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The Effects of Pretreatments on Kinetics of Phenolic Degradation in Rice Paddy Herb (*Limnophila aromatic* Merr.) Stems During Hot Air Drying

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This study evaluated the effect of blanching combined with an osmotic dehydration system (ODS) pretreatment on phenolic degradation kinetics in rice paddy herb (*Limnophila aromatica* Merr.) stems during hot air drying. Fresh stems were blanched (15 s) and cooled in an ODS containing 3% NaCl, 0.05% ascorbic acid, 0.5% citric acid, and 0.37% $MgCO_3$ for 30 min before drying at 55-85°C. Pretreatment increased the measured initial total phenolic content (TPC) in the material prior to drying (around 30% higher) by facilitating the release of bound phenolics. It also helped inactivate oxidative enzymes while reducing the drying duration, hence minimizing phenolic degradation. At drying completion, phenolic degradation in pretreated stems was lower (35.87-48.01%) than untreated ones (56.38-72.83%). Kinetic modelling reflected the more predictable degradation pattern for pretreated samples when enzymatic activity and oxidative instability were reduced by pretreatment. In pretreated samples, degradation followed first-order kinetics ($R^2 = 0.954-0.977$) with the rate constant (k_1) rising from 0.0369 h^{-1} at 55°C to 0.0960 h^{-1} at 85°C. Activation energy was 29.54 kJ mol^{-1}, indicating moderate temperature sensitivity. Thermodynamic analysis showed positive Gibbs free energy, positive enthalpy, and negative entropy changes, suggesting an endothermic, non-spontaneous reaction with decreased molecular randomness. The combined blanching and ODS pretreatment effectively slowed phenolic degradation, offering a practical strategy to improve the quality and stability of dried rice paddy herb.

1. Introduction

Rice paddy herb (*Limnophila aromatica* Merr.) is a tropical aquatic plant belonging to the Scrophulariaceae family, commonly found in Southeast Asia. Traditionally used as both a culinary herb and folk remedy, it is valued for its unique aroma and medicinal properties, including antioxidant, anti-inflammatory, and antimicrobial activities (Parr and Bolwell, 2000). These bioactivities are largely attributed to its rich phytochemical composition, comprising over 50 identified compounds, notably phenolics, terpenoids, and flavonoids (Gorai et al., 2014). Despite its functional potential, the use of rice paddy herb in fresh form is often constrained by seasonality, postharvest perishability, and limited shelf life during distribution. Therefore, developing effective preservation strategies to retain its quality in dried forms is essential.

Drying is a widely adopted method to enhance product stability, extend shelf life, and reduce microbial risks. Freeze-drying is generally considered as the most effective technique for minimizing nutrient loss and preserving the shape, colour and flavour of plant food due to its low-temperature and low-pressure process. However, the need for specialized equipment, high capital investment and high energy consumption, along with the long processing time, are notable drawbacks. On the other hand, hot-air drying is commonly preferred in food processing due to its low cost and operational simplicity. However, this method can lead to substantial degradation of heat-sensitive compounds, particularly phenolics, due to prolonged exposure to high temperatures. To mitigate such degradation, pretreatments prior to drying have been studied. Among them, blanching is known to improve drying efficiency and partially inactivate oxidative enzymes such as polyphenol oxidase (PPO) and peroxidase (POD) (Ando et al., 2016). Nevertheless, the process of blanching, if not followed by effective cooling, may trigger oxidative damage due to enzyme reactivation and exposure to oxygen during

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the cooling phase. Osmotic dehydration is a non-thermal process used to partially remove water from fruits and vegetables by immersing them in hypertonic solutions, such as sugar syrups or saline brines. During the process, water migrates out of plant tissue into the solution while solutes diffuse inward until equilibrium is reached, resulting in reduced water activity and improved stability of the colour, flavour and nutrients (Yadav and Singh, 2014). Various osmotic agents and conditions have been successfully applied for numerous plants. Typically, the process is carried out at 25 – 60°C, using sucrose (10 – 80%) or salt (5 – 18%), either individually or in combination, for 30 minutes to several hours, depending on desired hydration levels. Overall, osmotic dehydration serves as an effective pretreatment before drying to maintain product quality and reduce degradation during subsequent thermal processing (Yadav and Singh, 2014). In this context, osmotic dehydration has gained interest as an alternative cooling method, where plant materials are immersed in a solute-rich solution containing functional ingredients. Such solutions can exert multiple effects, namely facilitating rapid cooling, reducing enzymatic activity through pH or ionic strength modulation, limiting moisture and phenolic leaching, and protecting phenolics through antioxidant additives. While osmotic treatment has been explored in other matrices, the synergistic effect of blanching combined with osmotic cooling using an antioxidant-rich solution has not been well studied in rice paddy herb.

Moreover, most studies assessing phenolic degradation during drying have focused on final content changes, with limited attention to kinetic modelling. Applying kinetic and thermodynamic models offers a deeper understanding of degradation mechanisms, enabling prediction and evaluation of drying conditions for better phenolic retention. Therefore, the present study aimed to evaluate the impact of blanching followed by immersion in an osmotic dehydration system (ODS) on the total phenolic content and its degradation kinetics in rice paddy herb stems during hot air drying.

2. Materials and methods

2.1 Materials and chemicals

Fresh rice paddy herbs were collected in Tien Giang province, Vietnam. The plant was chosen for these characteristics, including: the stems were about 3.5-5 mm in dimension and above the water surface from 40-50 cm in length. The chemicals of the analytical grade were purchased from local distributors.

2.2 Sample preparation and pretreatment application

The stems were removed from chops, leaves, small branches and roots. They were then washed with tap water and rinsed with distilled water. The blotting paper was subsequently used to remove water on their surface. Before drying, fresh stems were cut into 12 cm in length and blanched for 15 s in boiling water. Right after blanching, the stems were immediately immersed in the cool ODS, including 3% sodium chloride, 0.05% ascorbic acid, 0.5% citric acid and 0.37% magnesium carbonate (w/v). After 30 min, OSD was drained out to obtain the treated samples.

2.3 Hot air drying

The untreated and treated samples were placed into an oven (Memmert UFE 700) preheated at four temperature levels of 55, 65, 75 and 85°C. This temperature range is commonly applied for hot-air drying (Wanyo et al., 2018). Lower temperatures could prolong the duration and hence be impractical, while higher temperatures may cause excessive phenolic degradation. Samples were collected at 1-h intervals for total phenolic content determination. The drying duration was recorded when no further change in moisture was observed.

2.4 Total phenolic content (TPC) determination

Samples were grinded by a grinder (Philips - HR2118) within 15 seconds and a certain weight of grinded samples (equivalent to 0.1 g dried weight) was mixed with 50 mL 80 %(v/v) acetone. The mixture was kept in an ultrasound bath (WUC-A10H, Daihan, South Korea) with a frequency of 40 kHz for 1 h at ambient temperature and then placed at - 20°C overnight. The extract was collected through Newstar filter paper 102 and stored at −20°C in dark tubes until TPC determination.

TPC was determined by using the Folin-Ciocalteu assay (Aryal et al., 2019) with slight modification. Briefly, 0.5 mL of the extract was pipetted into tubes containing 2.5 mL of 10% (v/v) Folin-Ciocalteu reagent. The mixture was incubated for 5 min in the dark. Next, 2.5 mL of 7.5% (w/v) sodium bicarbonate solution was added to the mixture. After 30 min of incubation in dark at ambient temperature, the absorbance was measured at 765 nm by an UV–Visible spectrophotometer (Genesys 10S UV-Vis, Thermo, USA). The result was expressed as milligram gallic acid equivalent per gram dried weight (mg GAE/g DW).

2.5 Phenolic degradation model construction

The degradation kinetics of phenolics could be described by the zero-, first-, or second-order models as presented in Table 1 (Fernández-Romero et al., 2020):

Table 1. Equation, plot and slope of zero-, first-, and second-order models for phenolic degradation kinetics

Order	Model	Plot	Slope	Eq.
0	$C_t = -k_0 t + C_0$	C_t vs. t	$-k_0$	(1)
1	$lnC_t = -k_1 t + lnC_0$	$\ln C_t$ vs. t	$-k_1$	(2)
2	$\frac{1}{C_t} = k_2 t + \frac{1}{C_0}$	$\frac{1}{C_t}$ vs. t	k_2	(3)

where C_0 and C_t are the total phenolic contents at the initial time and the time t, respectively; k_0, k_1, and k_2 are the kinetic rate constants of zero-, first-, and second-order equations, respectively; and t is the time (h). The half-life $t_{1/2}$ was calculated from the first-order model by following Eq. (4) (Lago and Noreña, 2017):

$$t_{1/2} = \frac{\ln(2)}{k} \quad (4)$$

where k is the kinetic rate constant (h^{-1}).

The activation energy (E_a) was estimated by following Eq. (5) (Lago and Noreña, 2017):

$$k = Ae^{-\frac{E_a}{RT}} \quad (5)$$

where k is the kinetic rate constant (h^{-1}), A is a pre-exponential factor or frequency (h^{-1}), E_a is the energy activation for degradation ($kJ.mol^{-1}$), R is the universal gas constant ($8.314\ J.mol^{-1}.K^{-1}$) and T is the temperature (K).

The available energy causing the degradation, the heat content, and the degree of randomness or disorder of a system were measured by the following Eq. (6 - 8), respectively (Lago and Noreña, 2017):

$$\Delta G = -R(T+273)ln\left(\frac{k \times h_p}{K_B(T+273)}\right) \quad (6)$$

$$\Delta H = E_a - R(T+273) \quad (7)$$

$$\Delta S = \left(\frac{\Delta H - \Delta G}{T}\right) \quad (8)$$

where ΔG is the Gibbs free energy change ($kJ.mol^{-1}$), ΔH is the enthalpy change ($kJ.mol^{-1}$), ΔS is the entropy change ($J.mol^{-1}.K^{-1}$), E_a is the activation energy for degradation ($kJ.mol^{-1}$), h_p is the Planck constant ($6.6262 \times 10^{-34} J.s$), K_B is the Boltzmann constant ($1.3806 \times 10^{-23} J.K^{-1}$), T is the temperature (K), and k is the kinetic rate constant (h^{-1}).

2.6 Statistical analysis

All experiments were performed in triplicate. Statistical analysis was carried out using SPSS Statistic v.20.0 with the confidence level of 95%. The evaluation of degradation kinetic models was performed by the linear regression analysis using LABfit software.

3. Results and discussion

3.1 Effects of pretreatment and drying temperature on phenolic degradation over drying

Table 2 compares TPC of samples after pretreatment and their total drying durations at 55°C, 65°C, 75°C and 85°C. Overall, the data indicated that the pretreatment had significant positive effects, not only on enhancing the initial TPC of the stems but also shortening drying duration across all temperatures. Before drying, pretreated samples had significantly higher TPC than untreated samples (20.32 mg GAE/g DW and 15.47 mg GAE/g DW, respectively) ($p < 0.05$). This increase could be attributed to the protective effects of the osmotic dipping solution applied immediately after blanching. Blanching could inactivate part of polyphenol oxidase (PPO) and peroxidase (POD) enzymes; however, residual activity and non-enzymatic oxidation could still cause substantial phenolic losses during cooling if plant tissues were exposed to oxygen. The pretreatment rapidly cooled the tissues and created an environment unfavourable for oxidation through multiple mechanisms. Sodium chloride exerted an osmotic effect, limiting water uptake and phenolic leaching, while also disrupting enzyme conformation and reducing their activity (Fan et al., 2005). Citric acid lowered the pH, thereby inhibiting oxidative enzymes, and chelated metal ions (Cu^{2+}, Fe^{3+}) essential for their catalytic function (Karaffa et al., 2001). Ascorbic acid acted as a reducing agent and preferential PPO substrate, scavenging reactive oxygen species and protecting phenolics from oxidation (Rababah et al., 2005; Yin et al., 2022). Magnesium carbonate buffered the acidification and contributed Mg^{2+} ions that could help maintain cell wall integrity, reducing leakage of phenolics (Walker, 1994).

Meanwhile, Table 2 also displays the substantial decrease in drying duration with increasing drying temperature for both untreated and pretreated stems. At the low temperature of 55°C, drying required 20 h for untreated stems and 16 h for pretreated stems, whereas at the high temperature of 85°C, drying time was reduced to only

5 h for both sample types. This inverse relationship between drying temperature and drying duration was expected, since higher temperatures could increase the vapor pressure deficit between the product surface and the drying air, accelerating moisture removal. The shorter drying time observed in pretreated stems at lower temperatures (e.g., 55 °C) compared with untreated stems could be attributed to pretreatment before drying. Blanching process could soften plant tissue and alter the microstructure, convert bound water to free water, hence enhancing water flux and replacing air within the porous voids by water, which all allowed water to migrate more easily during drying (Xiao et al., 2017). These structural changes, combined with partial water removal by osmotic dehydration induced with sodium chloride and other solutes in the dipping solution, could lower initial moisture content and improve moisture transfer (Deng et al., 2019). Consequently, these combined effects accelerated the drying rate and shortened the overall drying time.

Table 2: Effects of pretreatment on total phenolic content in fresh sample, drying time and phenolic degradation at different temperatures

	TPC before drying (mg GAE/g DW)	Drying temp. (ºC)	Drying duration (h)	TPC degradation (%)
Untreated stem	15.47±0.64^{b}	55	20	56.38±3.58^{c}
		65	16	62.60±1.78^{c}
		75	8	72.83±1.42^{d}
		85	5	70.65±1.64^{d}
Pretreated stem	20.32±0.32^{a}	55	16	48.01±1.46^{b}
		65	9	49.32±1.26^{b}
		75	6	44.58±0.66^{b}
		85	5	35.87±4.27^{a}

Different letters in the same column indicate significant differences ($p < 0.05$)

Figure 1 illustrates the phenolic degradation behaviours of untreated and pretreated stems over drying. In untreated stems (Figure 1A), TPC decreased rapidly within the first few hours of drying, particularly at higher temperatures (75–85 °C), where more than half of the initial phenolics were lost within 2-4 h. At lower temperatures (55 °C and 65 °C), the decline was slower, although a gradual reduction still occurred over the prolonged drying period. Interestingly, fluctuations in TPC were observed at certain drying time, possibly due to the release of bound phenolics as cell structures broke down under heat. In pretreated stems (Figure 1B), the rate of phenolic loss was markedly slower across all drying temperatures, suggesting that the pretreatment effectively protected phenolics from thermal and oxidative degradation. Also, the decrease was gradual and progressive without fluctuations of sudden increase. Particularly at 55°C, the reduction in TPC was the least pronounced, with more than 50% of the initial phenolics retained even after 15 h of drying.

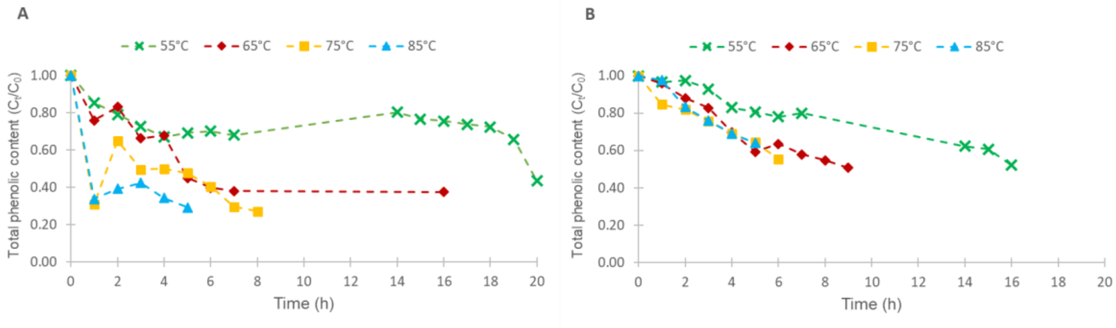

Figure 1: Effects of temperature on phenolic degradation of (A) untreated and (B) pretreated stems of rice paddy herb during hot air drying.

At the end of drying, TPC degradation in untreated stems increased markedly with drying temperature (Table 2). The lowest degradation occurred at 55 °C (56.38%), while the highest losses were observed at 75 °C (72.83%) and 85 °C (70.65%), indicating that elevated temperatures accelerate phenolic breakdown through thermal and oxidative mechanisms. In contrast, pretreated stems consistently showed lower TPC degradation across all temperatures, with values ranging from 48.01% at 55 °C to only 35.87% at 85 °C. Notably, the degradation at the highest temperature (85 °C) in pretreated stems was significantly lower than even the lowest-temperature treatment (55 °C) in untreated stems, demonstrating the strong protective effect of the osmotic dipping pretreatment. This improved retention is likely due to the synergistic effects of blanching and cooling including enzyme inhibition, antioxidant protection, and reduced leaching, which together minimized thermal and oxidative losses during drying.

3.2 Phenolic degradation kinetic models and thermodynamic analysis

Table 3 describes the degradation kinetics of phenolics during drying, which were evaluated using zero-, first-, and second-order models for both untreated and pretreated stems. For untreated stems, none of the models showed consistently high coefficients of determination ($R^2 < 0.65$), indicating great variability and poor model fit, likely due to rapid and irregular phenolic losses from simultaneous thermal degradation, enzymatic oxidation, and leaching effects as presented in Figure 1. Without pretreatment, endogenous enzymes (i.e. PPO, POD) remain active, promoting enzymatic oxidation of phenolics into quinones, which can polymerize into brown pigments. Non-enzymatic oxidation driven by reactive oxygen species and transition metals also contributed to phenolic degradation during drying. Additionally, the cellular integrity in untreated stems limited the uniform diffusion of heat and moisture, creating uneven temperature and oxygen gradients that triggered localized oxidative reactions. Consequently, the degradation behaviour of phenolic compounds in untreated stems was governed by multiple overlapping mechanisms. In contrast, pretreated stems exhibited markedly higher R^2 values (> 0.93) for all models, with the first-order and second-order models exhibiting the better overall fit than the zero-order one. This stronger correlation reflects the more predictable degradation pattern when enzymatic activity and oxidative instability were reduced by pretreatment, thereby making the degradation pattern primarily dependent on the concentration of remaining phenolics and drying temperature.

Table 3: Kinetic and thermodynamic parameters of phenolic degradation in pre-treated stems.

Sample	Tempt (°C)	Zero-order		First-order		Second-order	
		k_0	R^2	k_1	R^2	k_2	R^2
Untreated stem	55	0.1607	0.2749	0.0124	0.2645	0.0010	0.2447
	65	0.6995	0.5965	0.0627	0.6377	0.0060	0.6651
	75	1.0349	0.4847	0.1053	0.4822	0.0121	0.4441
	85	1.3664	0.4909	0.1718	0.5297	0.0244	0.5651
Pre-treated stem	55	0.5579	0.9588	0.0369	0.9643	0.0025	0.9509
	65	1.2231	0.9396	0.0794	0.9545	0.0053	0.9590
	75	1.7518	0.9688	0.0892	0.9770	0.0046	0.9646
	85	1.8455	0.9706	0.0960	0.9792	0.0051	0.9815

Sample	Tempt. (°C)	Time (h)	k (h^{-1})	$t_{1/2}$ (h)	A (h^{-1})	E_a ($kJ.mol^{-1}$)	R^2	ΔG ($kJ.mol^{-1}$)	ΔH ($kJ.mol^{-1}$)	ΔS ($J.mol^{-1}.K^{-1}$)
Pre-treated stem	55	16	0.0369	18.8	2242	29.54	0.78	67.26	26.81	-191.39
	65	9	0.0794	8.7				67.23	26.73	-187.91
	75	6	0.0892	7.8				68.98	26.65	-189.71
	85	5	0.0960	7.2				70.83	26.56	-191.70

Based on R^2, the first-order kinetic models of pretreated samples were further employed to determine thermodynamic parameters of phenolic degradation, including the rate constant (k), half-life ($t_{1/2}$), activation energy (E_a), Gibbs free energy change (ΔG), enthalpy change (ΔH), and entropy change (ΔS) as summarized in Table 3. The rate constant increased with temperature, from 0.0369 h^{-1} at 55°C to 0.0960 h^{-1} at 85°C, indicating faster degradation under elevated thermal conditions. Correspondingly, the half-life decreased substantially from 19 h at 55 °C to only 7 h at 85 °C, demonstrating the strong temperature dependence of phenolic stability. It was noticed that despite faster phenolic degradation at higher temperatures; the application of pretreatment shortened the drying duration less than the degradation time, resulting in low phenolic degradation at high temperatures. The Arrhenius analysis yielded an activation energy of 29.54 $kJ.mol^{-1}$, which is within the range reported for phenolic degradation in other plant matrices (Karaaslan et al., 2014), suggesting moderate thermal sensitivity. Thermodynamic analysis revealed positive Gibbs free energy values (ΔG = 67.23-70.83 $kJ.mol^{-1}$), indicating that the degradation was non-spontaneous under the tested conditions. The positive enthalpy change (ΔH = 26.56-26.81 $kJ.mol^{-1}$) confirmed an endothermic reaction, while negative entropy values (ΔS = -191.70 to -187.91 $J.mol^{-1}.K^{-1}$) suggested a decrease in molecular randomness at the transition state, possibly due to structural reorganization before phenolic breakdown (Lago and Noreña, 2017).

4. Conclusion

Blanching followed by immersion in an osmotic dehydration system (ODS) proved effective in preserving phenolics in rice paddy herb stems during hot air drying. Pretreatment significantly increased initial TPC, shortened drying duration, and reduced phenolic losses across all drying temperatures compared with untreated samples. Even at 85°C, phenolic degradation in pretreated stems was still lower than that of untreated stems at 55°C, highlighting the strong protective effect of the pretreatment. Kinetic analysis revealed that phenolic degradation in pretreated samples followed first-order kinetics with high R^2 values, enabling accurate modelling of degradation behaviour. The calculated activation energy and thermodynamic parameters indicated that the process was temperature-dependent, endothermic, and non-spontaneous, with reduced molecular randomness. Although shorter drying duration could preserve the product quality and reduce energy demand, large-scale applications require process optimization to ensure economic feasibility and environmental performance. Further research should focus on developing sustainable recycling systems for osmotic solutions and assessing the life-cycle impacts of process. In addition, the sensory attributes and potential applications of the dried product should also be discovered.

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