



Research Article

Assessment of volatile oil content for quality control of commercial Samples of *Guggulu* viz. *Commiphora wightii* (Arn.): A critically endangered species

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Abstract

The oleo-gum resin of *Commiphora wightii* (Arn.), aka *Guggulu*, is a popular and widely used drug in Indian Ayurvedic pharma industries and in rest of the world. To get the best and desired clinical effects, authenticity and quality of raw drugs is essential. For *Guggulu*, along with other parameters, volatile oil determination can be regarded as simple and reproducible test for confirming valid samples of *Guggulu*. We investigated the current scenario of market availability of raw *Guggulu* exudate in Pune city, India and four market samples from local vendors were investigated. Volatile oil from the raw sample was determined by Clevenger's apparatus. It is noted that poor quality samples are available in markets, and these might be used for manufacturing of Ayurvedic products by some pharma industries, which further raises the quality control concern. To reduce the cost and make the product comparable with others in the market, quality of raw drugs must not be compromised. This study indicates the necessity of proper quality control of *Guggulu*. More cultivation and proper collection methodology of *Guggulu* is also needed to match the huge demand of this drug.

Keywords: Guggulu, Quality control, Ayurveda, *Commiphora wightii*, Volatile oil, Oleo-gum resin.

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Introduction

Guggulu is the oleo-gum resin obtained as an exudate from the tree of *Commiphora wightii* (Arn). *Guggulu* is classified as "Critically Endangered" by the International Union for Conservation of Nature (IUCN) due to overexploitation and habitat degradation (1). It is one of the most widely used drugs of Ayurveda, and hence its demand as a raw drug is high. In India *Guggulu* is used for preparing Ayurvedic medicines. *Guggulu* formulations are very commonly prescribed in Ayurvedic clinics all over. It is also used for preparing *dhupa* incense sticks, *havan* substances required for holy purposes. Trade of *Guggulu* is on rise, however the stocks are limited (2). Due to the high demand and limited supply, there is an issue about the quality of raw drug which we get in Indian markets.

The tree is found in arid areas of India, Bangladesh and Pakistan. It is a small bushy tree with thorny branches and produces yellowish coloured gum resin. The local people collect gum resin by making incisions on the trunk and major branches. The gum

resin in presence of the sunlight and heat exudes and gradually hardens in tears and frequently drops down. The incisions are made on the tree bark from November to January and the resin is collected through May to June (3). Being an exudate, the oleo gum resin is almost always contaminated with soil and organics materials like sticks, dry leaves. The *Guggulu* tree yields 250-500gm of resin each collection season. The aromatic fumes from burnt *Guggulu* are recommended for purification of air owing to its potent anti-microbial and anti-viral effects. *Guggulu* is praised in Ayurvedic literature as '*Devadhupa*' (incense for the almighty) (4). Its more than 100 formulations are mentioned in the ancient texts. It has a wide range of usefulness in indigenous medicine and is a part of many formulations due to its wide therapeutic actions. It is used as a rejuvenator, and it is used for management of arthritis, obesity, wound care, and fractures. *Guggulu* is known to possess multi-faceted biological roles viz. anti-inflammatory, anti-arthritis, anti-rheumatic, chondroprotective, anti-hyperlipidaemic, and anti-atherosclerotic activities, as evidenced from various experimental and clinical studies (5).

Guggulu being a significant drug for therapeutic purposes, it is made available from various sources in the country as well as imported from neighbouring countries. The difference between the yield of oleo-gum resin of *Guggulu* and its demand is very huge. High values and rising demands, improper methods of collection, uncontrolled harvesting and lack of cultivation decreased the number of *Guggulu* trees and are now categorized as threatened or

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critically endangered plant (6,7). To meet the demand, unfortunately *Guggulu* is adulterated with various other resins. Also, there is a variation in the appearance, colour, purity, and cost of various market samples of *Guggulu*. Hence, it is crucial to contemplate on the current scenario of market availability of *Guggulu*.

The present study is based on our observations regarding availability and quality of *Guggulu* in Maharashtra. The assessment of *Guggulu* samples was done on parameters mentioned in the Ayurvedic pharmacopoeia of India. While we studied the efficacy of *Abha Guggulu* (a herbal *Guggulu*-based formulation) to treat arthritis (8), we explored the market in Pune for availability of *Guggulu*. We came to know that there are issues regarding the quality of *Guggulu* available in markets. Hence this study highlights the facts regarding the availability and essential steps for quality control of *Guggulu* samples.

Materials and Methods

Four samples of *Guggulu* were purchased from local vendors in Pune city, India. All samples were older than one year as informed by the vendors. The samples were labelled as CW 1, CW 2, CW 3 and CW 4. The samples varied largely in their physical appearance (figure 1).

The samples were purchased from different vendors in Pune, the cost of each sample varied, as follows. The market cost of four samples (CW1, CW2, CW3, and CW4) was 800 INR per Kg, 1200 INR per Kg, 1000 INR per Kg, and 2500 INR per Kg respectively. The sample CW1 was having the lowest cost, while CW4 was costlier. The four samples have been deposited at herbarium collection at College of Ayurved, Bharati Vidyapeeth Deemed to be University, Pune.

The samples were tested for its physico-chemical characteristics as well as organoleptic parameters. The parameters used for the assessment of raw *Guggulu* were as follows, volatile oil, alcohol soluble extract, water soluble extract, ash value and pH. Volatile oil is one of the active components of the drug which is also responsible for its therapeutic effect. A genuine sample of *Guggulu* should contain not less than 1% of volatile oil (3,9). Volatile oil of *Guggulu* was extracted using Clevenger's apparatus.

Figure 1: Variable appearance of four market samples of *Guggulu* from market in Pune

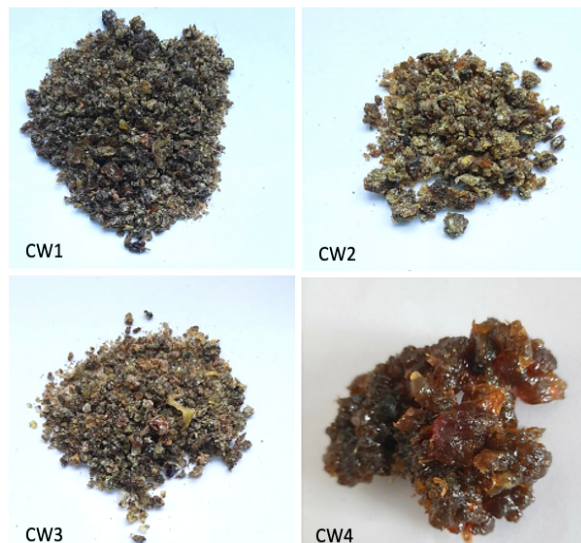
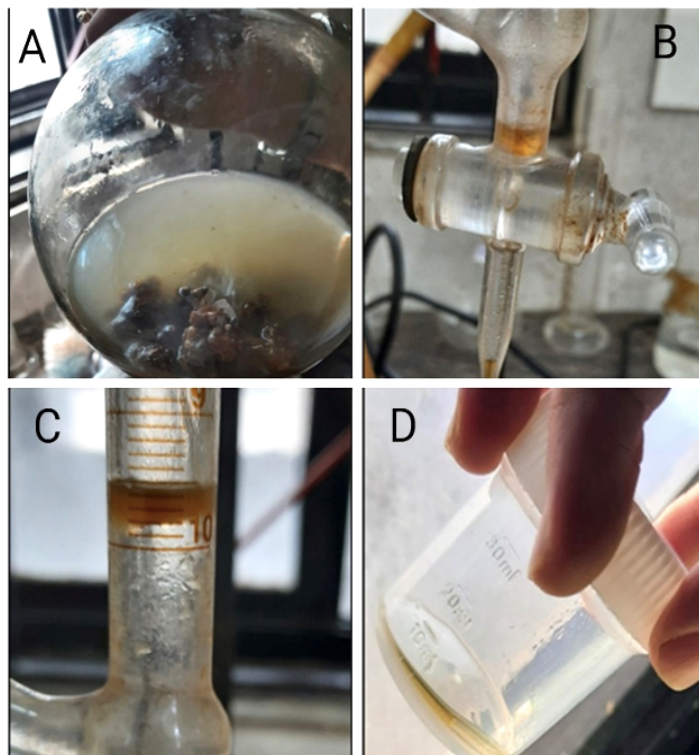


Figure 2: Volatile oil determination using Clevenger's apparatus. Figure 2A: sample with water in round bottom flask of apparatus. Figure 2B, 2C and 2D: volatile oil obtained from sample CW4



Crushed raw *Guggulu* in 50 gm quantity was mixed with 250 ml of water in a round bottom flask and was shaken until maximum of the *Guggulu* was dissolved in the water. The water turned milky white. The round bottom flask was placed in the heating mantle, the instrument was assembled, and heating was initiated.

Methodology of tests as per API (Ayurvedic Pharmacopoeia of India)

Water soluble extractive, alcohol soluble extractive, ash value, pH was carried out as per test procedures given in the Ayurvedic Pharmacopoeia of India (9).

Results

Results of authentication of *Guggulu* samples by organoleptic and physico-chemical parameters are reported in tables 1 and 2. CW4 resembles the *mahishaksha guggulu*, having reddish or brownish black colour (like buffalo's eye) as described in Ayurvedic literature. CW1, CW2, CW3 do not resemble with any of the type in Ayurvedic literature. These 3 are of inferior quality.

Table 1: Organoleptic analysis of samples of *Guggulu*

Parameters	CW1	CW2	CW3	CW4
Touch	Rough and dry	Rough Dry	Rough Dry	Soft sticky
Colour	Light brown	Light brown	Light brown	Honey brown
Taste	Mild bitter	Mild bitter	Mild bitter	Strong Bitter
Odour	Unspecific	Unspecific	Unspecific	Aromatic, specific Guggulu like
Foreign matter	8%	7%	8%	1%

Table 2: Physico-chemical Analysis of samples of Guggulu

Parameters	CW1	CW2	CW3	CW4	Standard values from API
Alcohol Soluble Extractive	24.2%	40.6%	37.8%	52.4%	NLT 27 %
Water Soluble Extractive	87.2%	55.2%	87.2%	90.8%	NLT 53 %
Volatile oil	Not detected	Not detected	Not detected	1.1%	NLT 1 %
Foreign matter	>4 %	>4 %	< 4 %	1%	NMT 4 %

NLT: 'not less than'; NMT: 'not more than.'

The presence of volatile oil was observed within 45 minutes in one of the samples. The measurable amount of volatile oil was collected gradually within the time span of 1.5 h from one of the samples of *Guggulu* viz CV4. From other three samples i.e. CW1, CW2, CW3, no volatile oil could be obtained.

The observations of organoleptic tests are mentioned in table 1. Ayurvedic guidelines emphasize on the parameters like touch, colour, taste and smell, which varied in case of all market sample.

Discussion

Guggulu is the oleo gum resin which has various therapeutic effects, and it is also used as a binding agent for Ayurvedic tablets which makes it an important and unique drug. There is a huge demand for *Guggulu*-based formulations viz. *Triphala Guggulu*, *Kaishor Guggulu*, *Yogaraj Guggulu*, *Amrutadi Guggulu* etc., in Ayurvedic clinical practice. Compelling scientific evidence validate ancient therapeutic claims and multiple traditional therapeutic uses of *Guggulu* (10). The raw *Guggulu* must be of authentic quality and strength. It should also be free from adulteration.

We procured 4 representative market samples of *Guggulu*, and it was found that there is huge variability in quality, purity, and strength of samples, their fragrance, appearance, cost in the market. It is noted that some of the samples had very poor aroma and visible adulteration. If such samples are being sold, it raises a concern of quality regarding this important raw material. This raises a concern of quality of formulations being consumed by patients who seek Ayurvedic treatment. Some other uses of this raw material are also there such as use in *havan samagri*, and incense. Its extracts are manufactured and marketed too.

To remove the physical impurities like sand, and organic impurities like leaves mixed with *Guggulu*, it is purified by dissolving in decoction of either of *Triphala* (equal parts of three myrobalans), *Vasa* (*Adhatoda vasica*), *Dashamula* (ten specific medicinal plant roots), or *Guduchi* (*Tinospora cordifolia*), followed by filtering. When good quality *Guggulu* is filtered, the remains are less. The filtered *Guggulu* is dried and stored for further formulation preparation. More the impurities, less is the yield. *Ghee* is added to prevent the loss of oil while heating with *Triphala* decoction. This preserves the pleasant fragrance even after purification process. Temperature is recommended to be maintained throughout up to 70°C to prevent the loss of oil. Volatile oil gets merged with ghee and hence it is preserved in the formulation, by minimising loss during formulation process (11). Volatile oil is an important parameter, which can be used as a main quality control parameter for ensuring the quality of *Guggulu*, with organoleptic study. Clevenger's apparatus can be

used for lab-based determination of volatile oil from sample of *Guggulu*. This is simple and cost-effective simple test that may be used by manufacturers.

To meet the rising demands, *Guggulu* is adulterated with physical impurities, other resinous material also finds place in market. The *Guggulu* found in market at lower prices is of questionable quality. To overcome this prevalent problem, the quality of *Guggulu* needs to be verified before purchasing and using it for making formulation. The analysis of each sample of *Guggulu* was done and volatile oil extracted with the help of an apparatus assembly called Clevenger was used in this study. The volatile oil in an ideal sample of *Guggulu* should be 1% in 100gm of sample. The sample which complied the standard was sample CW4, which was the costlier sample among the four.

Cunningham et al have reported regarding the global trade and rising demands of *Guggulu* (12). Due to rising demand, *Guggulu* has been over-harvested, more efforts for regeneration are needed. In the attempt to save the species efforts for cultivating the *Guggulu* plants should be done. This situation demands efforts for conservation and cultivation of *Guggulu*. The challenge of availability of *Guggulu* has been discussed previously as well (9,13). The soil and environment of Gujarat and Rajasthan is suitable for cultivation of *C. wightii*. In the past, there have been efforts for cultivation and conservation (14).

The recent conservation efforts by the government are commendable. Currently, forest department in Kutch is cultivating this medicinal plant in around 5,000-hectare area with grants specially received from AYUSH ministry's National Medicinal Plants Board, Government of India (11,15). This will improve the production and availability in a long run. Each plant requires eight years to mature before gum can be obtained from it. Once India was an exporter of *Guggulu*, however due to the rising demand and poor cultivation practices, currently India is dependent to import *Guggulu*. Hence the efforts for cultivation and scientific ways of collection of gum *Guggulu* from mature tree are necessary. A tree can yield gum for 50 years if proper method of collection is employed. Since the quality of *Guggulu* is variable in market, each manufacturer must ensure that they are using good quality *Guggulu* for medicine preparation.

Our study observed that three out of four samples from Pune market did not comply with quality standards in API. A good quality of *Guggulu* must yield not less than 1 % of volatile oil. The oleo gum resin should be aromatic, honey brown coloured and sticky in nature. Along with the organoleptic and other tests, volatile oil determination is a simple, quantitative, and valuable test to check the quality of raw *Guggulu*. This must be used by small and large manufacturers of Ayurvedic drugs to ensure quality of their products. The tests like HPLC and HPTLC profiling of *Guggulu* are useful for correct quantitative determination of phytoconstituents in *Guggulu* resin. Testing parameters involving use of higher instrumentation is also required. Estimation of Guggulusteron E and Z can be carried out in samples from all over. However, this is not possible for all small-scale industries, Ayurvedic physicians and academicians. We have investigated few samples only which were representative. Further a Pan India level study of sample of *Guggulu* from markets all over India should be carried out. This will give a clearer idea about the availability and quality of *Guggulu* in markets. Furthermore, stringent policy guidelines can be issued to industries for ensuring optimum quality of *Guggulu* resin which they are using.

Looking at the market availability, stringent quality check is essential to ensure good quality of oleo-gum resin of *Guggulu* used for formulating Ayurvedic drugs.

Conclusion and Future perspectives

This study was focussed on cost effective practical solution of determining quality of raw *Guggulu* by volatile oil assessment. This is a simple test which is possible for all small scale Ayurvedic industries to easily follow and ensure the quality of *Guggulu* they are using for manufacture of medicine. The outcome of the study suggests that very stringent quality check is essential to ensure quality of oleo-gum resin of *Guggulu* used for formulating Ayurvedic drugs.

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