An improvement of rose flowers drying process recovering volatile compounds by heat pump systems

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Abstract. Low-temperature drying is becoming recognized as an optimal type of hot drying technology for agricultural and sideline products. This study presents an experimental study on drying roses with a heat pump dehumidifier. The rose drying process took 40 hours across three drying stages, where the hot air temperatures were 45°C, 50°C and 55°C respectively. The dried roses were brightly colored and good quality, also, rose water was obtained during the drying process when the waste heat was recovered. The major components of the rose water were rose ether, nerol, citronellol, acetic acid, phenylethyl alcohol, methyleugenol, and so on, which are typical compounds of rose essential oil. The results show that a heat pump is one of best choices for drying rose flowers, and that rose water can also be effectively recovered during the drying process.

1. Introduction

The rose is one of the most important crops in the floriculture industry for its economic importance as a source of natural fragrances and aromas. Containing important aromatic and volatile compounds, roses have significant value in food, perfume, cosmetic, and pharmaceutical industries [1, 2].

Dried flowers are similar in appearance to their natural state, having beauty as well as an everlasting value if preserved and processed with appropriate dehydration technology. A wide range of dry flower products are popular, like pictures, frames, flower balls, greeting cards, covers, candles, collages, bouquets and wreaths, and sweet-smelling potpourris.

Furthermore, rose water is one of the most valuable raw materials. In the food industry, rose water is used in the production of tea, jam, biscuits, cake, confectioneries, and beverages due to its aromatic and volatile characteristics [3, 4]. Besides its role as a fragrance [5], rose oil has nutritional, pharmacological, and industrial properties [6, 7]. Clinically, it has been used as a remedy for gastrointestinal complications, acne [8], bacterial infections [9], and gastric and duodenal spasms [8]. And it has been reported to provide relief from toothache, aphthous lesions, and gingival and throat inflammation. The cosmetics industry uses rose- oil in the production of tonics, creams, soaps, and
other cosmetic products such as skin care lotion for its antiseptic and refreshing effects [10, 11]. Also, rose water is used in religious ceremonies in some countries such as Iran [12].

Drying roses and extracting cellular fluids are important ways to increase the value of the rose. Drying is one of the oldest methods for the preservation of agriculture products, and it represents a very important aspect of food processing. The dehydration of products is aimed at producing a high-density product with low water activity.

Conventionally, fresh rose flowers are dried by hot air at a suitable temperature, while rose water is obtained by steam distillation of fresh rose flowers. The heat pump dehumidifier (HPD) is a convection air dryer that operates at low temperatures. It has an inbuilt refrigeration system that dehumidifies and recovers the latent heat of evaporation from the exhaust gas. Therefore, evaporated moisture and volatile compounds can be recovered. Consequently, the HPD is an environmentally friendly, closed controlled system with higher energy efficiency than conventional dryers.

This work covers experiments on the simultaneous preparation of dried roses and rose water. Focus is given to the effect of heat pump operating conditions, and the quantitative compositions of rose water in different stages are analyzed. The study reports some new and important findings emerging from this work.

2. Materials and Method

2.1. Materials
Fresh rose flowers from Pingyin, Province of Shandong, China, harvested in May 2019, were used as the raw material in the drying experiments. The botanical name of the present rose is Rosa rugosa Thunb, local named Fenghua Rose No.1 [13]. Fenghua rose is bred by crossing single red rose (R.rugosa) as female parent and double red rose (R.rugosacuplena) as male parent. A total of 7.5 kg of rose flowers were picked. The morphology of the strain of rose considered in this study is shown in Figure 1.

The moisture content of fresh rose flowers was measured by an automatic moisture meter (Meitler Toledo HE53/02). The moisture content of three group samples was measured respectively and gave the average value 78%.

After harvesting, the rose flowers were immediately spread on the drying shelf and placed in the drying chamber of a heat pump system.

![Figure 1. Morphology of rose in Pingyin, China.](image)

2.2. Drying equipment
Experiments were carried out in a dryer built in the laboratory, consisting of a closed system with forced air circulation and adequate air temperature and humidity controls. The experimental process was designed based on Zhao Haibo's heat pump drying experimental system as presented in Figure 2, which consisted of a refrigerant circuit and a drying air circuit [14].

In the present work, the heat pump system, which was charged with R134a, was configured with a medium-high temperature Copeland compressor. The heat pump consisted of an inverter compressor,
an evaporator, a condenser, an auxiliary condenser, a thermal expansion valve, a chiller unit, and an insulated piping system. The air circuit contained air ducts connecting the evaporator and the condenser with a blower to drive airflow. In a typical drying operation, the moisture-saturated air discharged from the drying chamber was dehumidified by the evaporator, and then heated by the condenser before entering the drying chamber via the main blower. In the drying chamber, the hot drying air took in moisture and entered the evaporator again to complete another cycle.

![Schematic diagram of the system structure.](image)

1-drying closet; 2-feeding port; 3-air return pipe; 4-air inlet pipe; 5-evaporator; 6-main condenser; 7- auxiliary condenser; 8-throttle valve; 9-compressor; 10-main fan; 11-fan-2; 12-fan-3; 13-Valve 1; 14-Valve 2

The drying air condition was regulated with an additional fin heat exchanger for cooling to maintain constant relative humidity constant inside the drying chamber by adjusting the water inlet temperature of the heat exchanger located in the outlet of the drying chamber. A programmable logic controller (SIMENS PLC57-200) was used to maintain constant air temperature and relative humidity inside the drying chamber by switching valve 1 and valve 2 ON/OFF.

### 2.3. Drying cycle

The closed system heat pump dryer, shown in Figure 2, consists of a drying air cycle and a refrigerant cycle. The heated air from the condenser flows through the drying chamber and extracts moisture from the fresh rose flowers. The humid drying air is then cooled to recover the heat and extract the moisture at the evaporator and the secondary evaporator. The cooling and dry air is sent to the condenser for heating again before the next drying cycle. The moisture from the fresh flowers, along with volatile organic compounds, which are present in the cell fluid of fresh flowers (a byproduct with high economic value), is condensed and stored. The refrigerant circulates among the evaporator, condenser, compressor, and expansion valve.

### 2.4. Experimental data acquisition technique

Air temperature was measured with thermocouples inserted into the drying chamber and connected by Agilent data acquisition instrument 34970A (Agilent Technologies, Inc.) with 0.5°C readability.
Air velocity and relative humidity content were measured with a Thermo Anemometer and Hygro Palm hygrometer, respectively. The dryer chamber contained a mesh-bottomed tray to hold the sample of particles. Due to the air flow disturbance of the sample in the drying chamber, the sample weight was measured by the weighing load cell. There were two fans on both sides of the drying chamber, which kept the drying chamber at zero pressure, to minimize the impact of the symmetrical weight of air flow.

2.5. Experimental procedures
To ensure the color and quality of rose flowers, when the moisture content of rose flowers was higher than 40%, the air temperature at the inlet of drying chamber was controlled to be not higher than 45°C. Within the drying process, the moisture content was reduced from 40% to 20%, the drying temperature gradually rose from 45°C to 50°C, while the moisture content was reduced from 20% to 15%, and the drying temperature gradually rose from 50°C to 55°C.

For all experiments, the drying system was run for about 20 min to obtain steady-state conditions, and when the drying chamber temperature reached 45°C, 7.5 kg of fresh rose flowers were put on the shelves of the drying chamber. When the wet basis (w. b.) moisture content of the flowers reached 15%, the rose flowers were taken out of the drying chamber.

2.6. GC/MS chromatography
The chemical class, retention time, and percentage of volatile compounds in three rose water samples recovered in three drying stages were investigated with a GC/MS analyzer (Agilent 7890A/5973N), and the NIST 11 standard spectrum database was used to identify the aroma chemical components of rose water. In the test, the chromatographic column number was DB-INNOWax (60 m × 320 μm × 0.25 μm ); the carrier gas was high-purity He; and the column temperature remained at 60°C for 2 min, rose to 220°C at a rate of 8°C per minute and then remained at 220°C for 10 min.

3. Results and Discussion

3.1. Drying behavior of rose flower
The initial moisture content of rose flowers was 78% (w. b.) (The determination method was described in section “material and methods”). The time required to dry the rose flowers to a final moisture content of about 17.5% (w. b.) was about 24 h. The color of dried rose is bright and beautiful with excellent quality as shown in Figure 3.

![Figure 3. Dried rose flowers.](image)

Based on experience, the whole drying process was divided into three different drying stages. The first stage took about 8 hours; during this process the drying temperature was set to 45°C and the relative humidity in the drying room was controlled at 55%. When the moisture content of the rose flowers was reduced to 40%, the second stage of the drying process was initiated. In this stage, the drying temperature was raised to 50°C and the relative humidity was reduced to 45% until the
moisture content dropped to 20%. At this point, the drying process entered the third stage, in which the temperature of the drying medium was set to 55°C and the relative humidity was set to 35%. During the whole drying process, the flow rate of hot air was kept at 0.5 m/s.

The variation in moisture content with respect to time is shown in Figure 4, it can be seen from the figure that the moisture content of rose flowers decreased curvilinearly over the course of with the drying process.

![Figure 4. Drying curves for different air conditions.](image)

In the first stage, the moisture content decreased in a nearly straight line, meaning the drying rate was basically maintained at a constant speed under the condition of high moisture content. With the decrease of moisture content and the adjustment of drying conditions, the drying rate decreased until the whole drying process was completed.

3.2. Rose water chemical analysis

The humid drying air was then cooled to recover the heat and extract the moisture at the evaporator. The condensed rose water was obtained through heat recovery. As shown in Table 1, 24 volatile compounds were identified determined in the five rose water samples by the GC/MS chromatography.

Rose water was sampled every 4 h during the drying process, and the composition of the rose water is shown in Table 1.

Table 1 shows the percentages of the components identified in each sample and compares the results with the work [15, 16]. Most samples contained the typical components of rose essential oil, such as rose ether, citronellol, nerol, phenylethanol, phenylethyl acetate, methyleugenol, etc. However, the precipitation regularity of a single compound in the drying process was not obvious, so future work is needed to achieve more accurate recovery and testing of rose water.

The results of chromatographic separation are shown in Figure 5-7. It can be seen from the spectrum analysis that the rose water obtained by heat pump drying had the typical components of rose essential oil and rose water extracted by conventional methods hence, heat pump drying is an effective technology for rose water production. Also, the components and contents of aroma compounds contained in the rose water recovered in different stages of drying were not exactly the same. According to the characteristics of the drying process, the desired aroma compounds could be recovered in different stages which may reduce the cost of purification.
Table 1. Chemical composition of the rose water.

| Compounds                                      | 1\# | 2\# | 3\# | 4\# | 5\# |
|------------------------------------------------|-----|-----|-----|-----|-----|
| Tridecane                                      | 0.62| 0.76|     |     |     |
| Rose ether (2H-Pyran, tetrahydro               |     |     |     |     |     |
| -4-methyl -2-(2-methyl-1-propenyl))           | 0.48| 0.58| 0.70| 1.01| 0.62|
| Tetradecane                                    | 9.40| 0.82| 1.63| 7.38| 0.88|
| Methyl tetradecane                             |     |     |     |     | 2.33|
| Pentadecane                                    | 12.13| 2.17| 5.20| 15.45| 2.34|
| Methyl pentadecane                             | 3.81|     |     | 3.96| 0.40|
| Hexadecane                                     | 28.24| 2.90| 7.79| 17.21| 2.52|
| Methyl cetane                                  |     |     |     |     | 2.41|
| Citronellyl acetate                            |     |     |     | 6.02| 2.52|
| Heptadecane                                    | 6.31| 1.14| 4.11| 9.19| 1.23|
| Citronellol (6-Octen-1-ol, 3,7-dimethyl-)      | 15.38| 44.87| 35.90| 18.86| 48.39| 3.10| 40.13|
| Nerol (2,6-Octadien-1-ol,3,7-dimethyl-, (2Z)-)| 1.87| 5.27| 3.74| 1.74| 1.29| 1.40|
| Acetic acid, 2-phenylethyl ester               | 1.70| 7.12| 5.72| 2.71| 7.68|
| Geranion                                       |     |     |     | 1.29| 1.87| 4.20| 15.97|
| Phenethyl Alcohol                              | 4.00| 14.34| 20.17| 4.94| 15.46| 80.00| 2.01|
| Benzene, 1,2-dimethoxy-4-(2-propenyl)-         | 3.84| 12.95| 14.39| 4.74| 13.96|
| Eugenol                                        |     | 1.15|     | 1.24| 1.60| 2.25|
| Hentriacontane                                 | 3.50|     |     |     |     |
| 1-Decanol, 2-hexyl-                            | 1.05|     |     |     |     |
| Dodecan, 2,6,10-trimethyl-                     | 3.73|     |     |     |     |
| Octadecane                                     | 1.42|     |     |     |     |
| Pyrene, hexadecachydro-                        | 0.41|     |     |     |     |
| Citral (2,6-Octadienal, 3,7-dimethyl-)          |     |     |     | 0.60|     |
| Dibutyl phthalate                              |     |     |     |     | 3.03|
Notes:
1- Rose ether, 2- Tetradecane, 3- Pentadecane, 4- Hexadecane, 5- Heptadecane, 6- Citral, 7- Citronellol, 8- Nerol, 9- Acetic acid, 10- Geraniol, 11- Phenylethyl Alcohol, 12- Methyleugenol, 13- Eugenol, 14- Dibutyl phthalate

**Figure 5.** GC-MS analysis of rose water from first drying stage.

**Figure 6.** GC-MS analysis of rose water from second drying stage.

Notes: 1- Rose ether, 2- Tetradecane, 3- Pentadecane, 4- Hexadecane, 5- Citronellyl acetate, 6- Heptadecane, 7- Citronellol, 8- Acetic acid, 9- Phenylethyl Alcohol, 10- Methyleugenol.
Notes: 1- Rose ether, 2- Tetradecane, 3- Pentadecane, 4- Hexadecane, 5- Citronellyl acetate, 6- Heptadecane, 7- Citronellol, 8- Nerol, 9- Acetic acid, 10- Geraniol, 11- Phenylethyl Alcohol, 12- Methyl eugenol, 13- Eugenol.

4. Conclusion
In this work, rose flowers were dried in three stages, which were characterized by different temperatures and humidity levels, using a heat pump dryer, and good-quality, brightly colored rose flowers were obtained. Furthermore, while the heat pump recovered heat, a large amount of rose water was recovered. It was confirmed that there were 15 kinds of volatile compounds in the rose water, and the composition of the rose water was very similar to that of rose water obtained by the extraction method. However, the composition of organic compounds of rose water recovered at different stages is not stable and regular, which needs further study. The processing of rose flowers by this method has remarkable energy-saving effects, and a large percentage of rose water can be recovered to minimize the loss of resources. This process is one of the best choices for the processing of fresh rose flowers.

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