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PENGENDALIAN MUTU SIMPLISIA DAN KAPSUL KUNYIT (*Curcuma longa* L.)

QUALITY CONTROL OF CRUDE DRUGS AND CAPSULES OF TURMERIC (*Curcuma longa* L.)

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ABSTRACT

Turmeric (*Curcuma longa* L.) is a medicinal plant commonly used in Thai Traditional Medicine (TTM), both in single use and formula. It has been known for its antioxidant activity and applied for management of many diseases. In this article, we reported some aspects of quality control of crude drugs and capsules of turmeric according to the standards set in Thai Herbal Pharmacopeia (THP). The results of our evaluations demonstrated that turmeric crude drugs met the criteria set in THP for microscopical identification, foreign matter (1.31%), loss on drying (6.89±0.174%), ethanol-soluble extractive (13.56%), water-soluble extractive (15.17%), and the profile of Thin Layer Chromatography (TLC) chromatogram. However, its volatile oil content (5.95%) was below the minimum value set in THP. The turmeric capsules met the criteria set in THP for loss on drying (8.64±0.093%), ethanol-soluble extractive (18.07%), water-soluble extractive (14.95%), and profile of TLC chromatogram.

Kata kunci: capsules, crude drugs, *Curcuma longa* L., quality control, standardization, turmeric.

Introduction

Turmeric (*Curcuma longa* L., Zingiberaceae), is widely cultivated in the tropics regions and commonly utilized in food, cosmetic, and dye industry. The medicinal potentials of this plant and its bioactive constituent, curcumin, has been studied and reviewed extensively. Turmeric is assigned for management of many diseases, such as cancer, inflammations, microbial infections, diabetes, arthritic, muscular disorders, biliary disorders, anorexia, cough, diabetic wounds, hepatic disorders, and sinusitis. Curcumin displays various pharmacological activities including antioxidant, antineoplastic, antiviral, antiinflammatory, antibacterial, antifungal, antidiabetic, anticoagulant, antifertility, cardiovascular protective, hepatoprotective, and immunostimulant (Lim, 2016; Meng et al., 2018; Omosa et al., 2017).

Turmeric is a well known ingredient in Thai Traditional Medicine (TTM) herbal formula. It is one of the components of the THR-SK010, a herbal formula used for wound treatment with a promising antibacterial activity against Methycillin Ressistant *Staphylococcus aureus* (MRSA) (Chusri et al., 2013). In the case of single use, turmeric is prescribed by local healers from Provinces of Pattani, Yala, and Narathiwat to treat urticaria and scabies (Neamsuvan et al., 2015). It is also used by 5% of cancer patients to relief of their dyspepsia (Poonthananiwatkul et al., 2015).

Quality control for efficacy and safety of the herbal medicine is important to be performed. Quality control and the standardization of herbal medicines involve several steps. The source and quality of raw materials, good agricultural practices, and manufacturing processes are certainly essential steps for the quality control of herbal medicines. For standard herbal medicines, quality refers to the status of the said drug and is based on three important pharmacopoeial definitions; they are identity, purity, and content of active constituents. Macroscopic and microscopic evaluation of voucher specimens are reliable reference sources to perform proper botanical identification. Purity evaluation includes ash values, contaminants, heavy metals, microbial contamination, aflatoxins, radioactivity, and pesticide residues. Analytical methods, such as photometric analysis, Thin Layer Chromatography

(TLC), High Performance Liquid Chromatography (HPLC), Gas Chromatography (GC), Mass Spectroscopy (MS) or GS/MS, can be employed to establish the constant composition of herbal preparations. Markers, if they are known, can be used for control purposes in the term of content. In other cases, where no active constituent or marker can be defined for the herbal drug, the percentage extractable matter with a solvent may be used as a form of content assay (Alamgir, 2017).

In this article, we reported the microscopical description, foreign matter, loss on drying, volatile oil, ethanol-soluble extractive, water-soluble extractive, and identification of curcuminoid in turmeric crude drugs. We also evaluated loss on drying, ethanol-soluble extractive, water-soluble extractive, and profile of TLC of a commercially available turmeric capsule.

Method

Material

The samples used were fresh, dried, and powder of dried turmeric provided by Laboratory of Pharmacognosy, Faculty of Pharmacy, Mahidol University. The turmeric capsule was brand "X" and was bought from a 7 Eleven store at Bangkok.

General

All the tests were conducted according to the methods in Thai Herbal Pharmacopeia (THP). The interpretation of obtained result was compared with the standards set in THP (THP, 2016).

Microscopical Description

The powder of dried turmeric was observed under light microscope to identify the diagnostic microscopical characters.

Foreign Matter

A hundred g of the dried turmeric was weighed and spread in a thin layer. The foreign matter was separated by hand as completely as possible, weighed, and the percentage present was calculated.

Loss on Drying

Two g of the powder of air-dried turmeric and capsules of turmeric was mixed and accurately weighed. Samples were put in tared glass-stoppered shallow weighing bottles that have been dried for 30 minutes under the same conditions to be employed in the determination. The samples were sidewise shaked gently to distribute them as evenly as practicable. The

loaded bottles were placed in the drying chamber, the stoppers were removed and leaved in the chamber. The samples were dried at the temperature of 105 ^{o}C for two hours. Upon opening the chamber, the bottles were closed promptly and allowed to come to the room temperature in a desiccator before weighing. The drying process was repeated until the constant weight was achieved.

Volatile Oil

Fifty g of fresh turmeric were sliced thinly and added with 200 mL of water. The mixture was then distilled in distillation apparatus for 5 hours. The obtained essential oil was carefully separated and its volume is noted. To estimate the equivalent volume of essential oil for 10 g dried turmeric as set in THP, we calculated the wet to dry turmeric ratio. A 79.02 g of the same sliced thinly turmeric were dried in a hot air oven until completely dry at temperature of 50 ^{o}C. The weight of dried turmeric was weighed. The wet to dry turmeric ratio was used for estimating the equivalent dry weight and volume of obtained essential oil from distilled turmeric, subsequently.

Ethanol-soluble Extractive

Two and half g of the powder of air-dried turmeric and capsules of turmeric were accurately weighed and macerated with 50,0 mL of ethanol in a closed flask for 24 hours, shaken frequently during the first 6 hours and then allowed to stand for 18 hours. The extract was filtered rapidly and 10.0 mL of the filtrate was evaporated to dryness in a tared dish and dry at 105 ^{o}C to constant weighed. The percentage of ethanol soluble extractive was calculated with reference to the air-dried turmeric.

Water-soluble Extractive

The powder of the dried turmeric and capsules of turmeric were conducted as those in ethanol-soluble extractive. The use of ethanol was replaced with water.

Identification with Thin Layer Chromatography (TLC)

The ethanolic extracts obtained from ethanol-soluble extractives were analyzed using silica gel G as the coating substance and mixture of 49 volumes of benzene, 49 volumes of chloroform, and 2 volumes of ethanol as the mobile phase. The chromatogram was visualized under daylight and UV light (254 and 366 nm).

Results and Discussion

Microscopic evaluation is important for the initial identification of herbs, as well as for identifying small fragments of crude or powdered herbs, and detection of foreign matter and adulterants (Alamgir, 2017). We did microscopic identification to ensure that the yellow powder of drug we evaluated was rhizome of turmeric. Observation on the powdered sample under light microscope resulted in identification of 5 diagnostic microscopical characters; they were parenchyma, vessel, unicellular trichome, starch granules, and cork (Figure 1). In THP, there are 7 microscopical characteristic in powder of turmeric for its identification. The two fragments that we could not manage to find in the turmeric crude drugs were spiral vessel and epidermis.

Foreign matter can be consisting of parts of the herbal material or materials; any organism, part or product of an organism other than those named with the limits specified for the herbal material concerned; or mineral admixtures not adhering to the herbal materials, such as soil, stones, sand and dust. Herbal materials should be entirely free from visible signs of contamination by moulds or insects, and other animal contamination, including animal excreta. No abnormal odor, discoloration, slime or signs of deterioration should be detected (WHO, 2011).

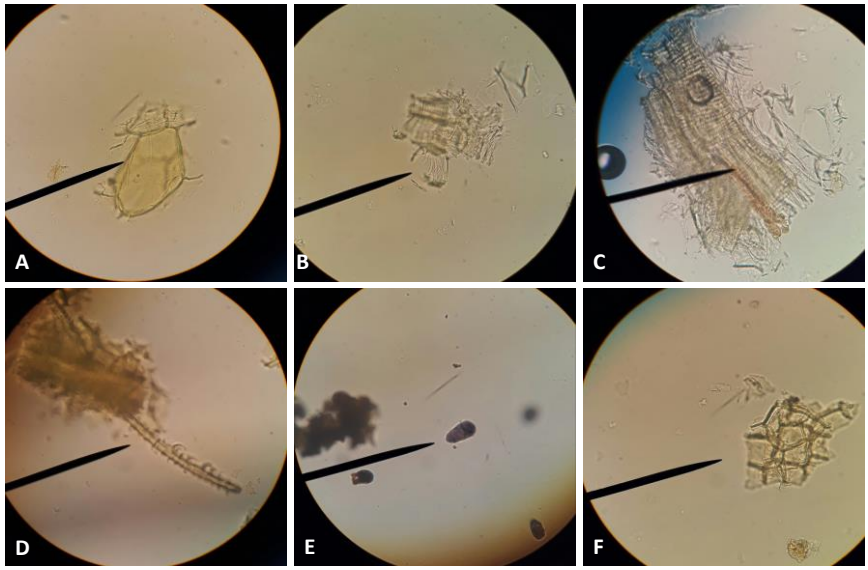

Figure 1. Microscopical characteristic of powdered dried curcumin (A) parenchyma, (B) reticulated vessel, (C) vessel, (D) unicellular trichome, (E) starch granules, and (F) cork in surface view.

Foreign matter analysis is performed to ensure the purity aspect of the turmeric used in this study. The foreign matter in the dried turmeric was 1.31% (Table 1). This value is still in the range permitted by THP (less than 2%).

The test for loss on drying of an herbal medicine determines both water and volatile matter. The existence of significantly high moisture in a crude drug will allow the microbial growth when the crude drug is stored. Hence, loss on drying of a crude drug is directly related to its stability and consequently with its shelf life. The lower the moisture content, the higher will be the stability of that drug and chance of microbial growth will be less and vice versa (Alam and Saqib, 2015). The loss of drying of turmeric sample was 6.89%. This level of moisture is still allowed by THP, that set water content for a crude drug should not more than 10.0% (THP, 2016). The moisture content of turmeric capsules was higher (8.64%) than that of the turmeric powder. However, both samples comply with THP standard (Table 1).

Essential oil content in a crude drug is a part of its content aspects. For many plants, essential oils are responsible for their bioactivity (Cimanga et al., 2002). Determination of essential oils by steam distillation is a special form of chemical assay to determine the quality of a crude drugs (Alamgir, 2017). Dried turmeric that was intended for medicinal purposes should contain essential oils not less than 6.0% (THP, 2016). In THP, the test should be subjected to 10 g of dried rhizome of samples, somehow we used fresh one to be distilled. We used 50 g of fresh turmeric rhizome that resulted in 0.35 mL of essential oil. To meet the determination of essential oil in THP, we dried the same fresh turmeric to calculate the ratio of fresh to dry rhizome. The ratio (79.02 to 9:29) was then utilized to calculate the equivalent essential oil content for 10 g of dried rhizome as in THP. The essential oil content of turmeric crude drugs was 5.95% (Table 1), which was below the standard set in THP.

Solvent-soluble extractive value determines the amount of active constituents extracted with solvents from a given amount of herbal material. It is employed for materials for which as yet no suitable chemical or biological assay exists (WHO, 2011).

Chemical evaluation of turmeric has been established with curcumin as the marker. However, THP set the standards for ethanol-, water-, and chloroform-soluble extractive value. Ethanol-soluble extractive value of turmeric crude drugs was lower than that of turmeric capsule (Figure 2). However, both samples were within the acceptance criteria, as THP set ethanol-soluble extractive value is not less than 10.0%. Water-soluble extractive values of crude drugs and capsules of turmeric were equal, 15.17 and 14.95%, respectively (Figure 2). These value were above the minimum water-soluble extractive set in THP, that is not less than 9.0% (THP, 2016).

Table 1. The identity, purity and content aspects in quality control of crude drugs and capsules of turmeric

Parameters	**Obtained Values (%)**		Standards in THP
	Crude Drugs	**Capsules**	
Foreign matter	1.31	-	not more than 2%
Loss on drying	6.89	8.64	not more than 10.0%
Essential oil content	5.95	-	not less than 6.0%

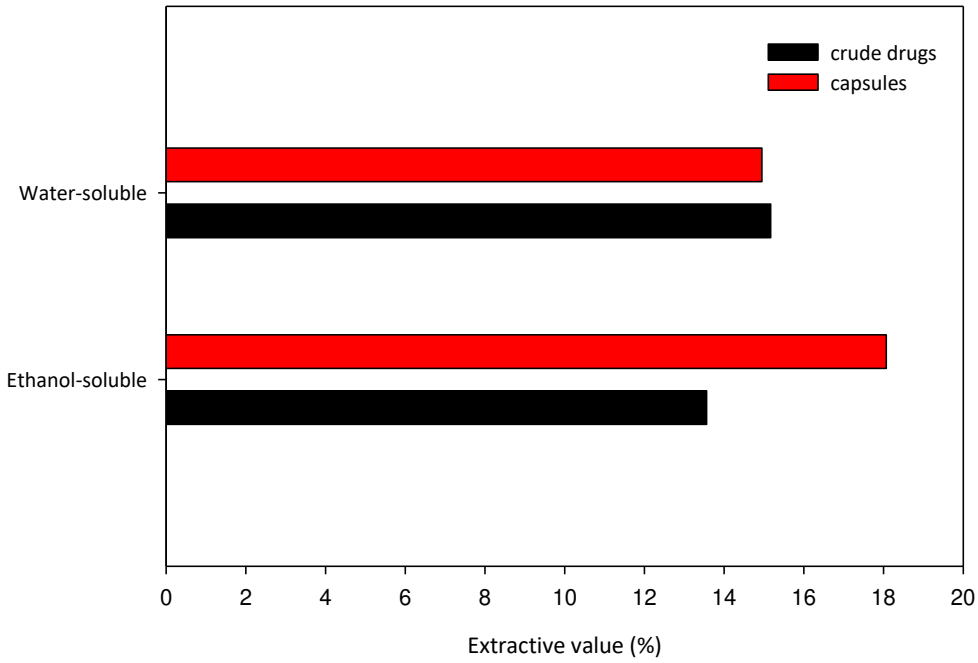

Figure 2. Comparison of water- and ethanol-soluble extractive values of crude drugs and capsules of turmeric.

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TLC of the extracts was employed to ensure the aspect of content in standardization. Curcumin is the definite chemical constituent to which biological or pharmacological activity of turmeric is attributed (Omosa et al., 2017) Curcumin in turmeric is found as the mixture with two other derivates (desmethoxycurcumin and bisdesmethoxycurcumin) and this mixture is commonly referred as curcuminoid. We used curcuminoid as the standard in TLC. Visualizations under direct light and UV lamp at wavelength of 254 and 366 nm confirmed the presence of those 3 compounds, with hRf value of 26, 14.6, and 6.67, respectively. Those 3 compounds were also identified in extracts of turmeric powder and turmeric capsule. Other than those 3 compounds, a spot with hRf value of 68 was also detected in both powdered and capsule turmeric under UV lamp at the wavelength of 254 nm (Figure 3). This profile showed that both samples contained curcumin as the bioactive compound that was responsible for their bioactivity.

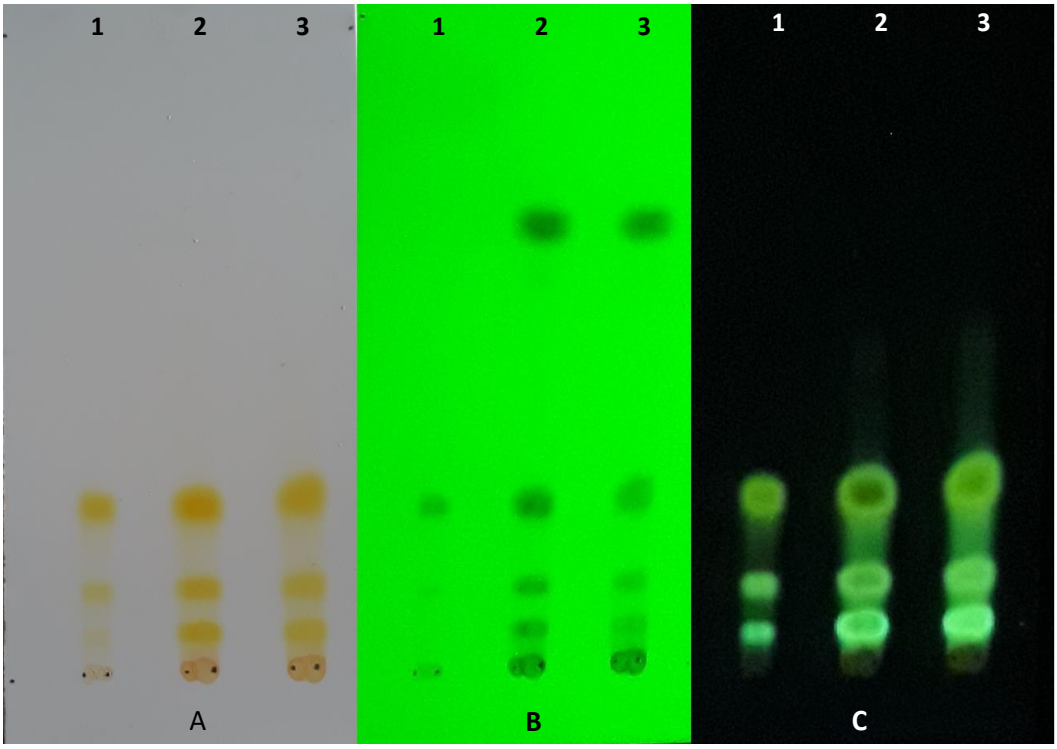

Figure 3. Chromatogram of ethanolic extract of turmeric (1) curcuminoid standard, (2) extract of turmeric powder, (3) extract of turmeric capsule, (A) detection under direct light, (B) detection under UV light at 254 nm, (C) detection under UV light at 366 nm.

Conclusion

Both crude drugs and capsules of turmeric analyzed in this study demonstrated good quality with most of values of quality parameters are within acceptance criteria of THP. The turmeric crude drugs meet the criteria for microscopical identification, foreign matter, loss on drying, ethanol- and water-soluble extractive values, and also the profile of TLC chromatogram. However, its volatile oil was below the minimum value set in THP. The turmeric capsules meet the criteria for loss on drying, ethanol- and water-soluble extractive value, as well as profile of TLC chromatogram.

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