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Preliminary physicochemical evaluation of *Kushta tutia*: A Unani Formulation

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ABSTRACT

Background: *Kushta* is an important solid dosage form of Unani system of medicine used to treat various ailments. Very small particle size of *kushta* is responsible for its rapid absorption in body leading to instant therapeutic actions. *Kushta tutia* (KT) is one such renowned formulation used by hakims for successful management of various disorders. However, there is lack of scientific work on KT. **Objectives:** The present study was performed to evaluate KT physicochemically by testifying it on classical tests along with modern scientific techniques. **Materials and Methods:** *Tutia* was first detoxified as per classical literature. It was triturated with water and dried, afterwards subjected to calcination in furnace rather than cow dung cakes due to isolation of material being heated and better temperature control. Finished product was evaluated for physicochemical characteristics including preliminary tests mentioned in classical literature. **Results:** Floating and finger test were positive. Curd test showed no discoloration after 48 h. These findings indicate correct preparation of KT according to classical literature. Bulk density (0.96 ± 0.00 g/ml); tapped density (1.53 ± 0.00 g/ml); Hausner ratio (0.62 ± 0.00), compressibility index (37.52 ± 0.19%); loss of weight on drying (0.08 ± 0.00%); pH of 1 and 10% (5.20 ± 0.00) and 5.62 ± 0.00, respectively); total ash, acid insoluble ash, and water soluble ash values 95.75 ± 0.09, 6.57 ± 0.02, and 45.02 ± 0.20%, respectively; and extractive values 0.85 ± 0.02% were reported in KT. **Conclusion**: Since this work has not been reported earlier, the results obtained could be considered as the standard for KT for future studies.

Key words: *Kushta,* standardization, *tutia,* Unani

INTRODUCTION

There are three principal systems of medicine practiced in India: Ayurveda, Siddha, and Unani-Tibb. These systems use mineral preparations mostly in calcined forms: *Bhasmas* in Ayurveda, *Kushtas* in Unani-Tibb, and *Parpams* in Siddha.[1] The term *kushta* (calcined product) is employed for a dosage form that is a blend of metals, metallic oxides, non-metals and their compounds, used in small quantity and one that is immediately effective. The preparation of a *kushta* results in increased efficacy of a medicine, and after affecting its entry into the body, the *kushta* discharges its curative role promptly and effectively.[2] *Tutia* is a semi-metal derived from copper.[3] It is known by various vernacular names like *tutiae akhzar* in Arabic, *tutia* and *nilathotha* in Hindi, and copper sulfate or blue vitriol in English. It is a blue colored crystalline substance which is available in native form occasionally.[4] In India, natural occurrence of *tutia*, in abundance, is uncommon. A little amount of *tutia* can be seen near copper mines in Rajasthan, Madhya Pradesh, and Jharkhand.[5] It is prepared by roasting copper pyrites with sulfur, then dissolving the roasted mass in water and evaporating the solution to obtain dark blue crystals of copper sulfate.[3] It is easily crystallizable and used in different industries for various purposes.[6] It is powerful *qabiz* (astringent), *muqee* (emetic), *daf-e-taffun* (antiseptic), *moharrik* (stimulant), and mild *kawi* (escharotic). It is used in case of poisoning by narcotics like opium (*Papaver somniferum* Linn.), nux vomica (*Strychnos nux-vomica* Linn), etc.[3] Externally copper sulfate is applied to indolent ulcers, exuberant, granulations, sinus, and fistula-in-ano.[3] Internally, it is used in the form of *kushta.* But before using *tutia* internally, it is always subjected to *tasfiya* or *shodhana*. Most of the raw drugs are procured from the mines, so there is a chance of

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impurities, toxicity, and heterogeneous qualities. Mixing of other substance and adulteration might have taken place commercially. *Shodhana* is indicated to eliminate all such impurities and to induce certain good qualities to enhance its pharmacotherapeutic properties.[7] This process results in the conversion of impure metal into pure or organometallic form, ready to be calcined.[8] Purified copper sulfate (*tutia musaffa*) in a dose of ¼ to 2 grains is beneficial in *ishal e muzmin* (chronic diarrhea) and *zaheer* (dysentery).[3] *Kushta tutia* (KT) is used by Hakims since ancient times in treatment of *aatishak* (syphilis), *sozak* (gonorrhea), *bawaseer* (haemorrhoids), *nasoor* (fistula),[9] *juzam* (leprosy), and *khunaq* (diphtheria).[10] Although this Unani compound formulation enjoy a very good reputation in treating several ailments efficiently, but still no scientific study has been carried out regarding its physicochemical evaluation. The National Formulary of Unani Medicine has mentioned 35 *kushtajat*;[11-14] whereas, in Physicochemical Standards of Unani Formulation[15-18] brief physicochemical standards of 22 *kushtajat* is mentioned, but KT has never been worked upon by Unani scholars. Therefore, present study was conducted to standardize KT on classical as well as modern parameters to establish the quality control parameters of KT which can be taken as standard for future references.

MATERIALS AND METHODS

Tutia was purchased from Shrinivasa Chemical Shop, Rajaji Nagar, Bangalore and was of analytical grade [Table 1].

Method of detoxification

Raw *tutia* was lavigated with water [Figure 1] until smooth paste was formed. Then it was dried on electrical heater at 100°C till whole of the *tutia* was converted into grayish white powder. The product obtained was *tutia musaffa* or detoxified *tutia* [Figure 2].[19]

Method of preparation of KT: KT was prepared as per method mentioned in *Kitab ul taklees*,[9] but with a slight modification, that instead of using the cow dung cakes it was prepared in Muffle furnace because the most important aspect of calcination procedure is heating. While preparing *kushta* from classical method, several precautions must be followed, for example, care of wind should be taken into consideration[9] as wind may cause uneven distribution of heat in cow dung cakes which result in incomplete preparation of *kushta* (*kushta kham*). So to convert this *kushta kham* into ideal *kushta* (*kushta kamil*) one more time the whole procedure of *kushta* making is repeated. This makes the process time consuming and uneconomical. Secondly, operation of heat using cow dung cakes requires labor which makes the procedure costlier. Muffle furnace is a better option over classical method preparation of *kushta* because being a closed chamber furnace gives better temperature control,[8,20] ease of preparation, simplicity of operation, and isolation of material being heated, saves time and labor. So, electric muffle furnace is better than conventional puta heating due to controlled heating system.[20]

Twelve gram *tutia musaffa* was kept in the crucible and closed with lid and placed inside furnace. For the operation of heat in furnace, a thermogram [Figure 3] given for preparation of *vanga bhasma* was followed,[21] because weight of cow dung cakes required for preparation of KT was equal to the weight of cakes used for the preparation of *vanga bhasma*. The peak temperature maintained was 1,008°C for 35 ± 5 min, above 800°C temperature was maintained for 20 ± 5 min and above 600°C temperature was maintained for 40 ± 5 min.[21] Heating was given only once. After self-cooling, KT [Figure 4] was removed carefully and stored in air tight bottle.

Table 1: Physical properties of raw *tutia*

Properties	Raw *tutia*
Nature	Crystalline lumps
Cleavage	Poor
Fracture	Conchoidal
Tenacity	Brittle
Transparency	Translucent

Figure 1: Raw *tutia* being triturated in mortar

Figure 2: *Tutia musaffa* (detoxified copper sulfate)

Physicochemical parameters

The prepared KT was evaluated for classical parameters of *kamil kushta* (ideal *kushta*) like floating test (*varitaratavam*),[22] grain floating test (*unama*),[23] finger test (*rekhapurnatvam*),[22] loss of metallic lustre (*nischandratva*),[23,24] wall stick test,[8] curd test (*dadhi pariksha*),[25,26] as well as modern scientific parameters like bulk density,[26] tapped density,[26] Hausner's ratio,[27] and Carr's compressibility index,[28] were evaluated in density tester (LABINDIA model no. 1025). pH in 1 and 10% solution[18] was observed by digital pH meter (Eutech instruments model no. 1544421). Loss of weight on drying[18] was measured in hot air oven (LABLINE, Anmatrix instrument technologies). Total ash,[29] acid insoluble ash,[30] water soluble ash,[30] water insoluble ash, and water soluble extractive value[31] were also assessed. The tests were performed on three batches to obtain mean ± standard error of the mean (SEM) value.

Floating test

If a small quantity of *kushta* is sprinkled on water surface then it should float on the surface.[22,32]

Grain floating test

Grain of rice, barley, etc., will float over the ideal *kushta* like a swan on a lake.[23,32]

Fineness test

On rubbing a small quantity of the *kushta* between the fingers, it should enter into the lines and creases of the fingers.[22,32]

Loss of metallic lusture

When visually examined preferably in presence of sunlight, no metallic luster should be observed.[23,24]

Wall stick test

On throwing on the wall, ideal *kushta* should stick to the wall.[8]

Curd test

Fifty gram of curd (pH - 3.5) was taken in Petri dish and 500 mg of KT sample was kept in it and observed for 48 h. No discoloration in the surrounding area was considered as proper *kushta* and greenish bluish discoloration if observed was considered as improper *kushta* or *kushta kham*.[25,33]

RESULTS AND DISCUSSION

The percentage of weight loss during detoxification of *tutia* was 35.22 ± 0.61%, while yield of KT was 11.21 ± 0.01 g [Table 2]. The color of KT was black. It was odorless, tasteless, lusterless smooth to touch, and very fine [Table 3]. Floating test, grain floating test [Figure 5], finger test [Figure 6], and wall stick test were positive [Table 4]. Curd test [Figure 7] showed no

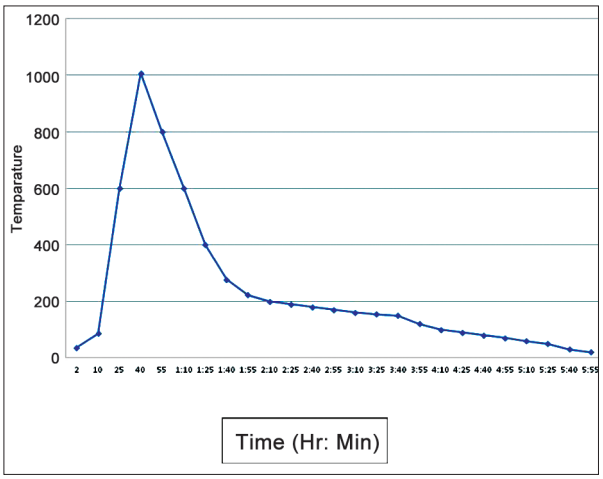

Figure 3: Thermogram followed for the preparation of *kushta tutia*

Figure 4: *Kushta tutia*

Figure 5: Grain floating test

Table 2: Detoxification (*tasfiya*) of raw *tutia*

Weight of raw *tutia* (g)	Quantity of water used for trituration (ml)	Duration of trituration (min)	Weight of *tutia musaffa* (g)	Weight loss (g)	% of weight loss during detoxification	Yield of *tutia* after heating
30	20	120	19.66	10.34	34.46	11.24
30	20	120	19.57	10.43	34.76	11.19
29	20	120	18.43	10.57	36.44	11.20

Table 3: Organoleptic properties of raw *tutia* and *kushta tutia*

Properties	Raw *tutia*	*Kushta tutia*
Color	Sky blue	Black
Odor	Odorless	Odorless
Taste	Metallic	Tasteless
Touch	Lumps	Smooth
Appearance	Shining	Lusterless

Table 4: Preliminary tests of *kushta tutia*

Tests	Observations
Floating test	Positive
Grain floating test	Positive
Fineness test	Very fine
Wall stick test	Positive
Finger test	Positive
Curd test	Passed

Table 5: Physicochemical tests of *kushta tutia*

Parameters	KT1	KT2	KT3	Mean±SEM
Bulk density (g/ml)	0.95	0.97	0.96	0.96±0.00
Tapped density (g/ml)	1.53	1.55	1.53	1.53±0.00
Hausner's ratio (HR)	0.62	0.62	0.62	0.62±0.00
Carr's index (%)	37.90	37.41	37.25	37.52±0.19
pH (1%)	5.62	5.62	5.63	5.62±0.00
pH (10%)	5.20	5.21	5.20	5.20±0.00
Loss of weight on drying (%)	0.09	0.08	0.08	0.08±0.00
Total ash (%)	95.85	95.67	95.74	95.75±0.09
Acid insoluble ash (%)	6.60	6.53	6.58	6.57±0.02
Water insoluble ash (%)	50.47	50.99	50.74	50.73±0.15
Water soluble ash (%)	45.38	44.68	45.00	45.02±0.20
Water soluble extractive (%)	0.81	0.87	0.89	0.85±0.02

KT1=*Kushta tutia* batch 1, KT2=*kushta tutia* batch 2, KT3=*kushta tutia* batch 3, SEM=Standard error of the mean

discoloration even after 48 h indicates absence of copper or copper sulfate.[26]

The mean value of bulk density and tapped density of KT were 0.96 ± 0.00 and 1.53 ± 0.00 g/ml, respectively [Table 5]. Bulk density is the mass per unit volume of a loose powder bed. The unit volume includes the spaces between the particles, and envelope volume of particles themselves. It is an essential parameter for process development of solid dosage manufacturing. It indicates the amount of powder that can fit in a space.[27] The tapped density represents the random dense packing of the material and is generally higher for regularly shaped particles (i.e, spheres) as compared to irregularly shaped particles such as needles.[27] The mean value of Hausner's ratio and compressibility index were 0.62 ± 0.00 and 37.52 ± 0.19%, respectively [Table 5]. The compressibility index is a measure of propensity of powder to consolidate. It is a measure of relative importance of interparticulate interactions. In a free flowing particle, these interactions are generally less significant; so bulk density and tapped density values are closer. For poorly flowing materials, there are frequently greater interparticle interactions, which results in lower bulk density and a greater difference between bulk and tapped densities. These differences in particle interactions are reflected as compressibility index. So, greater difference between bulk and tapped densities of KT indicated poor flowability which is further confirmed by the fact that compressibility index of KT was greater than 37. Powders having compressibility index more than 37 have very poor flowability.[27,34] pH in 1 and 10% solution were 5.62 ± 0.00 and 5.20 ± 0.00, respectively [Table 5]. It is mentioned that most of the *kushtajat* are alkaline.[35] It is also mentioned that pH value of aqueous solutions of metal oxides are basic, but acidic value of pH indicated that the end product in this study was not a content of metallic oxide (CuO), it was metallic sulfide (CuS). The mean percentage of loss of weight on drying was 0.08 ± 0.00% [Table 5]. Loss of weight on drying is a method to measure the loss in mass of the sample. This was done to determine the amount of water, all or a part of the water of crystallization, or volatile matter in the sample, which was removed during drying.[36] As the prepared *kushta* showed very less weight loss on drying, it could be assumed that the finished product was devoid of water and organic matters. The mean percentage value of the total ash, acid insoluble ash, water soluble ash, and water insoluble ash were 95.75 ± 0.09, 6.57 ± 0.02, 45.02 ± 0.20, and 50.73 ± 0.15%, respectively [Table 5]. High ash value shows the presence of very high inorganic content. Lower value of the acid insoluble ash suggests the greater physiological availability of the drug.[25] The mean percentage of the water soluble extractive value

Figure 6: Finger test

Figure 7: Curd test

was 0.85 ± 0.02% [Table 5] Extractive values help in the determination of the adulteration and is an index of the purity of the drug. In case of *kushta* extractive value is performed to extract out organic matter if present.[24] Low extractive values again confirm that *kushta* was prepared properly [Table 5].

CONCLUSION

Since there is lack of previous research work on standardization of KT, this study gave valuable information method of preparation and quality control parameters of KT on classical as well as modern techniques for the first time. However, further studies like clinical trials, animal studies, etc., are needed to be done on KT to convince the conventional society regarding safety and high efficacy of this Unani compound formulation.

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Professor Hakim Khaleefathullah Awarded Padmashree

Eminent scholar and teacher of Unani medicine Professor Hakim Syed Khaleefathullah was recently conferred 'Padmashree' by the President of India. Professor Khaleefathullah was founder member of 'Central Council of Research in Unani Medicine' and President of 'Central Council of Indian Medicine'. He was appointed as honorary physician to the President of India. He started Niamath Research Foundation for scientific research in Unani medicine. He was also honored with 'Lifetime Achievement Award' by M.G.R. Medical University, Chennai.

J-AIM Editorial Team congratulates Professor Khaleefathullah.