parameters. Heavy metal analysis, aflatoxins contamination, pesticide residue analysis was done and found absent as reported in the present investigation. Consequently, the drug was used to determine and ascertain its quality standard. The study is likely to help in the quality assurance of drug used in the Unani System of Medicine and in standardization of the compound formulation Majoon-a-Aaradkhurma.

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