The influence of drying temperature on the quality, morphology and drying characteristics of *Cosmos caudatus*

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**Abstract.** This study scrutinizes the influence of oven drying temperature (40, 60, and 80 °C) on the product quality (phenolic content and antioxidant activity), morphological appearance, and drying characteristics of *Cosmos caudatus* (*C. caudatus*). A spectroscopic UV assay and a Field emission scanning electron microscope (FESEM) were used to analyse the product quality and the morphological appearance of the *C. caudatus* leaves. The drying characteristics were assessed based on the pattern from the drying curve (moisture ratio versus drying time) at the tested temperatures. It was observed that a drying temperature of 40 °C preserved the quality of the dried *C. caudatus* leaves. This may be seen where a significant reduction of the phenolic content and antioxidant activity occurred at 60 to 80 °C, probably due to the decomposition of the valuable phytochemical compounds. This effect correlated with the physical damage that took place to the vegetal tissues. The damage was recorded as 40 < 60 < 80 °C according to the severity (from minor to severe damage). The drying process of *C. caudatus* occurred mainly in the falling rate period, in the absence of an initial rate period. Higher drying temperatures resulted in a shorter drying time as proven by the value of the effective moisture diffusivity that increased from $4.12 \times 10^{-12}$ m²/s (40 °C) to $24.71 \times 10^{-12}$ m²/s (80 °C). The *C. caudatus* sample dried with an oven dryer at 40 °C possessed the best condition of drying without compromising the quality of the dried *C. caudatus* leaves.

1. **Introduction**

*Cosmos caudatus* (*C. caudatus*) is a traditional medicinal plant belonging to the family Asteraceae, which is scientifically proven as a source of antioxidants and is a free radical scavenger [1–3]. Known as “Ulam Raja” by the native Malays, the plant is generally consumed raw as a condiment or added to the main dish or drinks. Many pharmacological properties have been reported for the plant extract, such as antidiabetic, antihypertensive, anti-inflammatory, bone-protective, and antimicrobial [4,5]. Due to its versatile applications, many health supplementary products are produced from the plant extract.

Drying is amongst the crucial steps in herbal processing, mostly for drying raw materials and plant extracts. This dehydration process is used to remove moisture from a product to prevent possible contamination (i.e. microbial or chemical) and to lengthen the shelf life of a product in the post-harvesting stage [6]. Water can be removed easily using heat, but the use of a high temperature may decrease the quality of the dried raw materials and plant extracts [7,8]. Most of the current drying by
using convective methods are developed with the incorporation of heat, such as ovens, cabinet trays, heat pumps, and vacuum tray dryers [9]. Oven dryers are used for drying plant samples due to the uniformity in the dried samples [7]. A previous study of oven drying reported that a high oven temperature increased the drying rate and shortened the drying time, but the dried product underwent thermal decomposition that reduced its quality [8]. Nevertheless, an extreme drying process may lead to morphological changes in the product, and this may cause an imbalance in its phytochemical and antioxidant properties [10]. Therefore, a strategy is needed to devise a suitable drying method with high efficiency to preserve the quality of the dried product.

The drying process of the plant materials can be further described by kinetic analyses, which is used to generalise the heat and moisture transfer mechanism [9]. By plotting the drying curve and drying rate curve, the drying cycle period can be determined. The drying cycle includes initial (settling down), constant and falling rate periods [9]. Studies have shown that using drying characteristic curves, the prediction of the optimum drying time can be carried out for the specific drying conditions [8-9]. This is important for a better understanding of the mechanism of drying involved in the particular process.

Over time, the effect of drying methods on the phenolic compounds and antioxidants on C. caudatus leaves has been reported by other researchers [11]. The optimal temperature in a drying oven has been found to be 43 °C, and this medium temperature could preserve the phenolic compounds and antioxidant properties in the dried C. caudatus leaves [11]. However, the drying kinetic analyses of C. caudatus leaves have not been discussed in any of the literature to date. Further, the current available drying data was not comprehensive enough to describe the overall performance of the drying process, and also the drying behaviour.

In this current study, the drying characteristic of C. caudatus leaves was studied in a drying oven. The sample was dried at 40, 60, and 80 °C. Then, the quality changes in terms of the phenolic content, antioxidant activity, and morphological appearances of the dried C. caudatus leaves were investigated and to determine the best drying temperature for the sample. The study is essential and can be used further to develop a mathematical kinetic drying model and to support the local herbal sector as the current data is very limited.

2. Materials and Methods

2.1. Material preparation
C. caudatus leaves were collected from a research farm located at Universiti Teknologi Malaysia, Pagoh, Malaysia. Subsequently, the harvested samples were brought to a sample preparation laboratory for cleaning purposes. The leaf parts were taken and kept for further analysis. The initial moisture content of fresh C. caudatus leaves was determined at 80.5% weight basic by a moisture analyser (MX-50, A&D Instruments Ltd, Oxfordshire, United Kingdom).

2.2. Determination of quality properties

2.2.1. Material preparation. The C. caudatus leaves were dried in a drying oven (Memmert UF110, Memmert Universal, Schwabach, Germany) at 40, 60, and 80 °C. The dried leaves were ground and sieved to a particle size of 850 microns using a sieving machine (Wst yler, Mentor, OH, USA). Some 1 g of dried sample was extracted with 80% ethanol by a sonication device for 20 min (WiseD, Daihan Scientific, Ltd Co, Korea). The extract was filtered using a vacuum filtration system to remove the ground leaves. The filtrate was dried using a solvent concentrator and the dried extract was kept in a sealed tube at -25 °C until used for analysis.

2.2.2. Total phenolic content(TPC). The TPC of the extracts was determined by the modified method of Safdar et al. [12]. Briefly, C. caudatus extract solution in methanol (40 µL) was placed in a 96-well plate and then mixed with 100 µL Follin-Ciocalteu reagent (10x dilution). After 5 min of reaction,
some 80 µL of 7.5% sodium carbonate was added into the solution and then was incubated at room temperature for the development of a blue colour. After 90 mins, the sample was measured at 765 nm using an ELISA microplate reader (VersaMax, Molecular Devices). Gallic acid was used as a standard in evaluating the TPC. The TPC was expressed as a gallic acid equivalent (g GAE/100g extract; mean values ± standard deviation, n =3).

2.2.3. Antioxidant by DPPH assay. The antioxidants were evaluated by the modified method of Lee et al. [13]. The free radical agent DPPH (1,1-diphenyl-2-picryl-hydrazyl) was prepared in methanol (0.1 mM). The C. caudatus extract was prepared at different concentrations (2000-7.8 mg/L) and was mixed with 10 µL DPPH reagent. Then, the working standard of ascorbic acid (1000-7.8 mg/L) was prepared and was mixed with the DPPH reagent. A parallel control with an absence of extract was also prepared and analysed similarly. These solutions were then kept under minimal light for 30 mins before measured using ELISA microplate reader (VersaMax, Molecular Devices) at 515 nm. All experiments were conducted were conducted in triplicate and the average reading was recoded. The percentage of DPPH inhibition was calculated using equation (1).

\[
\text{% Inhibition of DPPH} = \left( \frac{A_c - A_s}{A_c} \right) \times 100
\]

Where \(A_c\) is the absorbance of the control, \(A_s\) is the absorbance of the sample.

2.3. Morphology assessment by Field Emission scanning electron microscopy (FESEM)
The FESEM image of C. caudatus leaves of fresh and treated samples under different drying temperatures were captured using Field Emission scanning electron microscopy (FEI Versa 3D dual beam, Hillsboro, Ore., US). Before analysis, the samples were coated with gold in a Q150R S sputter coater (Quorum Technologies Ltd, East Sussex, U.K). The FESEM images were captured under a 5 kV vacuum with 1000 times magnification.

2.4. Drying experiments
The drying process were carried out in a drying oven (Memmert UF110, Memmert Universal, Schwabach, Germany) following the steps elucidated by Alara et al. (2019) with slight modification. The temperature of the ambient air was between 26 to 28 °C and the relative humidity was 80%. The samples were dried at different temperatures level, 40, 60, and 80 °C. The samples were placed in trays with a single layer density (5g) and positioned in the middle of the drying chamber to ensure uniform drying. The weight loss of samples was measured at selected intervals at three measurements using an analytical balance (PA214C, OHAUS Corporation, USA). For the 40 °C experiment, an interval of 10 mins was set up over 3 hours and another 30 mins added for the following 2 hours until a constant sample weight was achieved. For the 60 and 80 °C experiments, an interval of 5 and 2 mins was set up at the initial drying stage, and another 10 mins added for the following 1 hour until the drying process ended. The drying process finished when there was not change observed in the reading of the mass. The moisture content (MC) was determined based on equation (2) [8].

\[
MC = \frac{m_w}{m_{dm}}
\]

where \(M\) represents the moisture content (g water/g dry matter), \(m_w\) is the mass of water in the sample (g) and \(m_{dm}\) denotes the mass of dry matter in the sample (g).

The characteristics of the drying process were elucidated using the drying curve and drying rate curve, using the following equations (3-4) [8]:

...
where \( M \) is the moisture ratio at any drying time (\( dt \)), \( M_o \) is the initial moisture and \( M_{eq} \) is the moisture at equilibrium (g water/ g dry matter).

The drying rate (kg water/kg dry matter min) of the sample was calculated using equation (4):

\[
DR = \frac{M_{t+\Delta t} - M_t}{\Delta t}
\]

where, \( DR \) is the drying rate, \( Mt \) (kg water/kg dry matter) is the moisture content at time \( t \), \( M_{t+\Delta t} \) represents the moisture at \( t+\Delta t \) (kg water/kg dry matter), and \( t \) is the drying time (min) respectively.

2.4.1. Calculation of the effective diffusivity coefficients. The effective diffusion coefficient (\( D_{eff} \)) was based on Fick’s second law equation as presented in equation (5). It was then simplified by plotting the ln MR against drying time and the value of \( D_{eff} \) was determined from the slope of the linear regression [8].

\[
MR = \frac{8}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{(2n+1)^2} \exp\left(-\frac{(2n+1)^2 \pi^2 D_{eff} t}{4L^2}\right)
\]

For a longer drying process, \( MR < 0.6 \) the equation is simplified to:

\[
\ln(MR) = \ln\left(\frac{8}{\pi^2}\right) - \pi^2 \frac{D_{eff} t}{4L^2}
\]

where MR, \( D_{eff} \), L, t and n represent the dimensionless moisture ratio, the effective moisture diffusion coefficient (m²/s), half the thickness of the initial \( C. caudatus \) sample (m), the drying time (s) and an integer value, respectively.

3.0. Results and discussion

3.1. Effect of drying temperature on product quality

Table 1 shows the results of the effect of drying temperature on the quality of the dried \( C. caudatus \) leaves. The total phenolic content (TPC) and antioxidant property were selected as the quality indicator for this study. Based on the TPC result, the drying temperature at 40 °C contained higher TPC compared to the other samples evaluated at different temperatures. The TPC dropped as higher temperatures were applied. The exposure to heat above 40 °C led to the loss of phenolic compounds. Another study also found that the oven temperature had the most significant effect on the TPC of \( C. caudatus \) leaves [11]. Pin et al. (2009) observed that a drying temperature above 80 °C caused a thermal decomposition of the phenolic compounds of dried betel leaves under such high temperatures. Further, drying at high temperatures (70 and 100 °C) had resulted in a significant loss of TPC in the \( Vitex \) species [14]. A study conducted by Tajudin et al. (2019) also found that the heat pump temperature affected significantly the TPC of Roselle. Such findings indicate that the TPC is vulnerable to the drying temperature.
Table 1. Change of total phenolic content and antioxidant activity of *C. caudatus* to different drying temperatures.

| Temperature (°C) | Total phenolic content, g/100g GAE | Antioxidant activity, % DPPH |
|------------------|-----------------------------------|-----------------------------|
| 40°C             | 31.437 ± 2.212                    | 87.327 ± 0.464              |
| 60°C             | 19.441 ± 1.226                    | 77.384 ± 1.712              |
| 80°C             | 18.211 ± 1.340                    | 76.264 ± 2.461              |

Meanwhile, there was a reduction in the DPPH inhibition from 40, 60, and 80 °C, respectively, as shown in Table 1. The antioxidant property of *C. caudatus* was measured at 250 mg/L (when the concentration reached a plateau). The highest antioxidant properties were measured at 87.327 ± 0.464 % at 40 °C, while the lowest was 76.264 ± 2.461 % at 80 °C. Most of the antioxidant compounds were heat-labile, thus being exposed to high drying temperatures may destroy the property [10,14]. Based on this study, the drying method at temperatures above 40 °C had a deleterious effect on the quality of the sample.

3.2. Drying characteristics of *C. caudatus*

Figure 1 outlines the drying curves for *C. caudatus* at different temperatures. Overall, the *C. caudatus* fresh leaves spent about 240, 70, and 10 mins when drying at temperatures of 40, 60, and 80 °C, respectively. The longest time was recorded at 40 °C, while the shortest drying time was recorded at 80 °C. Drying at a lower temperature took a longer time to dry the sample, probably due to the insufficient heat for vaporisation of moisture from the surrounding air. Meanwhile, a quick loss of moisture occurred when drying the *C. caudatus* leaves at 80 °C, where it took only 10 mins to reduce the moisture. This quick loss of moisture content occurred because the heat supply was sufficient for vaporisation of the moisture and therefore increased the drying rate [8,15].

The high drying rate resulted in the rapid loss of moisture content which accelerated greater heat transfer, which caused rapid moisture removal from the wet material [16]. A slower drying rate occurred at 40 °C that required a longer drying time compared to 60 and 80 °C. As seen in figure 2, the drying process of *C. caudatus* mostly took place in the first falling rate period, and this period was believed to be controlled by internal diffusion [15]. The constant rate period was neglected, assuming
that the entire process of *C. caudatus* oven drying occurred mainly in the falling rate period. The result was also similar to the drying characteristics reported by other researchers [15,16]. After reaching a certain drying time, the moisture content reached equilibrium moisture content. The drying process stopped when no further moisture was diffused from the material.

![Drying curves of *C. caudatus* at different drying temperatures](image)

Figure 2. The drying curves of *C. caudatus* at different drying temperatures

3.3. Effective moisture diffusivity ($D_{eff}$)
The drying mechanism may be described using Fick’s second law with the assumption that the diffusion of moisture transfer is the main major case of drying, there are negligible diffusional coefficients, shrinkage, and a constant temperature [8]. Table 2 shows the approximate values of $D_{eff}$ at different drying temperatures. The $D_{eff}$ values ranged from $4.12 \times 10^{-12}$ to $24.716 \times 10^{-12}$ m$^2$/s. The average results of $D_{eff}$ showed an increase in the value as the drying temperature increased. This result may be attributed to higher drying rates that cause rapid absorption of heat which increases moisture diffusion in the product. Similarly, a drying temperature between 50 to 80 °C, increased the $D_{eff}$ in dika nuts and kernels [17]. The $D_{eff}$ calculated in this current study falls within the acceptable range of 10-9 to 10-12 m/s for food drying [18]. Using a different drying process either by a thermal convection oven or a hot air dryer, also indicated an increase in the $D_{eff}$ coefficient when the temperature was increased [8,15].

| Temperature (°C) | $D_{eff}$ (m$^2$/s) x 10$^{-12}$ |
|-----------------|-------------------------------|
| 40°C            | 4.1194                        |
| 60°C            | 9.6111                        |
| 80°C            | 24.716                        |

Table 2. The value of $D_{eff}$ at drying temperatures

3.4. Effect on drying temperature on morphological appearance

Figure 3 shows the field emission scanning electron microscopy images obtained of the surface of the (a) fresh *C. caudatus* leaf and treated under different drying temperatures of (b) 40 °C, and (c) 80 °C. The effect of temperature on the proportion of the ruptured or deflated trichomes on the epidermal
surface of the *C. caudatus* leaves was observed. The images show that the glandular and non-glandular trichomes on the epidermal surface started to be damaged at 40 °C, showing a minimal reduction when compared to the image of the fresh leaf. A significant reduction of deflated trichomes was observed at 80 °C. Nevertheless, the more intense heating caused a greater shrinkage effect and increased the proportion of the ruptured trichomes. The application of high temperature may lead to violent evaporation and enhanced removal of moisture content from the surface of plant materials. According to some studies, high-temperature treatment may result in a change in the physical structure of the plant cell wall which might lead to damage of plant tissue and thus affect the quality of the phytochemicals [8]. Therefore, the appropriate drying temperature is vital to create a minimal impact on the physical structure as well to preserve the plant phytochemical compounds.

![Figure 3](image_url)

**Figure 3.** The FESEM image of *C. caudatus* of fresh leaf and dried under 40 and 80 °C

4. **Conclusion**

In this study, different oven drying temperatures have been shown to have an effect on the drying quality, morphological appearance and drying behaviour of *C. caudatus* leaves. A comparison between the drying temperatures showed a difference in the quality of the dried *C. caudatus* leaves. Higher drying temperatures (60 °C and above) caused alteration of the phenolic compounds and reduced the antioxidant value. The drying characteristics of *C. caudatus* leaves occurred in the falling rate period. The drying process was found to be shorter at higher temperatures. However, it affected the product quality and caused an imbalance in the leaf microstructure. The value of $D_{eff}$ increased when a higher drying temperature was used to dry the sample. More so, the value was calculated to evaluate the temperature dependence of the moisture diffusion. This study suggests the use of a drying temperature of 40 °C, as it preserves the product quality even though the process requires an extended drying period.

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