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

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ABSTRACT

Unani doctors have long relied on a coral mixture called Kushta Marjan (KM) to cure a wide range of illnesses. In its internal form, kushta, coral—the calcareous skeleton of the little marine creature that belongs to the phylum coelenterate—is used. Cough, asthma, and anorexia are conditions in which it works well. There has been no research to far that can be used to develop KM quality control. Thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), Fourier-transform infrared spectroscopy (FTIR), and classical testing have all contributed to illuminating KM's preparation methods, structural, functional, and physico-chemical properties in this study. A number of compounds were identified in the FTIR spectra, including organic compounds, calcium oxide, and others. The DSC curve showed maxima at 20°C, 65°C, 183°C, and 261°C, and the total weight loss throughout TGA was 30.09%. The findings might serve as a benchmark for kushta marjan and inform future efforts to standardize and refine KM in regular analysis, given that this is the first research of its kind.

Unani medicine, thermal gravimetric analysis, quality control, Kushta marjan, Fourier-transform infrared spectroscopy, differential scanning calorimetry all play a role.

INTRODUCTION

Kushtajat (singular: *kushta*) are 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 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).

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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*

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Fig. 2 *Marjan* Bag dipped in milk

Fig. 3 After 2 hours boiling

Fig. 4 *Kushta Marjan*

RESULTS AND DISCUSSION

A flavorless, odorless, and glossy kushta is ideal. No flavor, no smell, no sheen, and no lustre could be detected in KM. Table 1 shows that according to traditional Unani literature, the results of the floating, fineness, and wall stick tests were positive, indicating kamil (proper) preparation.

Raw Marjan and KM perform preliminary testing, as shown in Table 1.

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

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.

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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 (CH2 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.

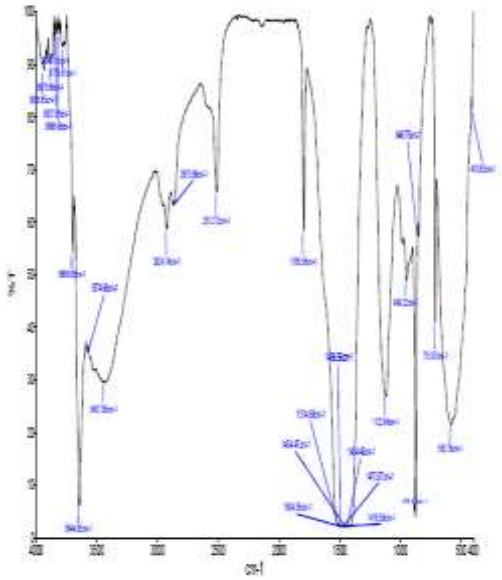

Fig. 5 FTIR of KM

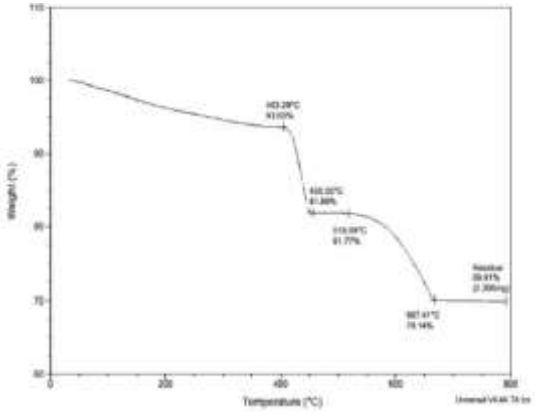

Fig. 6 TGA Curve of KM

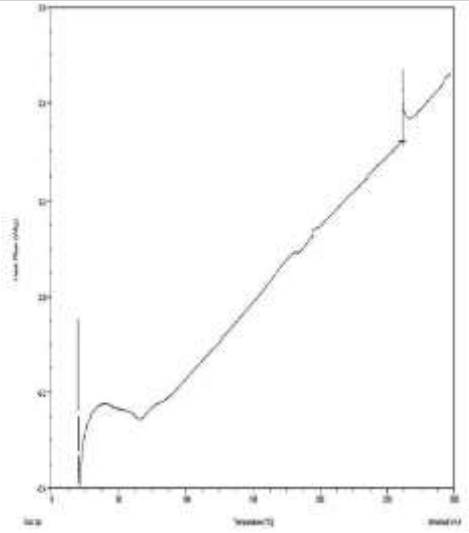

Fig. 7 DSC curve of KM

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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