Moisture adsorption isotherms of dried *Ficus deltoidea* herbal leaves

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**Abstract.** *Ficus deltoidea* is a popular traditional medicine that widely used to treat various kind of diseases because of its high number of phenolic contents. In order to meet the demand of the high usage in the market, it is very important to study the behaviour of the dried herbal leaves to prolong the shelf life during storage. Hence, the study of moisture adsorption isotherm is one of the crucial parameters to minimize the deterioration of the quality. The moisture adsorption data of dried *Ficus deltoidea* leaves were monitored using gravimetric static method. Six different saturated salts such as potassium chloride, sodium chloride, sodium nitrate, magnesium nitrate, magnesium chloride and potassium acetate were utilized in this experiment. The equilibrium relative humidity range was setup between 23 to 86%. Three different temperatures of 8, 30, 40 °C were used as a treatment of experiment. From the final results, it is clearly claimed that the moisture adsorption isotherm data exhibited J shaped and type III of Brunauer, Emmet and Teller (BET) classification. In addition, the results of experimental data as shown that the best mathematical model fitted to Peleg model compared to GAB and Oswin models.

1. Introduction

*Ficus deltoidea* is an indigenous plant in Malaysia and locally known as mas cotek. It is still commonly used as traditional medicinal treatments to treat various kind of ailments [1]. At present, the extract of *F. deltoidea* is being invented to form several of commercial products including pharmaceutical medicine and food supplement. The chemical content in *F. deltoidea* show signs of medicinal effects such as antioxidant [2], antidiabetic [3] and anti-inflammatory [4]. The best features of *F. deltoidea* leaves extracts that it has superior amount of phenolic compounds and antioxidant [5].

To guarantee good quality herbal healthcare products, the storage of dried herbal material becomes an important process for commercialization purposes. The data of moisture sorption is one of important factors to provide safe moisture level at a specific temperature and relative humidity conditions [6]. The moisture sorption isotherm is an valuable instrument to expect potential variations in food constancy, determining storage condition system, and packaging selection [7]. Therefore, the objectives of this research were to determine the moisture sorption isotherms of *Ficus deltoidea* dried leaves at 8, 30, and 40 °C, and to validate the best model that describe the adsorption isotherms of *F. deltoidea*. 
2. Materials

2.1. Material preparation
Fresh *Ficus deltoidea* leaves were freshly picked at Institute of Sustainable Agrotechnology (INSAT), Sungai Chucuh, Perlis. Impurities were then removed by cleaning all the leaves with water thoroughly and patted with dried clean tissue paper to remove excess water. The leaves of *F. deltoidea* were dried using laboratory oven at 40 °C until the weight of leaves was constant.

2.2. Salt preparation
Static gravimetric static method were used according to Greenspan’s method [8]. Six saturated salt solutions were used in different desiccators to get constant relative humidity settings. Table 1 below showed the saturated salt used in this method. Saturated solutions were placed in an amount to occupy a space at the bottom of the desiccator as in Figure 1. Hygrometer were placed in each of the desiccator to monitor the relative humidity readings. A small quantity of solid salt was added to make the solutions stay saturated to make sure the readings of relative humidity did not fluctuate.

| No | Salt                | Relative humidity (%) |
|----|---------------------|-----------------------|
|    |                     | 8 °C | 30 °C | 40 °C |
| 1  | Potassium acetate   | 23.38 ± 0.53          | 23.28 ± 0.24 | -    |
| 2  | Magnesium chloride | 33.47 ± 0.24          | 32.44 ± 0.14 | 31.60 ± 0.13 |
| 3  | Magnesium nitrate  | 57.36 ± 0.33          | 57.40 ± 0.24 | 48.42 ± 0.37 |
| 4  | Sodium nitrate      | 77.53 ± 0.45          | 73.14 ± 0.31 | 71.00 ± 0.34 |
| 5  | Sodium chloride     | 75.67 ± 0.22          | 75.09 ± 0.11 | 74.68 ± 0.13 |
| 6  | Potassium chloride  | 86.77 ± 0.39          | 84.34 ± 0.26 | 82.32 ± 0.25 |

2.3. Experiment setup
5 grams of *F. deltoidea* dried leaves were placed in petri dish and put inside desiccator with three replications in 6 different desiccators and placed in three specific temperatures (8, 30 and 40 °C). The changes in samples weight were measured every two days by using analytical balance until the sample weight reach constant weight. Constant weight were defined as there were no obvious weight changes recorded at differences more less than 1 mg between three following readings. The equilibrium moisture content (EMC) was established by the oven method dried at temperature 105 °C.
2.4. Sorption isotherms modelling

All equations in Table 2 are associated with sorption models that were fitted into the experimental data. These sorption models are commonly applied to clarify the sorption isotherms of food products. The most beneficial for establishing the optimum moisture conditions for good storage steadiness, particularly for dried foods are BET model that provided $M_0 [9]$. Nonlinear regression analysis were utilized to evaluate the constants of the models from the experimental results of sorption isotherms by using statistical analysis software IBM SPSS version 23. The potential of each model to fit the experimental data were calculated by the mean relative percent error ($P$), the root mean standard error (RMSE) and coefficient of correlation ($r^2$) between the experimental and the predicted figures were determined using the following equations:

$$P = \frac{100}{N} \sum_{j=1}^{N} \left| \frac{y_{j,\text{cal}} - y_{j,\text{exp}}}{y_{j,\text{exp}}} \right|$$

(1)

$$\text{RMSE} = \sqrt{\frac{1}{N} \sum_{j=1}^{N} \left(\frac{y_{j,\text{cal}} - y_{j,\text{exp}}}{N - n_p} \right)^2}$$

(2)

$$r^2 = \frac{S_t - SCE}{S_t}$$

(3)

where,

$y_{j,\text{cal}}$ = calculated values of moisture content

$y_{j,\text{exp}}$ = experimental values of moisture content

$$S_t = \sum_{i=0}^{n} (\bar{y} - y_{j,\text{exp}})^2$$

$$\bar{y} = \frac{\sum_{j=0}^{N} y_j}{N}$$

$$SCE = \sum_{j=1}^{N} (y_{j,\text{cal}} - y_{j,\text{exp}})^2$$

According to Lomauro [10], the model is considered to be suitable when the P-value is fewer than 10%. The model was also assumed to be good when the $r^2$ is high when RMSE and P (%) values were low.
Table 2. Established mathematical models used in sorption isotherms evaluation [11]

| No | Model name | Equation |
|----|------------|----------|
| 1  | GAB        | $X_e = \frac{CABa_w}{(1 - Ba_w)(1 - Ca_w) + (ABa_w)}$ (4) |
| 2  | Oswin      | $X_e = A\left(\frac{a_w}{(1-a_w)}\right)^B$ (5) |
| 3  | Peleg      | $X_e = Aa_w^B + CAa_w^D$ (6) |

Where,
- $A_w$ = water activity
- $X_e$ = moisture content (dry basis)
- $A$, $B$, $C$, $D$, = constants in isotherms model
- $M_0$ = monolayer moisture content (dry basis)

3. Results
The equilibrium moisture content (EMC) of $F. deltoidea$ leaves were analyzed versus relative humidity (RH) levels ranging from 23.28 to 86.77 % at 8, 30 and 40 °C as shown in Figure 2. The moisture adsorption curve is assigned to have type III which shown as J-shaped isotherm according to Brunauer’s finding [12]. Food supplies such sugars, apple, raisins, apricot, pineapple and beef shows similar behaviour has been stated earlier [9]. The type III isotherm is defined by a product which can keep small quantities of water at low relative humidity levels and large quantities of water at high relative humidity levels. Referring to Brunauer, Emmett and Teller (BET) theory, a type III isotherm shows when the binding force of the first layer is lesser than the binding force among water particles [13].

![Figure 2. Experimental moisture adsorption for F. deltoidea](image-url)
The result of fitting criteria value for every selected model showed that all the values are within the adequate range as referred in Table 3. The Peleg model had showed the best performance for both desorption and adsorption isotherms at both temperatures as compared to the other models. The model had highest values of $r^2$ (0.9981 to 0.9717) and the lowest values of P (0.0958 to 0.5409) and RMSE (0.0025 to 0.0067) for the isotherms. On the other hand, GAB model shows the second-best performance. The fitting criteria range were 0.9956 to 0.9630, 0.0080 to 0.3559, and 0.0042 to 0.0077 for $r^2$, value of P and RMSE respectively. The least fitting criteria values are from the Oswin model where the $r^2$ value range between 0.9639 to 0.9178, P value were from 0.0094 to 1.3074 and the RMSE values were 0.0079 to 0.0141.

Table 3. Model parameters for moisture adsorption of F. deltoidea dried leaves

| Model | Temp (°C) | $r^2$ | RMSE | (P%) | A       | B       | C       | D       | $M_0$ |
|-------|-----------|-------|------|------|---------|---------|---------|---------|-------|
| GAB   | 8         | 0.9956| 0.0042| -0.0080| -14.5790| 0.8961  | 0.0604  |         |       |
|       | 30        | 0.9841| 0.0053| -0.0245| -20.8994| 0.8495  | 0.0527  |         |       |
|       | 40        | 0.9870| 0.0046| -0.0365| -12.1571| 0.8775  | 0.0473  |         |       |
| Oswin | 8         | 0.9495| 0.0141| 1.3074 | 0.1290  | 0.9390  |         |         |       |
|       | 30        | 0.9639| 0.0079| 0.0094 | 0.1060  | 0.3260  |         |         |       |
|       | 40        | 0.9300| 0.0107| 0.0723 | 0.1050  | 0.3200  |         |         |       |
| Peleg | 8         | 0.9963| 0.0038| 0.0958 | 0.3848  | 7.0436  | 0.1464  | 0.2682  |       |
|       | 30        | 0.9952| 0.0029| 0.2873 | 0.2190  | 2.5700  | 0.0440  | -0.3890 |       |
|       | 40        | 0.9962| 0.0025| 0.5409 | 0.2300  | 2.8420  | 0.0420  | -0.4940 |       |

4. Conclusions
From the results, it is shown that temperature and relative humidity affect the EMC. It is recommended that the constant parameter predicted by the Peleg model can be used to define the moisture adsorption isotherm of F. deltoidea leaves for storage and other further use.

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