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FORMULATION AND CHARACTERIZATION OF A TRADITIONAL UNANI FORMULATION: *KUSHTA QALAI*

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ABSTRACT: *Kushta* literally means burning of drugs of metallic, mineral or animal origin into their ash. *Qalai* (tin) based Unani compound formulation *Kushta qalai* (KQ) is used since antiquity for the treatment of various ailments. Although it is used on consistent basis by Hakims but at present there are no scientific data available about its quality control parameters. So, in this research article an emphasis is given on preparation and physicochemical assessment of KQ using classical as well as modern scientific parameters with an eye towards its standardization. In finished product floating test, grain floating test, thumb finger test and fineness test were positive. The mean value of bulk density and tapped density were $1.32 \pm 0.00 \text{ gm/ml}$ and $2.24 \pm 0.02 \text{ gm/ml}$ respectively. Hausner's Ratio and Compressibility Index were 1.77 ± 0.02 and $44.15 \pm 0.86\%$ respectively. pH in 1% and 10% solution were 10.47 ± 0.00 and 11.17 ± 0.02 respectively. Loss of weight on drying was $0.019 \pm 0.00\%$. Total ash, acid insoluble ash, water soluble ash and water insoluble ash were $96.47 \pm 0.07\%$, $36.87 \pm 0.06\%$, $5.1 \pm 0.10\%$ and $91.64 \pm 0.20\%$ respectively. The water soluble extractive value was $0.34 \pm 0.00\%$. The results obtained might be treated as standard for future references.

INTRODUCTION: *Kushta* (organo-metallic preparation) is the finest class of solid dosage forms ¹ used in Unani system of medicine obtained by incineration of metals, minerals or animal origin drugs with herbal extracts. These are administered orally in minute quantity with appropriate vehicle to make them biocompatible and are used for broad range of therapeutics ².

They address different ailments depending upon their method of preparation. *Qalai* (tin) is one of the most popular metal renowned for its wide spread applicability since Vedic period ³, It is used internally in the form of *kushta*. It is known by diverse vernacular names like *rasas* in Arabic, *urziz* in Persian, *rang* in Hindi, *vanga* in Sanskrit and *kalai* in Gujarati. It occurs as silver like metal, softer than gold, harder than lead, malleable and sparingly ductile with little elasticity ⁴. There are two varieties of *qalai* viz. *khuraka* and *mishraka*, only the former being acceptable for therapeutic applications. Samples having the characteristics; bright white in color, soft, shiny, smooth, easily melts and heavy are identified as *Khuraka tin* ⁵.

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Qalai which is available in the market is almost pure and is not adulterated with any other metal. So it is not a necessary to purify it⁶ but reports are available that showed raw tin or improperly prepared *kushta qalai* may cause various ill effects like diabetes (*ziabetus*), leprosy (*juzam*), abdominal tumor, cardiac diseases (*amraz qalb*), spasmodic pain, piles (*bawaseer*), gout (*niqras*), goiter, cough (*sual*), dyspnoea (*usr-e-tanaffus*), weakness (*zof*) and vomiting (*qai*)⁷.

So in this study, before using *qalai*, it was subjected to *tasfiya* or *shodhana* (purification) which makes it compatible for the body⁸. *Qalai* is used chiefly in diseases of genitourinary organs⁹, blood and lungs. In the west oxide of tin is preferred as therapeutic agent for staphylococcal infections⁴. KQ is considered to be highly efficient in spermatorrhoea (*jiryana*), leucorrhoea (*sailan ur rehem*), gonorrhoea (*sozak*)^{10, 11}. It is also used as a remedy for premature ejaculation as an enhancer of viscosity of seminal fluid¹².

Earlier few clinical researches have been documented regarding the efficacy of KQ in certain diseases. But till date no systematic effort has been carried out on this preparation with respect to physicochemical classification, which is an integral part for drug standardization. So in this research work KQ was prepared according to the classical text of Unani literature by employing Electric Muffle Furnace as heating device for incineration. To ensure the proper preparation of *kushta*, classical standard tests were performed. Further KQ was subjected for modern quality control parameters of analysis. The results obtained are taken as standard for *kushta qalai* for future references.

MATERIALS AND METHODS: *Qalai*, *haldi* (*Curcuma longa*- root) powder and *aspgol* (*Plantago ovata*) were purchased from the local market in Bangalore.

Method of detoxification: Raw *Qalai* (**Fig. 1**) was melted and dipped in *aab sambhalu* (*Vitex negundo* decoction) in which a pinch of *haldi* (*Curcuma longa*) was added. The procedure was repeated three times¹³. By this procedure *qalai musaffa* (detoxified tin) was obtained (**Fig. 2**).



FIG. 1: RAW QALAI



FIG. 2: QALAI MUSAFFA (DETOXIFIED TIN)

Method of preparation of Kushta qalai: *Kushta* was prepared as per *Kitab ul taklees*¹⁴ with a minor modification, that instead of using the cowdung cakes it was prepared in Muffle Furnace because of ease of preparation and better temperature control. In 120 gm *aspgol* husk 150 ml water was added which was absorbed till morning (**Fig. 3**).



FIG.3 ASPGOL (PLANTAGO OVATA)

In the morning, two pellets of *aspgol* were made and 12 gm *qalai musaffa* was placed between them¹⁴. Heating was given as per Parmar et al. The peak temperature maintained was 1008°C for 35±5 minutes, above 800°C temperature was maintained for 20±5 minutes and above 600°C temperature was maintained for 40±5 minutes⁸.

After completion of heating *Kushta* was taken out from the furnace (**Fig. 4**) and was triturated separately in mortar and pestle and finished product obtained i.e. *Kushta qalai* (**Fig. 5**) was preserved in an air tight clean bottle.



FIG. 4 KUSHTA TAKEN OUT FROM FURNACE



FIG. 5 KUSHTA QALAI

Physico-chemical parameters: The prepared KQ 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, pH, loss of weight on drying at 105°C, total ash, acid insoluble ash, water soluble ash, water insoluble ash and water soluble extractive value. Quantitative estimation of tin and qualitative estimation of other metals was also performed. The tests were repeated thrice to obtain mean values.

Floating test: If a small quantity of *kushta* is sprinkled on water surface it should float on the surface.

Fineness test: On rubbing a small quantity of the *kushta* between the fingers it should enter into the lines on the fingers.

Loss of metallic lustre: When visually examined preferably in presence of sun light no metallic luster should be observed.

Bulk density and tapped density: LBD (Loose Bulk Density) and TBD (Tapped Bulk Density) were ascertained by following method¹⁵. 2 g KQ was placed into a 10 ml measuring cylinder and the initial volume was noted. Then tapping was done. The tapping was continued until no further change in volume was observed. LBD and TBD were calculated by the following equation.

LBD = Weight of the powder / volume of the packing. TBD = Weight of the powder / Tapping volume of the packing.

Hausner's ratio: Hausner's ratio was determined as the ratio between the tapped density to bulk density and was calculated by the following equation¹⁶.

Hausner's ratio = Tapped density / Bulk Density

Carr's index: Carr's compressibility index was calculated by¹⁷;

$$\text{Carr's index} = (\text{T.d} - \text{B.d}) / \text{T.d} \times 100$$

Where T.d=Tapped density, B.d- Bulk density

Loss of weight on drying at 105°C: 200 mg of *kushta* was spread homogeneously in petridish and was heated at 105°C then cooled in a desiccator and weighed. The process was repeated till two consecutive weights were constant. The percent loss in weight was calculated¹⁸.

Determination of pH in 1% solution and 10% solution

The pH value of 1% solution: 1 gm KQ was dissolved in 100 ml distilled water and filtered through whatman filter paper and pH was measured with digital pH meter¹⁸.

The pH value of 10% solution: 10 gm KQ was dissolved in 100 ml distilled water and filtered through whatman filter paper and pH measured with a digital pH meter¹⁸.

Determination of total ash: 2 g *kushta qalai* was incinerated in silica dish at a temperature not exceeding 450°C. The percentage of ash was calculated with reference to air dried drug¹⁹.

Determination of acid-insoluble ash: To the crucible containing total ash 25 ml of dilute HCl

was added. The insoluble matter was collected on Whatman ash less filter paper and washed with hot water until the filtrate is neutral. The filter paper containing the insoluble matter was transferred to the original crucible, dried on a hot-plate and ignited to constant weight. The residue was allowed to cool in a suitable desiccator for 30 minutes and weighed without delay. The content of acid-insoluble ash was calculated with reference to the air-dried drug²⁰.

Determination of water soluble ash: The ash was boiled for 5 minutes with 25 ml of water and insoluble matter was collected in a crucible or on an ashless filter paper and wash with hot water, and ignited for 15 minutes at a temperature not exceeding 450°C. The weight of the insoluble matter was subtracted from the weight of the ash; the difference in weight represents the water-soluble ash²¹.

Determination of extractive value: 4.0g of *kushta* was accurately weighed and put in a conical flask. 100 ml of water was added and weighed to obtain the total weight including the flask. It was shaken well and was allowed to stand for 1 hour. A reflux condenser was attached to the flask and boiled for 1 hour. 25 ml of the filtrate was transferred to a tared petridish and evaporated to dryness on water-bath then dried at 105°C for 6 hours. Then it was cooled in a desiccator for 30 minutes and weighed²².

Quantitative estimation of *qalai* (Tin): 0.5g of the *Kushta qalai* was taken in 250 ml beaker and dissolved in 25ml distilled water. 10ml concentrated Nitric acid was added into solution and the solution was digested, diluted and boiled on water bath. After cooling the solution was filtered through whatman 42 number filter paper. The precipitate was washed several times with 1% hot Nitric acid solution and dried. The precipitate was transferred along with filter paper to the weighed silica crucible which was then heated. The process of heating and cooling was repeated till the constant weight was obtained as Tin oxide²³.

Qualitative estimation of Iron compounds: 20-25 mg *kushta* was dissolved in distilled water; 1 ml of 5% w/v solution of potassium ferrocyanide was added. A dark blue colour precipitate appeared which was insoluble in 5 ml of 2M HCl²⁴.

Qualitative estimation of Aluminium compounds: 20-25 gm *kushta* was dissolved in 1 ml distilled water then 0.5 ml hydrochloric acid was added followed by 0.5 ml thioacetamide reagent.

Drop wise 2 M sodium hydroxide was added, a gelatinous white precipitate appeared that got dissolved in an excess of sodium hydroxide solution. Gradually 2 M ammonium chloride solution was added, the gelatinous white precipitate reappeared²⁴.

Qualitative estimation of Lead compound: 20-30 mg of *kushta* was dissolved in 1 ml of 5 M acetic acid and 5 ml of distilled water was added followed by 0.2 ml of potassium iodide. A yellow precipitate was formed²⁴.

RESULTS AND DISCUSSION: The colour of KQ was light grey. It was odorless, tasteless, lusterless, smooth to touch, and very fine (Table 2). Floating test (Fig. 6), grain floating test (Fig. 7), thumb finger test (Fig. 8) and fineness test were positive (Table 3). All these parameters satisfied the standards of a *kamil kushta* (*ideal kushta*) on classical parameters.



FIG. 6 FLOATING TEST



FIG. 7 RICE FLOATING ON WATER



FIG. 8 FINENESS TEST

The mean value of bulk density and tapped density of KQ were 1.32 ± 0.00 gm/ml and 2.24 ± 0.02 gm/ml respectively (Fig. 9). Density is defined as mass of a substance per unit volume²⁵. Bulk density largely depends upon particle shape. As the particle size increases bulk density decreases. High value of bulk density of KQ indicates very small particle size²⁶. The mean value of Hausner's ratio and Compressibility index were 1.77 ± 0.02 and 44.15 ± 0.86 % respectively (Table 4). Hausner's ratio of KQ was greater than 1.2 (Fig. 9), indicated poor flow properties²⁷.

More compressible the drug, less flowable the powder will be²⁵. Compressibility higher than 38 indicates very, very poor flowability of KQ^{16, 28}. pH in 1% and 10% solution were 10.47 ± 0.00 and 11.17 ± 0.02 respectively (Table 4). It is mentioned that most of the *kushtajat* are alkaline²⁹. It may also be concluded that the finished product was content of metallic oxides (stannous oxide) as the fact that pH value of water solutions of metal oxides is basic³⁰.

The mean percentage of loss of weight on drying was $0.019 \pm 0.00\%$ (Table 4). 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 $96.47 \pm 0.07\%$, $36.87 \pm 0.06\%$, $5.1 \pm 0.10\%$ and $91.64 \pm 0.20\%$ respectively (Table 4). High ash value (Fig. 10) shows the presence of very high inorganic content. The mean percentage of the water soluble extractive value was $0.34 \pm 0.00\%$ (Table 4). Extractive values help in the determination of the adulteration and is an index of the purity of the drugs. In case of *kushta* extractive value is performed to extract out organic matter if present³¹.

Less extractive values again confirm that *kushta* was prepared properly and finished product was free from organic material. The analytical results of *kushta qalai* showed that the mean percentage of the tin oxide in *Kushta qalai* was $90.04 \pm 0.04\%$ (Fig. 11). It indicated that majority of the *kushta* was in oxide form. Qualitative test showed presence of iron, aluminium and absence of lead (Table 5).

TABLE 1: PHYSICAL PROPERTIES OF RAW QALAI

Properties	Raw Qalai
Nature	Hard
Color	Silvery white
Lusture	Metallic
Streak	White
Transparency	Opaque

TABLE 2: ORGANOLEPTIC PROPERTIES OF RAW QALAI AND KUSHTA QALAI

Properties	Raw Qalai	Kushta Qalai
Color	White	Light grey
Odor	Odorless	Odorless
Taste	Tasteless	Tasteless
Touch	Smooth	Smooth
Appearance	Metallic	Lusterless

TABLE NO. 3: PRELIMINARY TEST OF KUSHTA QALAI

Floating test	Positive
Fineness test	Very fine
Wall stick test	Positive
Finger test	Positive

TABLE 4: PHYSICO-CHEMICAL TESTS OF KUSHTA QALAI (n=3)

Parameters	Mean \pm SEM
Bulk density (gm/ml)	1.32 ± 0.00
Tapped Density (gm/ml)	2.24 ± 0.02
Hausner's ratio (HR)	1.77 ± 0.02
Compressibility index (%)	44.15 ± 0.86
pH (1%)	7.35 ± 0.00
pH (10%)	7.22 ± 0.00
Loss of weight on drying (%)	0.019 ± 0.00
Total ash (%)	96.47 ± 0.07
Acid insoluble ash (%)	36.87 ± 0.06
Water insoluble ash (%)	91.64 ± 0.20
Water soluble ash (%)	5.1 ± 0.10
Extractive value (%)	0.34 ± 0.00
Tin oxide (%)	90.04 ± 0.04

Table 5: Qualitative estimation of Kushta Qalai

Iron	Present
Aluminium	Present
Lead	Absent

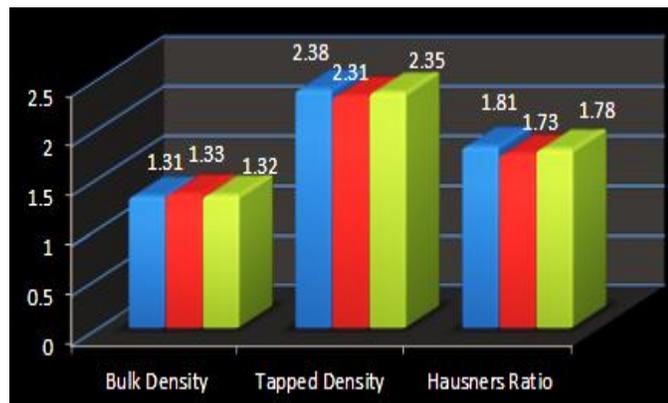


FIG. 9: BULK DENSITY, TAPPED DENSITY AND HAUSNER'S RATIO OF *KUSHTA QALAI*

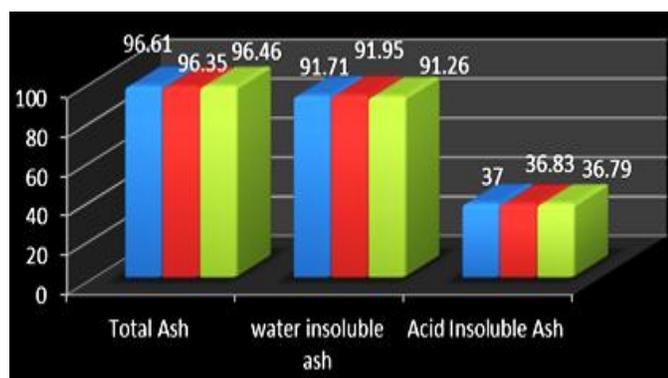


FIG. 10: TOTAL ASH, WATER INSOLUBLE AND ACID INSOLUBLE ASH OF *KUSHTA QALAI*

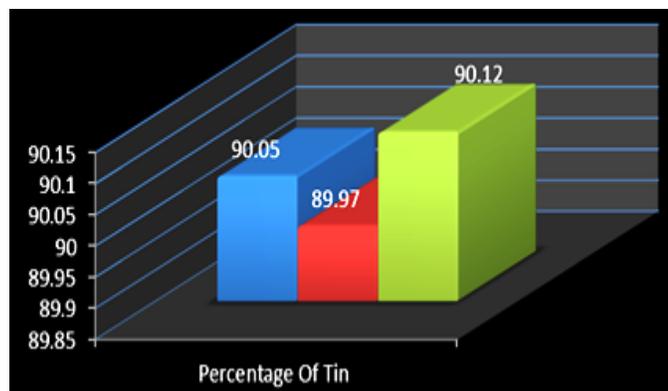


FIG. 11: QUANTITATIVE ESTIMATION OF STANNOUS OXIDE IN *KUSHTA QALAI*

CONCLUSION: Metal and mineral preparations used in Unani-Tibb are regularly subjected to scientific analysis and research laboratories now and then publish articles about the existence of heavy metals in Unani compound formulations. However, it is necessary to take into consideration that many metals are essential components of vital molecules of the body. This study gave detailed information about the method of preparation and standardization parameters of *kushta qalai* on classical as well as modern parameters.

As no standard physicochemical profile of *kushta qalai* is reported till date, the current data may be considered as standard for future studies. The analytical result shows the presence of Iron and Aluminum in KQ whereas only pure tin was taken for the preparation of *kushta*.

The method of preparation of KQ consists of the treatment of *qalai* with *aab sambhalu* (*Vitex negundo*), *haldi* (*Curcuma longa*) during detoxification process and *aspgol* (*Plantago ovata*) during calcination procedure therefore these herbs might be the source of iron and aluminum in the finished product. These two components might have synergistic effect as therapeutic agent instead of providing any harmful and undesirable effects.

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