Effect of Microwave Vacuum Drying on the Drying Characteristics and Chemical Compositions of Turkish Gall Residues

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Abstract. Recently, the secondary use of waste has received increasing attention. The raw material of Xipayi mouth rinse is Turkish gall. During the industrial production of Xipayi mouth rinse, a lot of gall residues are produced. If these drugs are directly discarded, it will cause waste of resources and also bring pressure to the ecological environment. Previous studies have found that the active ingredient content of Turkish gall residues remains high, therefore the residues are still a valuable resource for exploitation. This study aimed to evaluate the effect of the chemical compositions and drying characteristics of Turkish gall residues during microwave vacuum drying (MVD). Different vacuum degrees, sample loads and sample sizes were used to investigate the drying characteristics of Turkish gall residues, and a kinetics model was established to describe the drying characteristics. We determined gallic acid (GA), methyl gallate (MG) and ellagic acid (EA) contents, total phenolic content (TPC) and total sugar content (TSC) of dried Turkish gall residue powder and investigated the influence of different MVD conditions on components. The results showed the good fit of the Page model equation in describing the drying kinetics of Turkish gall residues during MVD. Different conditions of MVD have different effects on the main components of residue. Thus, MVD can be used to desiccate Turkish gall residues. The Page kinetics model predicts the changes in dehydration rate during MVD of Turkish gall residues, providing a proven drying method for gall residues, and provides a theoretical basis for the industrial mass production of pharmaceutical residues.

1. Introduction

Xipayi mouth rinse is a gargle formulation, made from Turkish gall (gall of Quercus infectoria). Quercus infectoria Olivier (Fagaceae) is an oak shrub species that produced chemical constituents such as tannins, gallic acid (GA), flavonoids, resins, and polysaccharides of the rinse²³. At present, Xipayi mouth rinse production is increasing, resulting in increased amount of gall residue. Studies have shown that Turkish gall residue still certain useful active ingredients⁴. A feasible method for ecologically utilizing the residue must be determined. New residue development in plant and fungus cultivation and animal breeding completely utilizes the potential value, such as environmental friendliness and broad application prospects⁵.

Turkish gall residues contain substantial amount of water. Thus, the residues must first be dried before analysis. Drying serves as basis for studying substance composition⁶. In general, the drying...
methods of traditional Chinese medicine include sun and hot–air drying. These methods evaporate water by heat transfer. However, these methods exhibit disadvantages, such as easy destruction of substance components, long drying time, and susceptibility to environmental effects.

Microwave vacuum drying (MVD) technology is an ingenious combination of microwave and vacuum drying; it combines the advantages of vacuum science, thermodynamics, mechanics, and programmable control. Microwave is added as heat source in vacuum drying; it is generated by a microwave generator during MVD chamber operation and fed into a microwave heater by an energy–feeding device. The material is sent to a heater by a transmission system. During this step, moisture in the material is heated and evaporated by microwave energy, which can rapidly heat the material starting from the inside, whereas water vapor is removed by a dehumidification system for drying. This method can effectively shorten the drying time and considerably improve the drying efficiency. MVD technology hastens heat transfer and enhances drying efficiency in traditional drying and can effectively and aseptically maintain the original effective ingredients and appearance quality of materials. Bacteria in the materials are killed by the biological and thermal effects produced by microwave electromagnetic field. At present, MVD technology is mostly used in food, fruit, and vegetable drying. MVD technology has been applied to dry Chinese medicinal materials and extracts.

In this study, MVD technology was applied in Turkish gall residue drying. The effects of vacuum degree, sample load, and sample size on the drying characteristics and chemical composition of the residues were investigated. The mathematical model of Turkish gall residue MVD was established by regression analysis, providing theoretical basis for application of Turkish gall residue MVD and an efficient drying method for Turkish gall residues.

2. Experimental

2.1. Sample preparation
Turkish gall (Quercus infectoria Olivier) residues were obtained from Xinjiang Newcicon Pharmaceutical Co. Ltd. (Urumqi, Xinjiang province, China). Before drying experiment, Turkish gall residues extract were divided into three groups (every group has three parts). The first group passed Turkish gall residues through the No.2 sieve (0.7 mm) and measured the weight of 40 g every part. The second group also passed the dregs through the No. 2 sieve (0.7 mm) and weighed 30, 40 and 50 g respectively. The third group weighed Turkish gall residues extract 40 g and passed through the No. 1, No. 2 and No. 3 sieve (2.0, 0.7, and 0.3 mm).

2.2. Microwave vacuum drying
Microwave vacuum drying (MVD) was carried out by a microwave vacuum dryer (Type JSWZ−6) purchased from Nanjing Huangming Ozone Mechanical and Electrical Equipment Factory (Nanjing, Jiangsu, China) was used in this study. The outline dimension are 171×117×196 cm and the microwave cavity dimension are 76×76×78 cm with microwave output power of 6 kw. The first group of samples dried in a condition of vacuum degree was 0.04, 0.06, and 0.08 MPa. The second and third group dried under the vacuum degree of 0.08 MPa. Dry samples weighed every 30 seconds until the moisture content reach equilibrium. Next, smash the MVD Turkish gall residues to 80−mesh and stored in the glass desiccator for further analysis.

2.3. Drying kinetics and mathematical model
Moisture content at time t, $X_t$ (100%, d.b.) was measured using an oven method (GB/T8858−88, National Standard of China) and calculated according to Eq. (1):

$$X_t = \frac{m_t - m_d}{m_d} \times 100\%$$  \hspace{1cm} (1)$$

Where refers to the moisture content at time t on the basis of dryness (% d.b.), t (s) denotes the
drying time, (g) represents the weight of the material after drying t, and (g) stands for the dry matter weight of the material.

Drying rate (DR) under various drying conditions was evaluated as the moisture content variation with time and calculated using Eq.(2):

\[ DR = \frac{m_t - m_{t+At}}{At} \]  

(2)

Where (g) indicates the water loss quantity between two adjacent measurements, and (min) is the interval time between two adjacent measurements.

The moisture ratio (MR) of Turkish gall residues was calculated in accordance with Eq.(3):

\[ MR = \frac{m_t - m_e}{m_0 - m_e} \]  

(3)

Where and represents the initial moisture content (g g\(^{-1}\), d.b.) and equilibrium moisture content (g g\(^{-1}\), d.b.), respectively. The values of can be approximately considered as 0, and the formula was simplified in accordance with Eq. (4)\cite{17}:

\[ MR = \frac{m_t}{m_0} \]  

(4)

The establishment of a model is very important to investigate the drying characteristics and effectively predict the parameters of the drying process\cite{18,19,20}. Three drying models have been used in this study (Table 1).

| No. | Model name | model | Model logarithms |
|-----|------------|-------|------------------|
| 1   | The exponential model\cite{21} | \( MR = e^{-Kt} \) | \( \ln MR = -Kt \) |
| 2   | The monomial diffusion model\cite{22} | \( MR = A e^{-Kt} \) | \( \ln MR = \ln A - Kt \) |
| 3   | Page\cite{23} | \( MR = \exp (-Kt^N) \) | \( \ln (-\ln MR) = \ln K + N \ln t \) |

According to the tested data and Table 1, the graphs of t-ln (MR) and In t-ln (-lnMR) for the different vacuum degrees, loads and sample sizes of Turkish gall residues during MVD were drawn.

2.4. Determination of chemical compositions of Turkish gall residues

2.4.1. Gallic acid (GA), methyl gallate (MG) and ellagic acid (EA) contents

High-performance liquid chromatography(HPLC) with a diode-array detector was used to determine the contents of GA, MG, and EA of dried Turkish gall residue powder, using the switching wavelength method\cite{24}. Approximately 3 μL sample was injected into the HPLC system. Agilent Zorbax SB-C18 column (250 mm×4.6 mm, 5 μm) was used. The mobile phase was Methanol (A) - 0.05% phosphoric acid aqueous solution (B). Flow rate was set at 1 mL min\(^{-1}\) with the following gradient elution procedure: 0-10 min, 5%-10% A; 10-26 min, 10%-30% A; 26-36 min, 30%-50% A; 36-45 min, 50%-85% A. The detection wavelength was 272 nm for GA and MG, 255 nm for EA.

2.4.2. Total phenolic content (TPC)

Approximately 1 g dried powder of Turkish gall residues was ultrasonic extracted with 50 mL water for 20 min and then filtered through 0.45 μm microporous membrane. The TPC of dried Turkish gall
residues under different drying conditions was determined using the Folin–Ciocalteu method with GA as standard\(^\text{[25]}\). In brief, 0.1 mL extract was diluted to 2 mL. Then, 1 mL Folin-Ciocalteu reagent and 10 mL distilled water were then added. The mixture was shaken, and 29% Na2CO3 solution was added. The solution was incubated in the dark at room temperature for 30 min. The absorbance of mixture solution was measured using an ultraviolet-visible (UV-Vis) spectrophotometer at 760 nm because a maximum absorption wavelength at 760 nm was observed. TPC was expressed as milligrams per gram.

2.4.3. Total sugar content (TSC)
Approximately 0.5 g dried powder added into water with a solid-to-liquid ratio of 1:30 was placed in a water bath twice for 2 h, and the supernatants were combined to obtain the test solution. The TSC of the dried Turkish gall residues was determined under different drying conditions by anthrone colorimetry\(^\text{[26]}\). In brief, 0.3 mL extract was diluted to 2 mL, and 5 mL of 0.2% sulfuric acid-anthracene copper solution was added and heated in boiling water bath for 15 min. The extracts were then removed and cooled in ice water bath. Absorbance was determined at 625 nm by UV-Vis spectrophotometry. TSC was expressed as milligrams per gram.

3. Results and discussion

3.1. Drying curves and drying characteristic curve under different conditions
Drying curves and drying characteristic curve under different conditions as shown in Figures 1-3. Figure 1 illustrates that drying time decreased with increased vacuum degree at 40 g sample load and 0.7 mm sample size. As shown in Figure 1a and 1b, vacuum degree evidently affected the MVD of Turkish gall residue. DR increased with increased vacuum degree. This finding is due to the decreasing boiling point of water molecule with increased vacuum degree. Under similar microwave power, that is, at constant drying temperature, an increased vacuum degree resulted in a reduced water boiling point, hastened water evaporation, shorter time to reach the drying end point, and faster DR.

![Figure 1](image)

**Figure 1.** (a) Drying curve of Turkish gall residues by different vacuum degrees and (b)drying characteristic curve at different vacuum degrees.

Drying time increased with increased load at 0.08 MPa vacuum degree and 0.7 mm sample size (Figure 2). Figure 2a shows that prolonged drying end point was due to increased sample loading. This phenomenon occurred as moisture content in the material increased with increasing sample load and reduced the microwave energy absorbed by each water molecule in unit time. As shown in Figure 2b, during the acceleration stage of drying, the material reached the phase. At the same moisture content, 50 g load showed the highest DR, and 50, 40, and 30 g then showed decreased DR. At the acceleration stage, the three curves showed a slight difference. The results showed that sample loading remarkably affected the later stage of drying.
Figure 2. (a) Drying curve of Turkish gall residues by different loadages and (b) drying characteristic curve at different loads.

From Figure 3 can conclude that at approximately 40 g load and 0.08 MPa vacuum degree, the drying time increased with increased sample size. However, the differences were negligible (Figure 3a). The MVD process for Turkish gall residues can be generally divided into three stages (Figure 3b), namely, accelerated, constant-velocity, and decelerated drying phases. In the constant drying stage, the sample with 0.07 mm sample size yielded the lowest DR, and this finding may be due to the small particle size observed with high moisture content of the sample. Thus, moisture content was the highest when the drying end point was reached.

Figure 3. (a) Drying curve of Turkish gall residues by different sizes and (b) drying characteristic curve at different sample sizes.

3.2. Mathematical modeling
Data were analyzed using Origin Pro 8.5 program, and the kinetics model fitting and regression analysis were conducted using 1stOp15Pro program.

Figures 4–6 show the curves of $-\ln (MR)-t$ and $\ln(-\ln MR)-\ln t$ under different vacuum degrees, loads and sample sizes, respectively.
Figures 4a-6a show that $-\ln (MR)$ were non-linear. Thus, the MVD of Turkish gall residues failed to conform to the monomial diffusion and exponential models. Figures 4b-6b show that $\ln \left(\frac{-\ln MR}{t}\right)$ and $\ln t$ exhibited a certain linear relationship. Thus, the MVD of Turkish gall residues conformed to the Page model. Different linear relationships were observed between $\ln \left(\frac{-\ln MR}{t}\right)$ and $\ln t$ under different drying conditions. $\ln K$ and $\ln N$ changed with changed vacuum degree $P$ (MPa), sample load $L$ (g), and sample size $S$ (mm). $a$, $b$, $c$, $d$, $e$, $f$, $g$, and $h$ are undetermined coefficients. In Page’s equation, $K$ and $N$ are respectively defined by the following equations:

$$K = a + bP + cL + dS$$ (5)
Multiple linear regressions of the experimental data were analyzed using 1StOp15PRO, and the results are shown in Table 2. Multiple linear regression analysis was utilized to determine the model equation. The multiple correlation coefficient (R) is generally used to indicate the closeness of relationship between variables. The fit of the mathematical model to experimental data was evaluated using the coefficient of determination (R²). Root-mean-square error=0.187, R²=0.936, X²=1.927, and F-Statistic at 586.243 showed that the model fits well the experimental date, and the experimental error was negligible. In the production and experiment, the law of water ratio changing with time under varying vacuum degree, load and sample size can be calculated according to the equation. Thus the drying end time can be better grasped and Turkish gall residues cannot be overheated during the MVD process.

Table 2 MVD kinetics model of Turkish gall residues

| Model       | Undetermined coefficient | RMSE  | R²    | X²    | F-Statistic |
|-------------|--------------------------|-------|-------|-------|-------------|
| Page model  |                          |       |       |       |             |
| MR = \exp(-Kt^N) |                         |       |       |       |             |
| a=-0.000305 |                          | 0.187 | 0.936 | 1.927 | 586.243     |
| b=0.005446  |                          |       |       |       |             |
| c=-0.000022 |                          |       |       |       |             |
| d=0.000336  |                          |       |       |       |             |
| e=2.836282  |                          |       |       |       |             |
| f=8.560366  |                          |       |       |       |             |
| g=-0.002788 |                          |       |       |       |             |
| h=-0.213616 |                          |       |       |       |             |

The regression equation is shown in Eq.(7):

\[
N = e + fP + gL + hS
\]  

\[
\ln(-\ln MR) = \ln(-0.000305 + 0.005446P - 0.000022L + 0.000336S) + (2.836282 - 8.560366P - 0.002788L - 0.213616S)ln t
\]  

(7)

Page model: \( MR = \exp(-Kt^N) \), where

\[
K = -0.000305 + 0.005446P - 0.000022L + 0.000336S ;
\]

\[
N = 2.836282 - 8.560366P - 0.002788L - 0.213616S
\]

3.3. Kinetics model verification test
A group of samples with 0.08 MPa vacuum degree, 40 g load and 0.7 mm particle size was selected to verify the fitting accuracy of the mathematical model for the experimental data. The actual experimental data of the group were compared with the predicted values obtained by mathematical model under the given conditions. The results showed that the predicted curve of Page equation was consistent with the actual curve shown in Figure 7. This result indicates that the Page model can accurately reflect the MVD of Turkish gall residues and can predict the MVD process of Turkish gall residues.
3.4. Determination of the chemical compositions of Turkish gall residues

3.4.1. GA, MG and EA content

Figure 8a, b and c shows the effects of MVD of different drying conditions on the GA, MG, and EA contents of Turkish gall residues. According to Figure 8a, b and c, vacuum degree and load significantly affected the GA, MG, and EA contents of Turkish gall residues (P<0.05). However, different sample sizes caused no significant effect on Turkish gall residue contents (P>0.05). The three component levels decreased with increasing vacuum degree. The result may be due to the decreased water vaporization temperature and rapid evaporation rate of water with increased vacuum, thus destroying the three components of Turkish gall residues and decreasing their contents. With increased load, the contents of the three components increased. This finding may be due to the increased sample loading, decreased microwave energy received per unit mass of Turkish gall residues in unit time, weakened microwave damage to the components in the sample, and increased component contents with increased sample loading.
3.4.2. Total phenolic content

Figure 9 shows the effects of MVD on the TPC of Turkish gall residues at different drying conditions. The figure shows the significant difference between different conditions (P<0.05). However, compared with vacuum degree and sample size, sample load exhibited a less significant effect on TPC (P>0.05). The TPC of Turkish gall residues increased with increased vacuum degree. This finding may be due to the decreased boiling point temperature of water and drying environment temperature, which reduced TPC of the heat-sensitive component. The effect of sample size on TPC initially decreased and then increased. At 0.3 mm sample particle size, the TPC of Turkish gall residues reached the highest at 137.82 mg g⁻¹, which may be due to the different absorption of water by different particle sizes. Thus, water loss rate in the drying process also differed. Approximately 0.3 mm sample size promoted water evaporation and reduced the loss of TPC, resulting in its highest content.

3.4.3. Total sugar content

Figure 10 shows the effects of MVD on the TSC of Turkish gall residues at different drying conditions. These effects showed significant differences between different conditions (P<0.05). However, compared with vacuum degree and sample size, the sample load showed less remarkable
effect on TPC (P>0.05). The TSC of Turkish gall residues increased with increased vacuum degree. This finding may be attributed to the decreased boiling point temperature of water and drying environment temperature, which reduced the loss of TSC of the heat-sensitive component. The effect of sample size on TSC initially decreased and then increased. At 0.3 mm sample particle size, the TSC of the Turkish gall residues reached the highest at 46.64 mg g⁻¹. This finding may be attributed to the different absorption of water by varying particle sizes. Thus, water loss rate in the drying process also differed. A 0.3 mm sample size promoted the water of sample evaporation and reduced the loss of TSC, resulting in its highest content.

Figure 10. Effects of MVD on the TSC of Turkish gall residues at different drying conditions

4. Conclusions
This study evaluated the effect of chemical compositions and drying characteristics of Turkish gall residues during MVD under different vacuum degrees, sample loads and sample sizes. The chemical compositions of dried Turkish gall residues powder, including GA, MG, EA, TPC and TSC were determined and the influences of different conditions of MVD on the contents were investigated. Based on the experimental results, can be concluded that different conditions of MVD remarkably affected the drying characteristics and chemical compositions of Turkish gall residues. Page equation model can fit for the drying process of Turkish gall residues according to the drying curve and drying characteristic curve. The GA, MG and EA contents decreased with increased of vacuum degree and increased with increased of load. TPC and TSC of Turkish gall residues increased with increased of vacuum degree, whereas the effect of sample size on the TPC and TSC initially decreased and then increased. Thus, MVD can be used for the drying of Turkish gall residues.

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Appendices

appendix A—Nomenclature
MVD — Microwave vacuum drying
GA — Gallic acid
MG — Methyl gallate
EA — Ellagic acid
TPC — Total phenolic content
TSC — Total sugar content
DR — Drying rate
MR — Moisture ratio
HPLC — High performance liquid chromatography
UV-vis — Ultraviolet-visible spectrophotometer
R² — Coefficient of correlation
X² — Chi-square
RMSE — Root mean square error
F — F-Statistic

appendix B—List of symbols
\( X_t \) — Moisture content at time \( t \) on the basis of dryness, %
\( t \) — Drying time, s
\( m_t \) — Weight of the material after drying \( t \), g
\( m_d \) — Dry matter weight of the material, g
\( m_r-m_t + \Delta t \) — Water loss quantity between two adjacent measurements, g
\( \Delta t \) — Interval time between two adjacent measurements, s
\( m_0 \) — Initial moisture content, g g⁻¹
\( m_e \) — Equilibrium moisture content, g g⁻¹
K, N — Page equation model parameter
P — Vacuum degree, MPa
L — Sample load, g
S — Sample size, mm
a, b, c, d, e, f, g, h — Undetermined coefficients