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Research Article

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Quality Control of a Marine Origin Based Herbo-Mineral Unani Formulation

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ABSTRACT
Kushta Marjan (KM) is one well-known formulation prepared from coral used by Unani physicians for effective treatment of a various ailments. Coral is the calcareous skeleton of the minute marine organism and belongs of phylum coelenterate and is used internally in the form of *kushta.* It is highly efficacious in Cough, Asthma and Anorexia. Till date no scientific work has been performed to establish quality control of KM. In this study we have enlightened the preparation procedures, structural, functional and physico-chemical properties of KM by analyzing it on classical tests along with modern scientific techniques like FTIR (Fourier-transform infrared spectroscopy), TGA (Thermogravimetric Analysis) and DSC (Differential Scanning Calorimetry) to establish its quality control parameters. FTIR spectra showed peaks attributed to calcium oxide, organic matter and several other substances. Total weight loss during TGA was 30.09% and DSC curve showed peaks at 20°C, 65°C, 183°C and 261°C. Since the study has been first of its kind the results obtained might be taken as standard finger print of *kushta marjan* and might be helpful for further formulation and standardization of KM in routine analysis.

Keywords: Differential scanning calorimetry, Fourier-transform infrared spectroscopy, *Kushta marjan*, Quality control, Thermo gravimetric Analysis, Unani Medicine.

INTRODUCTION

Kushtajat (singular: *kushta*herbo -mineral compound preparations used in Unani system of medicine. According to traditional concepts preparing any drug in form of *kushta* remarkably improves the potency, efficacy and safety of the individual components used. However, preparation of a particular *kushta* depends on several factors like intended use, type of mineral or herbs used [1] or quantum of heat given during the process. *Kushta* gives different pharmacological effects and address different ailments depending upon their method of preparation. [2] Coral is the calcareous skeleton of the minute marine organism and belongs of phylum coelenterate [3] and is used internally in the form of *kushta*. *Kushta marjan* (KM) is highly efficacious in *khansi* (Cough), *dama* (Asthma), and *zof ishtaha* (Anorexia). [4] Although *kushtas* have been regularly used by Unani physicians

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however, little attempt has been made to study this type of dosage form in a scientific manner. This is because of a lack of communication among traditional healers, physicians and scientists and the unavailability of the literature in English. [1] So, this research work seeks to physico-chemically evaluate *kushta marjan* prepared for the establishment of its quality control on classical Unani parameters of ideal *kushta* like floating, fineness, wall sticking tests and also through modern sophisticated instrumental analysis like FTIR (Fourier-transform infrared spectroscopy), TGA (Thermo gravimetric Analysis) and DSC (Differential Scanning Calorimetry).

MATERIALS AND METHODS

Marjan, milk *and misri* (crystalline sugar) were purchased from the local market.

Method of purification of *Marjan*

Marjan (Fig. 1) was put inside a cotton bag (Fig. 2) and dipped in milk and boiled for two hours (Fig. 3).[5]

Method of preparation of *Kushta Marjan*

Kushta Marjan was prepared as per the methods mentioned in *Kitab ul taklees* [4] by using electric muffle furnace as heat source. *Marjan* and *misri* were kept inside crucible in muffle furnace and thermogram given by Parmar DK *et al* was adopted. [6] After self cooling *kushta* was removed carefully and was labeled as KM (*Kushta marjan*) (Fig 4).

Physico-chemical parameters

KM was evaluated for classical parameters like organoleptic properties, floating test, fineness test, wall stick test, thumb finger test as well as modern scientific parameters like bulk density, tapped density, Hausner's ratio, Carr's compressibility index, [7] pH, [8] ash values. [9] The tests were repeated thrice to obtain mean values. FTIR (Fourier-transform infrared spectroscopy), TGA (Thermo gravimetric Analysis) and DSC (Differential scanning colorimetry) analysis of KM was also done.

The testing methodology was as followed.

(i) Floating test: If a small quantity of *kushta* is sprinkled on water surface then ideally it should float on the surface. [10]

(ii) Grain floating test: Grain of rice, barley, etc. will float over the ideal *kushta* like a swan on a lake. [10]

(iii) Finger test: On rubbing a small quantity of the *kushta* between the fingers, it should enter into the lines and creases of the fingers. [10]

(iv) Loss of metallic lusture: When visually examined preferably in presence of sunlight, no metallic luster should be observed. [11]

(v) Wall stick test: On throwing on the wall, ideal *kushta* should stick to the wall.

Fourier-transform infrared spectroscopy (FTIR)

Fourier-transform infrared spectroscopy (FTIR) of KM was performed at Bio-Products Laboratory, Central Leather Research Institute (CLRI), Council of Scientific and Industrial Research (CSIR), Adyar, Chennai. The spectra were recorded on a Nicolet 360 Fourier Transform Infra Red (FTIR) Spectrometer using KBr pellet containing 2–6 mg of sample; it took 15 min to complete an assay using Perkin-Elmer Spectrum 2000 instrument. [12]

Thermogravimetric Analysis (TGA)

The Thermal analysis of KM was carried out using thermo gravimetric Analyser, (TA Q 50 V 20.13 build 39) with 20°C/minutes increment in temperature in inert (dry nitrogen) atmosphere. TGA was carried out by raising the temperature of the sample gradually. Weight loss was recorded from 0°C to 800°C and plotting weight (percentage) against temperature. After the data was obtained, curve smoothing and other operations were done to find the exact point of inflection. TGA results were analyzed using TA universal analysis NT software. [13]

Differential scanning colorimetry (DSC)

The Differential Scanning Calorimetric analysis was performed using TA-DSC Q 200 V 24.10 build 122. Thermograms were analyzed with TA universal analysis NT software. [14]

Fig. 1 ***Marjan***

Fig. 2 ***Marjan*** **Bag dipped in milk**

Fig. 3 After 2 hours boiling

Fig. 4 ***Kushta Marjan***

RESULTS AND DISCUSSION

Ideally *kushta* should be tasteless, odourless and lustureless. KM was tasteless, odourless, smooth to touch and lusterless. Floating test, fineness test and wall stick test were positive indicated *kamil* (correct) preparation as per classical Unani literature (Table 1).

Table 1: Preliminary tests of Raw *Marjan* and KM

Properties	*Marjan* powder	KMM
Colour	Red	White
Odour	Odourless	Odourless
Taste	Tasteless	Tasteless
Touch	Smooth	Very Smooth
Floating test	Absent	Present
Fineness test	Fine	Very fine
Wall stick test	Absent	Present
Finger test	Negative	Positive
Lusture	Present	Absent

Table 2: Physicochemical Tests of KM (n=3)

Parameters	Mean ± SEM
Bulk Density	0.35 ± 0.00
Tapped Density	0.59 ± 0.00
Hausner's Ratio	1.96 ± 0.00
Carr's Index	43.60 ± 0.14
pH (1%)	11.13 ± 0.02
pH (10%)	11.27 ± 0.01
Total ash (%)	81.65 ± 0.04
Acid insoluble ash (%)	76.65 ± 0.17
Extractive value	1.20 ± 0.03

The mean value of bulk density and tapped density of KM was 0.35 ± 0.00 g/ml and 0.59 ± 0.00 g/ml respectively (Table 2). Bulk and tapped density estimation is a method to determine the densities of powder under loose and packed conditions respectively. It is one of the measures of packing, compressibility and flow properties. [14] The mean value of Hausner's ratio of KM and Compressibility Index of KM were 1.96 ± 0.00 and 43.60 ± 0.14% respectively (Table 2). The compressibility index is a measure of the propensity of a powder to consolidate. It is a measure of the relative importance of inter-particulate interactions. [7] Hausner's ratio and compressibility index of KM was greater than 1.60 and 38 respectively hence indicating very, very poor flowability. The pH value of KM was 11.13 ± 0.02 in 1% and 11.27 ± 0.01 in 10% aqueous solutions respectively (Table 2). The mean percentage values of the total ash and acid insoluble ash in KM were 81.65 ± 0.04% and 76.65 ± 0.17% respectively (Table 2). High ash value in both *kushtas* showed the presence of very high inorganic content. The mean percentage of the water soluble extractive value of KM was 1.20± 0.03% (Table 2). Very low extractive values were indicative of very low organic matter in both *kushtas* and maximum quantity of inorganic substances.

FTIR spectra of KM (Fig. 5) showed peaks at 3696 cm−1 (kaolinite), [15] 3644 cm $^{-1}$ (Calcium oxide), [16] 3,574 cm^{-1} (carboxylic acid), [17] 3437 cm^{-1} (amine group), [18] 3431 cm^{-1} (OH vibration in absorbed water on sample surface), [19] 2925 cm $^{-1}$ (CH stretching of methyl group), [20] 2512 cm $^{-1}$ (organic matter), 1795 cm $^{-1}$ (water), [21] 1504 cm $^{-1}$ (para-substituted benzene rings), [22] 1454 cm^{-1} (CH_2 scissoring), [23] 1434 cm $^{-1}$ (carboxylate ion), [24] 1122 cm^{-1} (Tricalcium phosphate), [25] 949 cm^{-1} (Ethylene group), [26] 874-876 cm $^{-1}$, 713 cm $^{-1}$ (calcite) [27] and 580 cm^{-1} (Magnetite) [28] were observed.

TGA was used to determine total weight change in KM during thermal treatment. The weight loss in KM was 6.37% (403°C), 18.11% (455°C), 29.86% (667°C) and 30.09% (800°C) (Fig. 6). These weight losses can be attributed to loss of organic substances and adsorbed water present in KM.

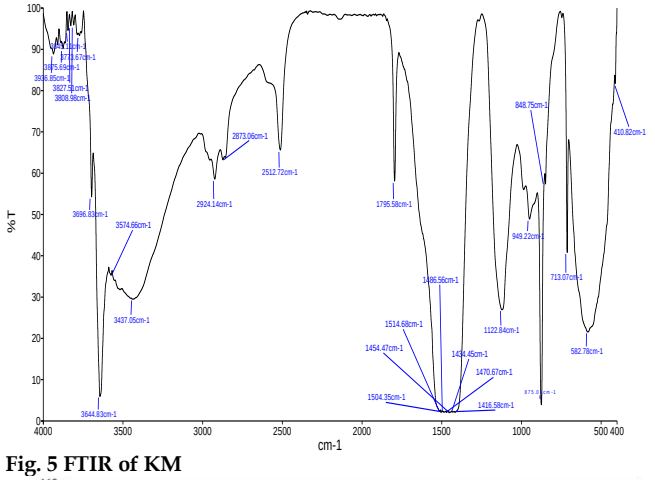

Fig. 5 FTIR of KM

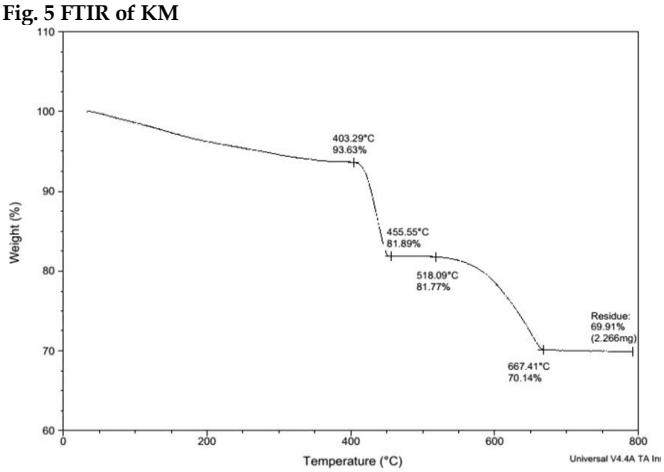

Fig. 6 TGA Curve of KM

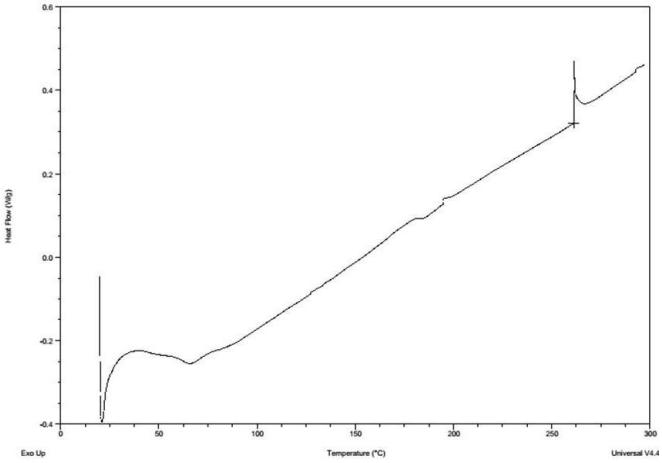

Fig. 7 DSC curve of KM

The DSC plot for KM (Fig. 7) showed four peaks in the range of 20°C-261°C (at 20°C, at 65°C, 183°C and at 261°C) which could be indicative for the decomposition

of water molecules as well as organic substances. The results obtained might be taken as standard finger print of KM and might be helpful for further formulation and quality control of KM in routine analysis.

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