Moisture Adsorption Isotherms of *Orthosiphon Stamineus* Leaves

Sriyana Abdullah, Ibni Hajar Rukunudin, Abd Razak Shaari, Muhammad Syarhabil Ahmad, and Rashidah Ruslan

Abstract—A herb, Orthosiphon stamineus is a popular traditional medicine for centuries to treat many human illness especially diseases of urinary tract and kidney stones. Moisture adsorption study of O. stamineus leaves is important because this herb is stored and consumed in its dried form. The behavior of dried leaves under different conditions during storage can have an impact on the final quality of end product. In this research, moisture adsorption isotherms were developed at 2 different temperatures of 5 °C and at ambient (30 °C) and at 6 levels of relative humidity (RH) ranging from 11.3 % to 98.5 % by using the static gravimetric method. Microbial growth on the samples were also observed. The moisture adsorption of O. stamineus was found to be of type III isotherm (J-shaped). The equilibrium moisture content (EMC) is higher at low temperature compared to the higher temperature at all RH levels. For example, at RH level of 98.5 % the EMC is 0.58 g water/g dry matter (dry basis- d.b) and 0.48 g water/g dry matter (d.b) at 5 °C and 30 °C respectively. Microbial growth was observed to present for samples at RH more than 75 % as early as day 6th of experiment at ambient temperature (30 °C) whereas for samples at 5 °C microbial growth was observed to present at RH of 98.5 % after 54 days of experiment.

Index Terms—Equilibrium moisture content, isotherm, microbial growth, moisture adsorption.

I. INTRODUCTION

Orthosiphon stamineus (*O. stamineus*) or popularly known in Malaysia as misai kucing is an herbal species indigenous to the South-East Asian region. This herbal plant belongs to a genus in the family of *Lamiaceae*. The plant can grow to about 1.2 m in height and it can be harvested in 2 to 3 months after transplanting. Although it was first introduced to the European consumers as an herbal tea in the 20th century, this species became popular, particularly in Indonesia and Malaysia in the last few decades [1]. It is believed that the bioactive compounds contained in the leaves of *O. stamineus* exert the effects as an antiallergic, antihypertensive, anti-inflammatory [2], antioxidant [3], [4], and diuretic properties [5]. It was reported that the phenolic compounds such as caffeic acid derivatives, lipophilic flavones, flavonol glycosidase, and polymethoxylated

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Sriyana Abdullah, Abd Razak Shaari, Muhammad Syarhabil Ahmad, Rashidah Ruslan, and Ibni Hajar Rukunudin are with the School of Bioprocess Engineering, University Malaysia Perlis, 02600 Arau Perlis Malaysia (e-mail: sriyana@unimap.edu.my, ibnihajar@unimap.edu.my, razakshaari@unimap.edu.my, syarhabil@unimap.edu.my, rashidahruslan@unimap.edu.my). flavones exhibited some medicinal properties [3]. It is now accepted that some of the polymethoxylated flavones such as sinensetin (SEN), eupatorin (EUP), and 3-hydroxy-5, 6, 7, 4-tetramethoxyflavone (TMF) together with a caffiec acid derivative compound such as rosmarinic acid (RA) contained in the leaves extract, are the marker compounds of *O. stamineus*. Currently, *O. stamineus* based products are being commercialized as food supplements in the forms of tea sachets, drinks, raw herbs, tablets, and capsules.

Storage of O. stamineus dried raw material for commercialization purpose becomes a crucial process to ensure good quality herbal healthcare products. The knowledge of moisture sorption is one of the most important considerations to provide safe moisture content level at a particular temperature and relative humidity conditions. Safer moisture content level is important to prevent the herbs microbial damage and bioactive compounds from degradation during long term storage period. From the literature, the study of moisture sorption isotherms had been carried out in many agricultural materials such as orange peel and leaves [6], corn stover components [7], garden mint leaves [8], safflower petals, and tarragon [9]. However, no information on sorption isotherms has been reported for local herbs especially O. stamineus. Thus this study is an attempt to establish the moisture adsorption isotherms on locally harvested O. stamineus leaves to provide references on the storage ability of local herbs. The objective of this work is to developed moisture adsorption isotherms for O. stamineus leaves at 5°C and at ambient temperature (30°C) at six relative humidity (RH) levels and to observe microbial growth on the samples.

II. MATERIAL AND METHODS

A. Raw Material

The raw material was harvested from a production plot at the School of Bioprocess Engineering's greenhouse, University Malaysia Perlis (UniMAP), Malaysia. The raw material was harvested at a maturity stage of 2 months after transplanting. After harvesting, the plants were washed to remove dirt and dust before the leaves were separated from the stems. The fresh leaves were kept in a cold room within 48 hours before further use.

B. Determination of the Equilibrium Moisture Content

1) Preparation of saturated salt solutions

Moisture sorption data were determined using the static gravimetric method at about 5°C and at ambient temperature of about 30°C. Six saturated salts solutions (Table I),

representing the relative humidity (RH) values ranging from 11.3 % to 98.5 % were prepared by dissolving an appropriate quantity of salt in distilled water at both temperatures. The salt solutions were poured into six desiccators to about one-quarter of the depth of the lower parts as shown in Fig. 1. A sufficient amount of salt crystal was put in the solution to ensure the saturated condition inside the desiccators. A porcelain plate was placed in each desiccator (Fig. 1) to hold the petri dishes containing the samples. The desiccators were tightly closed using high vacuum grease and were placed in a cold room maintained at 5 °C and at ambient condition of 30 °C for 24 hours in order to stabilize them.

TABLE I: DATA OF SALT SOLUTION AND RELATIVE HUMIDITY (%) AT $5^{\rm o}{\rm C}$ and $30^{\rm o}{\rm C}$ [10]

	Salt	Relative Humidity (%)	
		5 °C	At ambient (30 °C)
1	Lithium Chloride	11.26	11.28
2	Magnesium Chloride	33.60	32.44
3	Magnesium Nitrate	58.86	51.40
4	Sodium Chloride	75.65	75.09
5	Potassium Chloride	87.67	83.62
6	Potassium Sulfate	98.48	97.00



Fig. 1. The desiccators.

2) Preparation of herb samples

The harvested fresh leaves of 81% initial moisture content (wet basis – w.b) were dried at 60° C for 48 hours with final moisture content of 4.5% (w.b) and used as samples for adsorption study [6]. The dried samples range from 0.4432 to 0.6416g were weighed and placed in clean glass petri dishes. The petri dishes were then placed on the ceramic plate inside the six desiccators. Sample weights were taken every two days until no discernible weight changes recorded at difference less than 1 mg between three consecutive readings were obtained. The samples reached an equilibrium after 8 weeks. The equilibrium moisture content (EMC) was then determined by the oven method at 105° C for 24 hours [11].

3) Observation of microbial growth

The microbial contamination were observed every two days and recorded when the samples registered mold growth. However no microbial count analysis was carried out.

III. RESULTS AND DISCUSSION

A. Moisture Adsorption Isotherms

The equilibrium moisture content (EMC) of *O. Stamineus* leaves were plotted against relative humidity (RH) levels ranging from 11.3 % to 98.5 % at the temperature of 5 °C and 30 °C as shown in Fig. 2. The moisture adsorption curve is

classified to have type III (J-shaped) isotherm according to [12]. The type III isotherm is characterized by a product which can hold small amounts of water at low RH levels and large amounts of water at large RH levels. It is also describes a multilayer adsorption phenomenon. A similar trend of type III adsorption behaviour was also reported for orange leaves at 40° C, 50° C and 60° C [6].



As it can be seen from Fig. 2, EMC is higher at low temperature (5°C) compared to higher temperature (30°C) at all RH levels. For example, at RH level of 11.3 %, the EMC is 0.053g water/g dry matter (dry basis- d.b) at 30°C while at 5 C, the EMC is 0.075g water/g dry matter (d.b) and at RH level of 98.5% the EMC is 0.58 g water/g dry matter (d.b) and 0.48 g water/g dry matter (d.b) at 5°C and 30°C respectively. This behavior may be explained by considering excitation states of water molecules. When temperature increases, the molecules are in an increased state of excitation which increases their distance apart and decreases the attractive forces between them. This leads to a decrease in the degree of water sorption at a given water activity with increasing temperature [13].

B. Observation of Mold Growth

Growth of mold on the samples was observed throughout the experiment. The global food stability map can be used to explain the activity of mold in food [14]. From the stability map the mold starts to grow at RH of > 60 %. The observation showed that at 30°C, the mold growth can be seen to occur as early as the day 6th after the experiment started particularly at RH more than 75%. However, at low temperature of 5°C, the mold growth was observed only after 54 days of experiment at relative humidity of 98.5%. The storage of herbs at ambient condition (30°C) and at RH of more than 75% may risk the herbs from microbial spoilage as the mold can be observed even after the day 6^{th} of storage. At low temperature, the mold activity is very slow resulting in late appearance of the mold in the samples at 5°C. However, a proper packaging technique has to be applied if the samples were to be stored at the ambient conditions especially in the equatorial climate countries.

IV. CONCLUSIONS

The moisture adsorption isotherms of the *O. stamineus* leaves were successfully studied. From the experimental results, some conclusions can be made; the moisture isotherms of *O. stamineus* leaf exhibited a type III (J-shaped)

as classified by Brunauer - Emett -Teller (BET) classification for both temperatures. Mold growth was observed in samples at the RH of 75% and 98.5% at ambient temperature (30°C) and 5°C respectively. The mold can easily grow in the samples at ambient temperature as compared to the samples at low temperature because ambient conditions may provide conducive environments for microorganisms' activity.

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REFERENCES

- N. L. T. I. B. Jaganath, M. Chinna, and R. A. Rahman, *Herbs The green Pharmacy of Malaysia*, Malaysia: Vinpress Sdn. Bhd., 2000, pp. 126.
- [2] S. Awale, Y. Tezuka, A. H. Banskota, and S. Kadota, "Siphonols A-E: Novel nitric oxide inhibitors from *Orthosiphon stamineus* of Indonesia," *Bioorganic & Medicinal Chemistry Letters*, vol. 13, pp. 31-35, 2003.
- [3] G. A. Akowuah, Z. Ismail, I. Norhayati, A. Sadikun, and S. M. Khamsah, "Sinensetin, eupatorin, 3'-hydroxy-5, 6, 7, 4'-tetramethoxyflavone and rosmarinic acid contents andantioxidative effect of Orthosiphon stamineus from Malaysia," *Food Chemistry*, vol. 87, pp. 559-566, 2004.
- [4] G. A. Akowuah, Z. Ismail, I. Norhayati, and A. Sadikun, "The effects of different extraction solvents of varying polarities on polyphenols of *Orthosiphon stamineus* and evaluation of the free radical-scavenging activity," *Food Chemistry*, vol. 93, pp. 311-317, 2005.
- [5] Y. Adam et al., "Diuretic properties of Orthosiphon stamineus Benth," Journal of Ethnopharmacology, vol. 124, pp. 154-158, 2009.
- [6] A. K. Bejar, N. B. Mihoubi, and N. Kechaou, "Moisture sorption isotherms - Experimental and mathematical investigations of orange (Citrus sinensis) peel and leaves," *Food Chemistry*, vol. 132, pp. 1728-1735, 2012.
- [7] C. Igathinathane, A. R. Womac, S. Sokhansanj, and L. O. Pordesimo, "Sorption equilibrium moisture characteristics of selected corn stover components," *Transactions of the ASAE*, vol. 48, 2005.
- [8] K. J. Park, Z. Vohnikova, and F. P. R. Brod, "Evaluation of drying parameters and desorption isotherms of garden mint leaves (Mentha crispa L.)," *Journal of Food Engineering*, vol. 51, pp. 193-199, 2002.
- [9] S. Kaya and T. Kahyaoglu, "Moisture sorption and thermodynamic properties of safflower petals and tarragon," *Journal of Food Engineering*, vol. 78, pp. 413-42, 2007.
- [10] L. Greenspan, "Humidity fixed points of binary saturated aqueous solutions," *Journal of Resources Natural Bereau of Standards, Section A. Physic and Chemistry*, vol. 81, pp. 89-105, 1977.
- [11] Moisture Content in Plant in Official Method of Analysis: 930.04, Association of Official Analytical Chemists (AOAC)-1990.
- [12] S. Brunauer, *The adsorption of Gases and Vapors*, NJ: Priceton, Priceton University Press, 1945,
- [13] J. V. Garcà a-Pérez, J. A. Cárcel, G. Clemente, and A. Mulet, "Water sorption isotherms for lemon peel at different temperatures and isosteric heats," *LWT - Food Science and Technology*, vol. 41, pp. 18-25, 2008.
- [14] T. P. Labuza, S. R. Tannenbaum, and M. Karel, "Water content and stability of low moisture and intermediate moisture foods," *Journal of Food Technology*, vol. 24, pp. 543-550, 1970.



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