

Flavor encapsulation capacities of amphiphilic polyvinylpyrrolidone as a carrier for amorphous solid dispersions

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ABSTRACT

This study investigates the characteristics of amorphous solid dispersions (ASDs) formed using an amphiphilic carrier polymer, focusing on the effects of source solution conditions on flavor retention. Cinnamaldehyde and polyvinylpyrrolidone (PVP) were dissolved as flavor and carrier components in methanol or ethanol under different conditions and subsequently spray-dried into fine powders. The amount of cinnamaldehyde retained in the spray-dried ASDs was quantified to assess the impact of solution conditions. The results indicate that cinnamaldehyde molecules were physically entrapped within the entangled PVP chains during the spray-drying process. The flavor encapsulation capacity of PVP reached a maximum of approximately 4 mmol/g-PVP under specific conditions. This maximum capacity implies that each cinnamaldehyde molecule is associated with approximately two PVP monomer units, which is higher than those reported for selected cyclodextrin-based systems in comparable contexts of cinnamaldehyde encapsulation. Additionally, the dissipation of cinnamaldehyde from the ASD samples during storage under vacuum was examined.

1. Introduction

Many commercial products, including food items, dairy products, and cosmetics, are infused with specific aromas tailored to their respective categories. When it is necessary to transform these aromas into solid or paste forms, converting them into powdered form is beneficial (Furuta & Neoh, 2021; Osorio-Tobón et al., 2016). The powdered aromas can be evenly applied to the product by spraying them onto its surface for coating, mixing them with other powdered ingredients, or thoroughly blending them into the product paste.

One method of aroma powderization involves encapsulating oily flavor in a solid carrier matrix in droplet form (Joye & McClements, 2014; Saifullah et al., 2019). In this technique, the oily flavor is typically emulsified with the assistance of a surface-active ingredient and subsequently dried in the presence of carrier-forming substances (Furuta & Neoh, 2021; Goubet et al., 1998; Wang et al., 2019). Various carrier-forming agents, including gums (Al-Hamayda et al., 2023; Carneiro et al., 2013; Krishnan et al., 2005), proteinaceous materials (Carneiro et al., 2013; Charve & Reineccius, 2009), and starch-based substances

(Carneiro et al., 2013; Krishnan et al., 2005; Shan et al., 2025), can serve dual purposes: they can emulsify the flavor oil and act as the encapsulation carrier for embedding the flavor droplets.

The encapsulation of flavor molecules using capturing materials, such as host agents or adsorbents, has also been explored (Ciobanu et al., 2013; Reineccius et al., 2002). Cyclodextrins (CDs) and porous materials with molecular sites designed to capture flavor molecules serve as effective entrapping materials. The process involves mixing the capturing agent with flavor oil in an aqueous solvent (Jafari et al., 2008; Shiga et al., 2001) or directly kneading the two components (Castro et al., 2016; Yuliani et al., 2006) to ensure adequate accumulation of the flavor within or on the entrapping materials. Subsequently, the mixture is converted into powdered forms (Shiga et al., 2001).

In addition to the two aforementioned flavor powderization strategies, a novel methodology for flavor powdering has recently been introduced (Nitta et al., 2024; Satoh et al., 2016), which is based on the concept of amorphous solid dispersion (ASD) within the field of pharmaceutical technology (Leuner & Dressman, 2000; Newman et al., 2012). Specifically, both the flavor and the carrier are dissolved in a

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miscible solvent, followed by desolvation to produce a homogeneous dry mixture devoid of segregation. To date, the ASD of flavor has been indicated to be effectively achieved without significant flavor loss through the vacuum-foam (Sato et al., 2016) and spray-drying (Nitta et al., 2024) techniques, employing disaccharides or the amphiphilic polymer polyvinylpyrrolidone as the flavor-entrapping carrier component. It is important to note that the ASD-based flavor powder comprises only two components: flavor and carrier. The simplicity of this ASD formulation may facilitate the determination and optimization of the flavor powder product design.

It is important to note that the three methodologies mentioned above for flavor powderization primarily depend on the physical entrapment of volatile flavors within a solid carrier matrix. Consequently, the flavor-entrapping carriers utilized in the first two methods—namely, emulsion-based (Furuta & Neoh, 2021; Goubet et al., 1998) and molecular-capturing-based techniques (Madene et al., 2006)—have been extensively studied for their efficacy in flavor entrapment. In contrast, research on the flavor-entrapping characteristics of the ASD-based flavor encapsulation technique has been limited to qualitative comparisons among a restricted range of carrier former types (Nitta et al., 2024; Sato et al., 2016). As a result, the quantitative relationships among carrier type and molecular weight, solution concentration, solvent type, and flavor retention capacity during spray-drying have not been comprehensively elucidated.

This study aimed to quantify and model the capacity of the carrier-forming agent in the entrapment of a flavoring substance within the ASD formulation, thereby providing essential information regarding the feasibility of the ASD-based technique. The ASDs of the flavor, prepared via spray-drying, were assessed to evaluate the capacity of the ASD carrier to retain the flavor both during the spray-drying process and throughout prolonged storage under vacuum conditions. Polyvinylpyrrolidone (PVP) with varying mean molecular weights served as the carrier for the ASD, while cinnamaldehyde was utilized as a model flavor. Mixtures of cinnamaldehyde and PVP were prepared as ASD samples by spray-drying different compositions of methanol or ethanol solutions. The amounts of flavor retained in the ASD post-spray-drying and during storage were measured and compared across various compositions of flavor and PVP, with a focus on evaluating the carrier's ability to entrap flavor molecules. Based on the findings, the potential mechanisms of flavor encapsulation and release during spray-drying, as well as the conditions that optimize the carrier's encapsulation capacity, were explored.

2. Materials and methods

2.1. Materials

Cinnamaldehyde (>98%, Cat. No. 031-03453, FUJIFILM Wako Pure Chemical Co., Osaka, Japan) was utilized as a volatile flavoring agent. Polyvinylpyrrolidone (PVP) with molecular weights of approximately 10 kDa (Lot. L00432010, Cat. No. 07-1815, STREEM Chemicals Inc., Newburyport, MA, USA), 24.5 kDa (Lot. M0F2079, Cat. No. 28316-62, Nacalai Tesque, Kyoto, Japan), 55 kDa (Lot. MKCH1344, Cat. No. 856568, Sigma-Aldrich Co., St. Louis, MO, USA), and 360 kDa (Lot. M9H4888, Cat. No. 28315-72, Nacalai Tesque) were employed as amphiphilic carrier formers. Methanol (≥99.8%, Cat. No. 21915-93, Nacalai Tesque) and ethanol (≥99.5%, Cat. No. 14722-75, Nacalai Tesque) were procured from Nacalai Tesque. All chemicals used in this study were of reagent grade and were employed without further purification.

2.2. Preparation of amorphous solid dispersions of flavors

A 50 mL aliquot of either methanol or ethanol was prepared, containing PVP as the matrix-forming agent and cinnamaldehyde as the flavoring component. In the mixed solutions of PVP and

cinnamaldehyde, the PVP concentration was fixed at 25 g/L, 100 g/L, or 200 g/L. The concentration of cinnamaldehyde was adjusted to achieve ratios relative to the PVP concentration ranging from 0.1 to 1.0. The resulting mixture of cinnamaldehyde and PVP was subjected to spray drying, as described in the following section.

The spray drying process of the mixed solution of cinnamaldehyde and PVP was conducted utilizing a Yamato ADL-311S spray dryer (Yamato Scientific Co., Ltd., Tokyo, Japan), equipped with a Yamato GF300-30100 two-fluid nozzle featuring a model 1 A nozzle (liquid nozzle diameter: 0.41 mm; air nozzle diameter: 1.63 mm) (Yamato Scientific Co., Ltd.) and a drying chamber with a diameter of 150 mm. Additionally, a cyclone (Yamato GF300-30060, inner diameter 100 mm, Yamato Scientific Co., Ltd.) was connected downstream of the spray dryer. The spray-drying conditions were applied as determined in our previous study (Nitta et al., 2024). Specifically, the prepared cinnamaldehyde/PVP mixed solution, with a total volume of 30 mL, was introduced into the spray dryer at a flow rate of 3.1 mL/min and subsequently atomized at an atomization gas pressure of 0.1 MPa. The inlet temperature and flow rate of the downstream hot air were set at 160 °C and 0.68 m³/min, respectively, while the outlet temperature of the hot air was maintained at 85 ± 5 °C. The spray-dried materials were separated from the drying air using a glass cyclone and collected in a glass collection container. These ASD sample preparations were performed in at least duplicate for each individual experimental condition. The spray-dried ASD samples obtained were subsequently analyzed as detailed in the subsequent sections. Portions of these samples were alternatively stored in a vacuum desiccator containing silica gel at a temperature of 30 ± 1 °C to assess the storage stability of the flavor ASD samples.

To evaluate the intensity of the spray-drying process used in this study, the PVP that was spray-dried alone was thoroughly dried overnight at 120 °C, and the mass loss was measured. The results demonstrated that approximately 7.0 ± 1.5 wt% of the solvent, either methanol or ethanol, remained in the ASD samples after the spray-drying procedure. Furthermore, it was determined that the residual solvent in the spray-dried PVP was eliminated within 10 days when stored in a vacuum desiccator over silica gel at 30 °C.

Under the experimental conditions of this study, the effect of cinnamaldehyde oxidation on flavor retention measurements is considered negligible. This assumption is substantiated by the comparative analysis of UV–Vis and IR spectra of both heated and unheated cinnamaldehyde, as well as spray-dried ASD samples (Fig. S1).

2.3. Estimation of flavor retention in amorphous solid dispersions

The flavor component cinnamaldehyde retained in the ASD sample was quantified immediately after drying and after subsequent storage over silica gel. This quantification was conducted using a methodology that combines a systematic organic solvent extraction procedure with UV–visible spectrophotometry (Zhang et al., 2024), as previously applied in our earlier study (Sato et al., 2016). In a standard analytical procedure, a known mass (5–10 mg) of the ASD sample was collected and suspended in a specified volume (1–5 mL) of methanol. The resulting methanol solution containing cinnamaldehyde was diluted by a known rate with methanol to reduce the cinnamaldehyde concentration below 5 µg/mL (38 nM). The ultraviolet (UV) absorbance corresponding to cinnamaldehyde was measured at 285 nm, using JASCO V-730 UV–visible spectrometer (JASCO Co., Tokyo, Japan). This absorbance value was then utilized to quantify the residual amount of the flavor component in the ASD sample, employing the extinction coefficient of cinnamaldehyde (148 Abs/(mg/mL), 19.6 × 10⁶ Abs/(mol/L)). The mass of PVP in the sampled ASD was calculated by subtracting the measured flavor amount from the total mass of the sampled ASD. Subsequently, the initially loaded flavor amount was estimated based on the predetermined flavor loading ratio. Finally, the flavor retention rate (%) was determined as the ratio of the detected residual flavor amount to the initially loaded flavor amount. In instances where the measured flavor

retention rate (%) deviated by more than $\pm 25\%$ from the mean—calculated excluding the value obtained under the specified condition—such measurements were identified as outliers due to error and were therefore excluded from the analyses.

This measurement was performed in triplicate or more for each batch of ASD preparation condition, which varied in terms of PVP molecular weight, initial PVP/cinnamaldehyde composition, and solvent type (methanol or ethanol).

2.4. Differential scanning calorimetry

A portion of the spray-dried ASD samples was stored for 20 days in a vacuum desiccator over silica gel to ensure thorough drying and subsequently analyzed using differential scanning calorimetry (DSC). The analysis was performed with a TA Q2000 calorimeter (TA Instruments Co., New Castle, DE, USA) equipped with an RCS90 cooling system (TA Instruments Co.). Specifically, a few milligrams of the ASD sample were hermetically sealed in an aluminum pan and scanned from $-20\text{ }^{\circ}\text{C}$ to $90\text{--}150\text{ }^{\circ}\text{C}$ at a heating rate of $10\text{ }^{\circ}\text{C}/\text{min}$, using an empty pan as the reference. The resulting DSC thermogram was employed to identify thermal events, including the glass transition temperature (T_g) of the ASD sample. As shown in Fig. S2, two sequential upward temperature scans were preliminarily conducted: the first scan reached approximately $50\text{ }^{\circ}\text{C}$ above the T_g to prewarm the samples and eliminate structural relaxation (Tant & Wilkes, 1981), and the second scan extended up to $130\text{ }^{\circ}\text{C}$. No differences were observed between the T_g values obtained from the first and second scans (Fig. S2). Furthermore, heating above $130\text{ }^{\circ}\text{C}$ frequently caused an abrupt decrease in heat flow, attributed to the rupture of the sealed aluminum pan. Therefore, in the DSC measurements performed in this study, only a single upward scan—typically up to a maximum of $130\text{ }^{\circ}\text{C}$ —was conducted. At least two DSC scans were conducted for each representative ASD sample.

2.5. Scanning electron microscopy

The spray-dried ASD samples were examined using scanning electron microscopy (SEM). Specifically, the ASD powder was affixed to the SEM sample stage using carbon double-sided tape and subsequently coated with a thin film of approximately 40 nm thickness by evaporating platinum with a Hitachi E-1030 ion sputter instrument (Hitachi High-Technologies Co., Tokyo, Japan). The SEM images of the ASD powders were acquired with a KEYENCE VE9800 scanning electron microscope system (KEYENCE Co., Tokyo, Japan) at an accelerating voltage of 15 kV . The obtained SEM images were analyzed using ImageJ software (version 1.54, NIH Image, Bethesda, MD, <https://imagej.net/ij/>) to determine the size distributions of the resultant ASD particles (Phonetip et al., 2019), following to our previous analyzing procedure (Araga et al., 2025). Specifically, all particles or fibers in each SEM image were measured for particle diameter or fiber thickness using ImageJ software. In total, more than 128 size measurements were collected for each individual condition and compiled into a cumulative distribution curve, with diameter size on the x-axis and the cumulative number of data points relative to the total (>128) on the y-axis. From this cumulative size distribution curve, the D_{n10} , D_{n50} , D_{n90} values, as well as the number-normalized average size, were manually extracted.

2.6. Fourier transform infrared spectroscopy

The infrared (IR) absorption spectra of the spray-dried ASD sample, composed of PVP and cinnamaldehyde, as well as those of spray-dried PVP alone, pure cinnamaldehyde, and PVP dissolved in ethanol, were obtained using a Fourier transform infrared spectrometer (FT/IR 4600, JASCO Co., Tokyo, Japan). For spectral acquisition, each sample was placed on the sample window of a Ge prism within an attenuated total reflection (ATR) accessory (ATR-PRO400-S, JASCO Co.). In the case of spray-dried powders, the samples were pressed against the Ge window

using a pressing device to ensure intimate contact. Spectra were recorded by scanning each sample 64 times over the wavenumber range of $600\text{ cm}^{-1}\text{--}4000\text{ cm}^{-1}$ at a resolution of 4 cm^{-1} . All FTIR measurements were performed in triplicate for each sample to ensure reproducibility.

To validate the flavor retention measurement based on the UV absorption of dissolved cinnamaldehyde described above, an alternative assessment was conducted by comparing IR spectra obtained before and after spray-drying (Fig. S3). Specifically, the band area of two IR bands at 1120 cm^{-1} (A_{1120}) and 1290 cm^{-1} (A_{1290}), corresponding respectively to cinnamaldehyde and PVP and exhibiting no spectral overlap, were first determined from a methanol solution containing 50 g/L cinnamaldehyde and 100 g/L PVP (24.5 kDa). Subsequently, the A_{1120}/A_{1290} ratio was measured for the IR spectrum of the spray-dried ASD prepared from the same solution. The flavor retention was then calculated as the ratio of A_{1120}/A_{1290} values before and after spray-drying. The flavor retention determined via IR spectroscopy was approximately $52 \pm 4\%$ ($N = 3$), which closely aligns with the value of $51 \pm 3\%$ obtained using the previously described UV absorption method (results presented subsequently). These results corroborate the validity of the UV absorption technique described above.

2.7. Prediction of overlap concentrations of PVP in alcohol

It is widely recognized that the condition of the solution undergoing drying, particularly the interactions among solute molecules, affects the physicochemical characteristics of the resultant dried material. Accordingly, the concentration of polymer solute, PVP, at which PVP molecules start to come into contact and overlap—referred to as the chain overlap concentration (c^*)—were determined by utilizing the previously reported data as described below. First, the c^* can be estimated as the inverse of the intrinsic viscosity $[\eta]$ (Colby, 2010; Teraoka, 2002).

$$c^* \approx 1/[\eta] \quad (1)$$

The relationship between the hydrodynamic parameter $[\eta]$ and the molecular weight of a polymer (M_w) is described by the following Mark-Houwink-Sakurada equation (Paul & Timothy, 2007; Rebelo et al., 2017; Wagner, 1985).

$$[\eta] = K_p \cdot M_w^a \quad (2)$$

The constants K_p and a are parameters that depend on the type of polymer and the solvent (Paul & Timothy, 2007; Rebelo et al., 2017; Wagner, 1985). Consequently, the values of K_p and a in Eq. (2) were determined based on the reported $[\eta]$ values for PVP (Guettari et al., 2010; Morioka et al., 2016; Othman et al., 2023). Subsequently, the c^* values for PVP with different molecular weights in both methanol and ethanol were estimated.

As mentioned above, the estimation of the c^* was based on a limited number of reported data sets for viscosity analyses involving PVP preparations different from those used in this study. Therefore, it should be noted that the estimated c^* values may differ slightly from the actual values, although they offer a reasonable approximation of their ranges.

2.8. Statistical analysis

Statistical analyses of the measured flavor retention rates (%) were performed. The p -values comparing the flavor retention values between two experimental conditions were calculated using the t -test, implemented via the T.TEST function in Microsoft Excel 365 (Microsoft Co., Ltd., Redmond, WA) (Tables 1, Fig. 2, Fig. 3, Table S1 and Table S2). A p -value less than 0.05 was considered indicative of statistical significance. On the other hand, to evaluate the flavor encapsulation capacity of the carrier PVP, the parameter fitting of the experimental data was conducted using the Langmuir adsorption model, as described below. The determination of R values for the Langmuir model fitting was performed

Table 1

Particle size distributions of spray-dried PVP-based ASD samples. The ratio of cinnamaldehyde to PVP in the mixed solution before spray-drying was 10:1. The *p*-values comparing particle sizes across different PVP concentrations were calculated using a *t*-test performed with the T.TEST function in Microsoft Excel.

PVP Mw (kDa)	solvent	conc. (g/L)	Encapsulated amount (mmol/g-PVP)	particle diameter (μm)				
				Mean	Dn10	Dn50	Dn90	
10	MeOH	100	0.78±0.07	3.2±1.7	1.4	2.9	5.3	
	EtOH	100	0.76±0.04	2.1±1.2	1.0	1.8	3.7	
24.5	MeOH	25	0.52±0.04	1.7±0.7	} <i>p</i> <0.05	0.9	1.6	2.5
		100	0.69±0.11	2.9±1.5		} <i>p</i> >0.1	1.4	2.5
		200	0.74±0.04	2.6±1.8	1.1		2.0	4.9
	EtOH	25	0.40±0.07	1.7±0.7	} <i>p</i> <0.05	0.9	1.6	2.5
		100	0.45±0.01	2.3±1.6		} <i>p</i> =0.05	1.0	1.7
		200	0.71±0.11	2.5±2.1	1.0		1.8	4.3
55	MeOH	100	0.73±0.10	2.9±1.9	1.0	2.4	5.6	
	EtOH	100	0.71±0.04	2.5±1.9	1.0	1.9	5.1	
360	MeOH	100	0.62±0.02	0.46±0.30*	0.21*	0.40*	0.87*	

*Diameter of the spray-dried ASD fiber.

using KaleidaGraph version 5.04 (HULINKS Inc., Tokyo, Japan) software (Table S3).

3. Results

3.1. Flavor retention characteristics during spray-drying and storage

Fig. S4 presents SEM images of representative spray-dried samples consisting of PVP and the residual flavoring agent. Generally, spray-drying produced spherical particles; however, PVP with a molecular weight of 360 kDa predominantly formed fibrous structures. Our previous research demonstrated that fibrous materials are generated during

spray-drying with a two-fluid nozzle when the viscosity of the solute solution exceeds a specific threshold (Araga et al., 2025). The ASD samples spray-dried with 100 g/L of PVP (360 kDa) exhibited smooth and continuous one-dimensional structures (Fig. S4) measuring more than 10² mm in length, which precluded the determination of their aspect ratio using SEM observation.

The surface structure appeared smooth under the condition of approximately 40 nm-thick Pt coating, as shown in Fig. S4(e). The fibrous morphology observed in spray-dried PVP may lead to mechanistically distinct encapsulation behavior compared to the conventional particulate form. Additionally, solutions of 360 kDa PVP at concentrations above 100 g/L could not be spray-dried because of persistent clogging at the tip of the two-fluid nozzle in the spray dryer. Therefore, in the current study, the ASD sample containing 360 kDa PVP was investigated under a limited set of conditions.

The ratios of encapsulated flavor to the initially loaded amount, referred to as the flavor retention rate (%), are presented in relation to the initial flavor content relative to the PVP content (Fig. 1(a–d)). Fig. 1(a–d) demonstrate that, regardless of the molecular weight of PVP and the type of solvent used (methanol or ethanol), the flavor retention rate generally increases with higher concentrations of the carrier PVP. However, as the initial flavor concentration increases, the flavor retention rate decreases, with the slope of this decline appearing less steep for larger PVP molecular sizes (Fig. 1(a–d)). This observation is supported by the smaller *p*-values observed at lower ranges of initial flavor concentration changes (Table S1). A comparison between methanol and ethanol reveals no significant differences in flavor retention rate values (Fig. 1(a–d)), as evidenced by the non-significant statistical differences reported in Table S2 and depicted in Fig. 1(a–d).

In order to conduct a comprehensive analysis of the characteristics of PVP as a carrier for the encapsulation of volatile flavors, the flavor retention rate was converted into the absolute quantity of flavor retained in the spray-dried samples, as illustrated in Fig. 1(A–D). Fig. 1(A–D) also depict theoretical values representing scenarios in which the loaded flavor is completely retained within the spray-dried sample, provided for comparative purposes.

As illustrated in Fig. 1(A–D), all combinations of molecular weight of PVP, PVP concentration, and solvent type (methanol and ethanol)

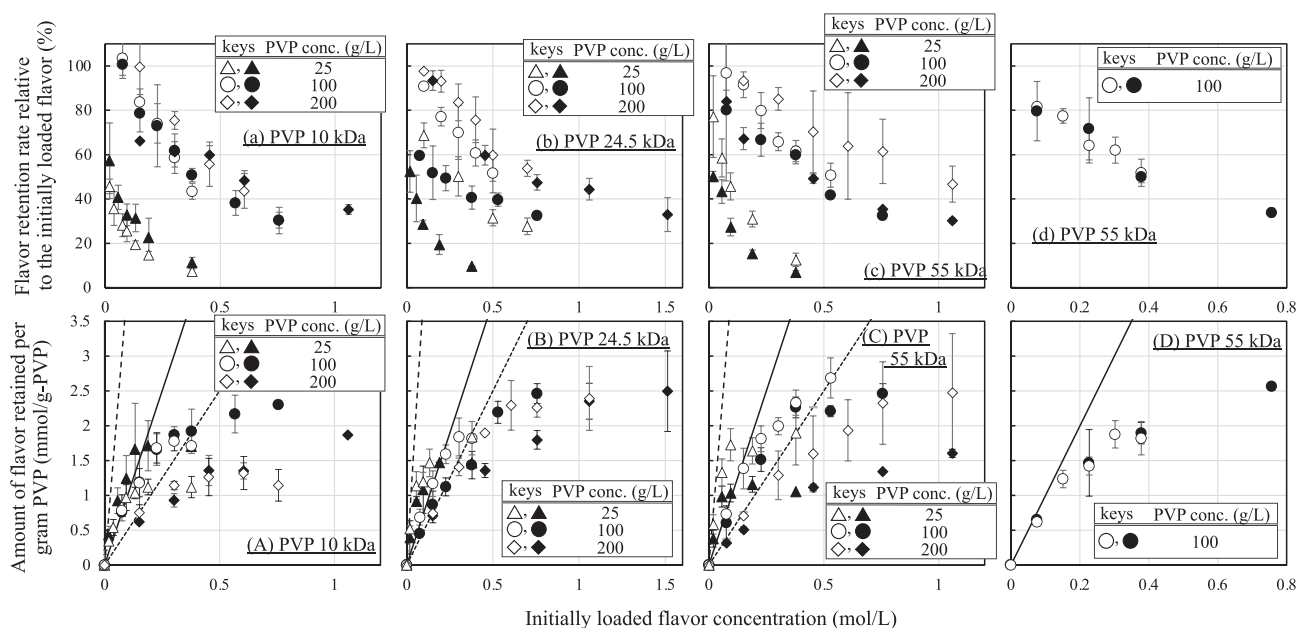


Fig. 1. Retention rate (%) of cinnamaldehyde within spray-dried ASD samples (a–d, upper row) and the amounts of cinnamaldehyde retained per gram of PVP (mmol/g-PVP) in these samples (A–D, bottom row), plotted as a function of the initial cinnamaldehyde concentration. Mean molecular weights of PVP were 10 kDa (a and A), 24.5 kDa (b and B), 55 kDa (c and C), and 360 kDa (d and D). Methanol (open keys) and ethanol (closed keys) served as solvents. The PVP concentration used in the spray-drying was set to 25, 100, and 200 g/L.

demonstrate a similar curve shape regarding flavor retention as a function of initial flavor concentration. Specifically, as the initial flavor concentration increases, the flavor retention amount significantly rises and subsequently approaches a maximum value.

The flavor retention curves shown in Fig. 1(A–D) demonstrate that the relationship between flavor retention and the initial flavor concentration prior to spray-drying is characterized by an initial increase, followed by a maximum retention value, as observed in the curves. To effectively characterize and compare the relationships between retention amount and initial flavor concentration, the maximum retention value (q_m , mmol/g-PVP) was determined (Fig. 2(a)), along with the magnitude of the initial increase in retention relative to q_m (K , L/mmol (Fig. 2(b)), as derived from Fig. 1(A–D). In cases where flavor retention did not reach its maximum within the tested concentration range, the well-established Langmuir equation (Eq. (3)) was applied to extrapolate the maximum values attained.

$$\text{Flavor encapsulation amount (mmol/g-PVP)} = q_m K C / (1 + K C) \quad (3)$$

where C represents the concentration of loaded cinnamaldehyde (mmol/L) before spray-drying, and q_m and K are as defined above. Judging from the R values listed in Table S3, the Langmuir model fitting to the flavor retention curves in Fig. 1(A–D) appear to be reasonable. Based on the R values presented in Table S3, the Langmuir model provides an appropriate fit to the flavor retention curves depicted in Fig. 1(A–D).

Fig. 2(a) shows that, across different average molecular weights of PVP and different solvent types, the q_m values at a PVP concentration of 100 g/L are consistently higher than those at concentrations of 25 g/L and 200 g/L, as indicated by the p -values. Regarding the influence of mean molecular weight of PVP on q_m , no significant differences are observed at a concentration of 25 g/L, regardless of whether methanol or ethanol is used as the solvent. Conversely, at PVP concentrations of 100 and 200 g/L, the q_m values increase with the molecular weight of PVP, ranging from 10 kDa to 55 kDa when methanol is employed. On the other hand, when ethanol is used as the solvent at PVP concentrations of 100 or 200 g/L, the PVP with a molecular weight of 24.5 kDa exhibits the highest q_m value among the three molecular weights tested (Fig. 2(a)). Consequently, the maximum q_m value reaches approximately 4 mmol/g-PVP at a concentration of 100 g/L, specifically for the combinations of methanol with PVP 55 kDa and ethanol with PVP 24.5 kDa (Fig. 2(a)).

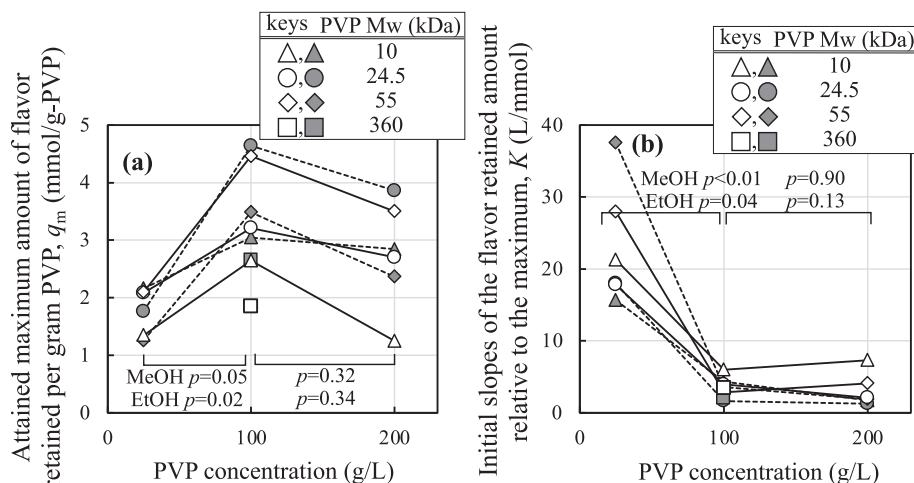


Fig. 2. (a) Attained maximum amount of flavor retained per gram PVP, q_m (mmol/g-PVP) and (b) initial slopes of the flavor retained amount relative to q_m , K (L/mmol) of the relationships between the amount of retained cinnamaldehyde per gram of PVP and cinnamaldehyde concentration for different PVP molecular weights and solvent types, as shown in Fig. 1(A–D), plotted as a function of PVP concentration. Methanol (open keys, solid lines) or ethanol (closed keys, dashed lines) served as the solvent. The p -values comparing the values at 100 g/L and 25 g/L concentrations of PVP, as well as those comparing the values at 100 g/L and 200 g/L concentrations of PVP for methanol and ethanol, respectively, were determined using a t -test performed with the T.TEST function in Microsoft Excel.

Fig. 2(b) illustrates the K values corresponding to various molecular weights of PVP and different solvent types as a function of PVP concentration. As shown in Fig. 2(b), the K values at a PVP concentration of 25 g/L are significantly higher than those observed at higher concentrations of 100 g/L and 200 g/L ($p < 0.05$), which consistently remain below 8 L/mmol. When comparing K values across different PVP molecular weights, the 55 kDa PVP exhibits the highest K values at 25 g/L in both methanol and ethanol solvents (Fig. 2(b)).

The temporal profiles of the amount of remaining cinnamaldehyde in representative spray-dried ASD samples stored in a vacuum desiccator over silica gel at 30 °C were evaluated (Fig. 3). As shown in Fig. 3, a substantial loss of cinnamaldehyde from the ASD samples typically occurred within the first 30 days of storage (0–30 days, $p < 0.05$), after which further loss was minimal. The residual cinnamaldehyde content in the ASD samples at the end of this initial flavor loss period (around 30 days of storage) was approximately 1 mmol/g-PVP when the initial cinnamaldehyde concentration exceeded 0.076 mol/L (with PVP concentration at 100 g/L). Conversely, when the initial cinnamaldehyde concentration was below 0.076 mol/L, negligible flavor loss was observed throughout the tested storage period (0–100 days, $p > 0.05$).

3.2. Characterization of spray-dried ASD samples

Table 1 presents a summary of the particle sizes of representative spray-dried ASD samples. The particle diameters of spray-dried PVP with molecular weights of 10, 24.5, and 55 kDa were found to be within similar ranges, irrespective of the solvent used (methanol or ethanol). Regarding the effect of PVP concentration during spray drying, samples prepared with 100 and 200 g/L PVP exhibited similar particle size distributions, which were marginally larger than those of samples prepared with 25 g/L PVP (Table 1, $p < 0.05$).

A representative spray-dried ASD sample, along with a pure PVP sample, was subjected to vacuum drying at 30 °C for 20 days, followed by DSC analysis. The DSC thermogram of the spray-dried ASD containing cinnamaldehyde exhibited a single heat capacity shift corresponding to the glass-to-rubber transition (Fig. S2), indicating a homogeneous distribution of cinnamaldehyde without detectable phase segregation (Satoh et al., 2016). The temperatures at which the heat capacity changes commenced in the thermograms were identified as the T_g and are plotted as a function of the residual cinnamaldehyde amount in the ASD samples in Fig. 4. While the T_g of the pure PVP samples slightly increased with higher PVP molecular weights, the T_g values of the spray-

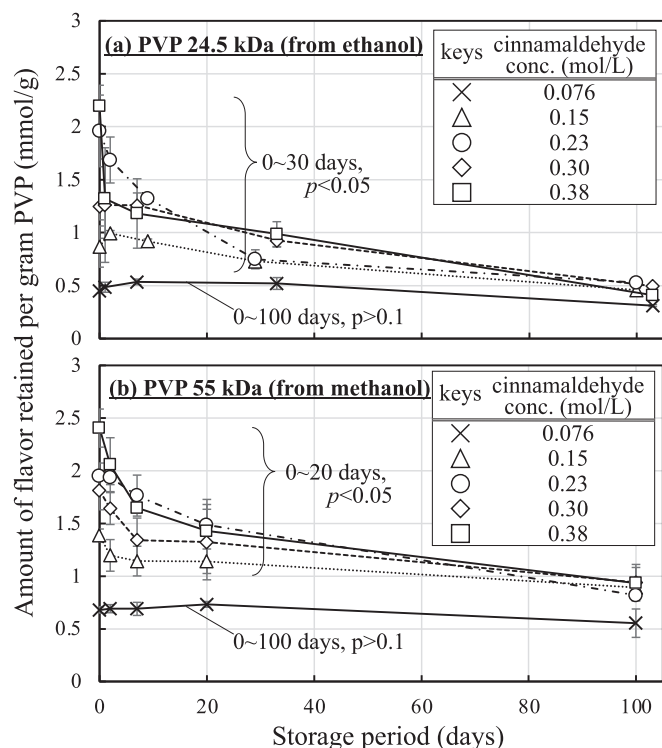


Fig. 3. Dissipation profiles of encapsulated cinnamaldehyde from spray-dried ASDs stored under vacuum over silica gel at 30 °C. The ASD samples analyzed were prepared from an ethanol solution containing 100 g/L of PVP with a molecular weight of 24.5 kDa (a) and from a methanol solution containing 100 g/L of PVP with a molecular weight of 55 kDa (b). These ASD samples exhibited the highest cinnamaldehyde encapsulation capacities, corresponding to one gram of PVP (q_m) for the ethanol and methanol solvent systems, respectively. The p -values between 0–30 days and 0–100 days were computed using a t -test performed with the T.TEST function in Microsoft Excel.

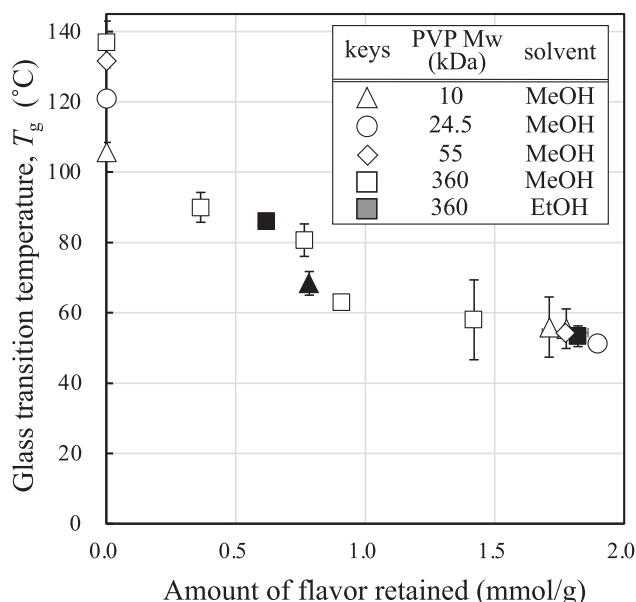


Fig. 4. Glass transition temperatures (T_g) of spray-dried ASDs as a function of the quantity of cinnamaldehyde encapsulated per gram of PVP. The ASDs were stored under vacuum at 30 °C over silica gel for 20 days to remove residual solvent prior to the determination of their T_g values.

dried ASD decreased with increasing amounts of residual

cinnamaldehyde, following a consistent single curve. This finding clearly demonstrates that cinnamaldehyde acts as a plasticizer for the dried PVP matrix within the ASD, consistent with observations reported in our previous study (Sato et al., 2016).

Fig. 5 presents the infrared (IR) spectra of spray-dried PVP with a molecular weight of 24.5 kDa, pure cinnamaldehyde paste, and an ASD prepared by spray-drying an ethanol solution containing 100 g/L of PVP and 100 g/L of cinnamaldehyde, subsequently stored for 20 days. The IR spectrum of the ASD closely resembles that of the spray-dried PVP alone, while also exhibiting a characteristic cinnamaldehyde absorption band near 1120 cm^{-1} due to aromatic C–H bending vibration.

Table 2 summarizes the values of c^* (g/L) for PVP of varying molecular weights and concentrations after spray-drying. As anticipated, the c^* values decrease with increasing molecular weight of PVP. On the other hand, the c^* values for PVP in ethanol are approximately 17 to 40% lower than those observed in methanol, implying that PVP adopts a more expanded conformation in ethanol compared to methanol.

4. Discussion

The ASD samples prepared using a 25 g/L PVP solution exhibit significantly smaller particle sizes compared to those prepared with 100 and 200 g/L PVP solutions, as shown in Table 1. This phenomenon may occur because PVP molecules within droplets, when present at concentrations below a certain threshold, are unable to form a continuous matrix during the drying process, resulting in shrinkage.

As illustrated in Fig. 1(A–D), when the initial flavor concentration is below 0.1 mol/L and the PVP concentration is equal to or exceeds 100 g/L, all combinations of solvent type and PVP molecular size demonstrate flavor retention amounts that align with the theoretical values indicative

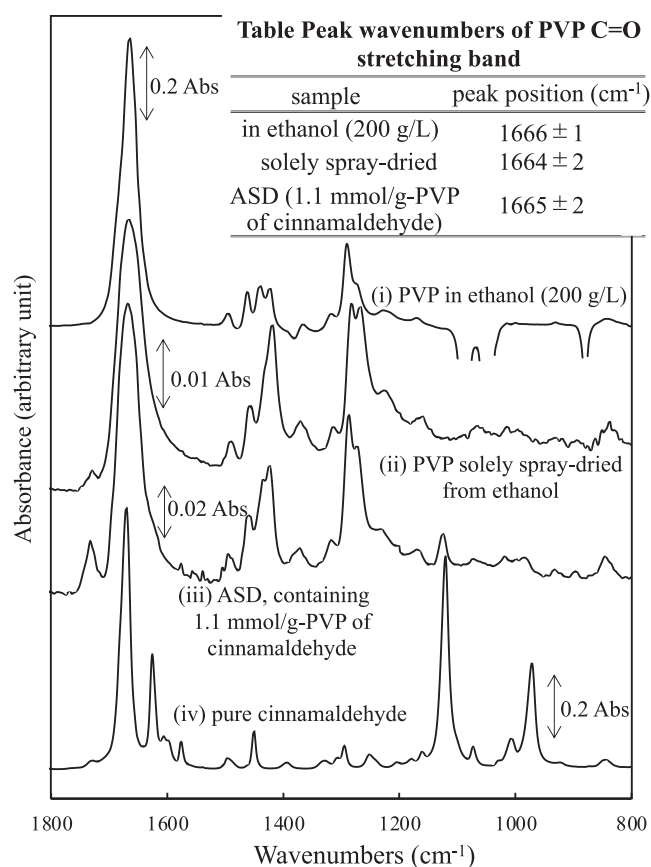


Fig. 5. IR spectra of (i) ethanol containing 200 g/L PVP, (ii) spray-dried PVP alone, (iii) an ASD containing 1.1 mmol/g-PVP of residual cinnamaldehyde, and (iv) pure cinnamaldehyde. The molecular weight of PVP was 24.5 kDa.

Table 2

Estimated overlap concentrations (g/L) of different molecular weight of PVP in methanol and ethanol.

Solvent	Molecular weight of PVP		
	10 kDa	24.5 kDa	55 kDa
Methanol	168	75	36
Ethanol	100	53	30

* K_p for methanol and ethanol were determined to be 1.36×10^{-3} and 1.44×10^{-3} , respectively. α was 0.91 for methanol and 0.71 for ethanol.

of complete flavor retention, suggesting full flavor retention.

It has been reported that the carbonyl groups of PVP play a significant role in maintaining the integrity of the dried structure (Taylor & Zograf, 1998). It is important to highlight that, in the IR spectrum of the ASD (Fig. 5), the absorption band associated with the stretching vibration of the PVP carbonyl group appears at the same wavenumber as that observed in pure spray-dried PVP. This finding persists despite the incorporation of approximately 1.5 mmol of cinnamaldehyde per gram of PVP within the matrix, suggesting the absence of specific interactions between the carbonyl groups of PVP and cinnamaldehyde. Consequently, flavor retention in spray-dried ASDs is primarily achieved through the physical entrapment of flavor molecules within distinct regions of the PVP matrix, rather than through specific chemical interactions that bind the flavor molecules.

The aromatic C—H bending vibration band observed at approximately 1120 cm^{-1} , characteristic of cinnamaldehyde (Fig. S3), appears broadened and negatively shifted in the ethanol solution compared to the pure cinnamaldehyde paste. This effect is likely due to the solvation of cinnamaldehyde molecules by ethanol, which imposes greater and more heterogeneous constraints on the aromatic C—H bending mode. In contrast, relative to the solution sample, the C—H bending band becomes sharper and shifts to a lower frequency upon spray-drying (Fig. S3). These observations suggest that the aromatic group of cinnamaldehyde experiences less restriction and greater homogeneity in the encapsulated state than in the solvated state. The observed decrease in restriction and enhanced homogeneity of cinnamaldehyde within the ASD sample appear to correspond with the previously noted absence of specific interactions with PVP (Fig. 5) and the uniform distribution throughout the ASD carrier matrix (Fig. S2), respectively.

As shown in Table 2, the c^* of PVP in methanol and ethanol were estimated using data collected at 25°C . However, it is important to note that during spray-drying, freshly atomized solution droplets are rapidly heated to temperatures near their boiling points immediately after spraying. A previous study (Aferni et al., 2016) reported that increasing temperature reduces the c^* for aqueous PVP solutions, with a decrease of approximately 25% observed when the temperature rises from 25°C to 60°C . Therefore, the actual c^* values for the freshly sprayed droplets are expected to be significantly lower than the estimated values presented in Table 2. Considering this, the data suggest that PVP molecules with molecular weights ranging from 10 kDa to 55 kDa predominantly exist as isolated entities in methanol and ethanol solutions at a concentration of 25 g/L, as indicated by c^* values well above this concentration. At a higher concentration of 100 g/L, molecular overlap is generally expected, including for 10 kDa PVP in ethanol, where the estimated c^* value (Table 2) is slightly below 100 g/L.

Our previous study (Nitta et al., 2024) analyzed the loss of flavor from a methanol solution containing carrier agents (PVP and α -maltose) during vacuum foam drying. The findings indicated that the initial stage of drying resulted in a substantial loss of flavor (Nitta et al., 2024). Therefore, flavor retention by ASD will be discussed herein, with emphasis on the initial phase of the spray-drying process (Fig. 6).

Initially, consider the scenario in which the concentration of PVP is below its c^* (left row in Fig. 6). Under these conditions, PVP molecules are considered to exist independently within the solvent, without interpenetration. Consequently, during the spray-drying process, the molecular cavities present within individual PVP molecules would entrap the flavor molecules. The number of such molecular cavities available for flavor entrapment, referred to as “entrapment pockets,” per unit mass of PVP is assumed to correspond to the maximum amount of flavor retained, denoted as q_m . This maximum retention capacity appears to be independent of the molecular weight of PVP, as indicated by the consistent range of q_m values observed (25 g/L, Fig. 2(a)).

On the other hand, it can be hypothesized that the K value increases with improved accessibility of flavor molecules to the entrapment pocket within PVP molecules, as well as with the increased probability of flavor molecules encountering these pockets during the drying process (left row in Fig. 6). Based on this hypothesis, it is expected that a PVP concentration slightly below, but close to, the c^* would result in the highest K value. Accordingly, at a PVP concentration of 25 g/L, the K

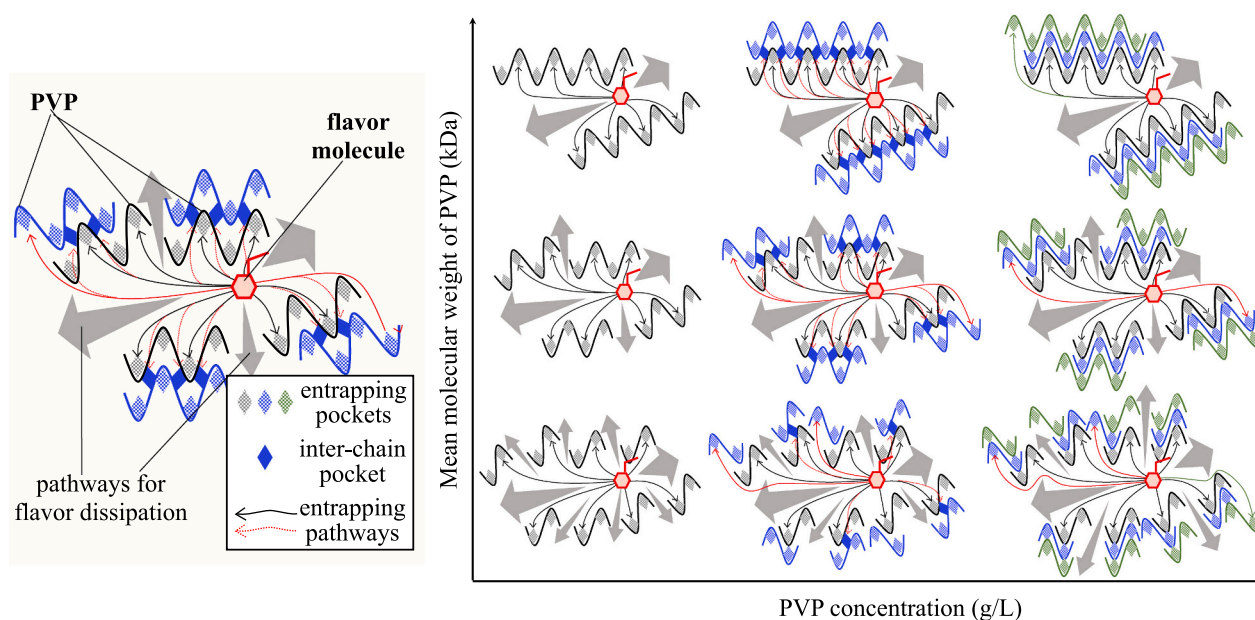


Fig. 6. Potential mechanisms by which PVP entrains flavor molecules (cinnamaldehyde) during the spray-drying process, alongside the associated flavor loss. These processes are depicted as functions of PVP molecular weight (vertical axis) and PVP concentration (horizontal axis).

value—particularly for the 55 kDa PVP ($c^* \approx 30\text{--}36\text{ g/L}$, Table 2)—may exceed that observed at higher PVP concentrations, as shown in Fig. 2(b).

Next, as mentioned above, increasing the PVP concentration from 25 g/L to 100 g/L is expected to result in significant intermolecular overlap among PVP molecules within the solution containing flavor molecules (center row in Fig. 6). Such overlaps may facilitate the formation of additional flavor entrapment sites, referred to as interchain pockets, in addition to the intrinsic entrapment pockets within individual PVP molecules. Consequently, the q_m for PVP is expected to increase due to the presence of these interchain pockets as the concentration rises from 25 g/L to 100 g/L, as illustrated in Fig. 2(a). However, these intermolecular overlaps are also anticipated to reduce the accessibility of flavor molecules to both the interchain and intrinsic entrapment pockets (center row in Fig. 6). This effect would result in lower K values at a PVP concentration of 100 g/L compared to those observed at 25 g/L, as shown in Fig. 2(b).

Finally, an increase in the PVP concentration from 100 g/L to 200 g/L may induce excessive intermolecular overlap and entanglement (right row in Fig. 6). This effect can cause overpacking of the PVP chains, subsequently reducing the interchain pockets and thus decreasing the q_m value (Fig. 2(a)).

The reduction of interchain pockets due to excessive overlap and subsequent overpacking of PVP is anticipated to occur at a PVP concentration of 100 g/L when the molecular size of PVP surpasses a certain threshold (from bottom to top, Fig. 6). This anticipated phenomenon may account for the observed decrease in the q_m value at 100 g/L PVP concentration, particularly when the molecular weight of PVP exceeds 55 kDa in methanol and 24.5 kDa in ethanol. Furthermore, the observation that the optimal PVP molecular weights for achieving the maximum q_m at 100 g/L are lower in ethanol than in methanol can be attributed to ethanol's lower c^* (Table 2), which leads to more intermolecular overlap compared to methanol.

The temporal profiles of the remaining cinnamaldehyde in the spray-dried ASD samples (Fig. 3) seems to suggest the existence of two distinct retention modes within the ASD. The first is an apparently irreversible retention mode, in which cinnamaldehyde molecules are predominantly entrapped and remain stable during vacuum storage when their initial cinnamaldehyde concentration is below approximately 0.076 mol/L. The second is a reversible retention mode, which becomes prominent at higher initial cinnamaldehyde concentrations, allowing cinnamaldehyde molecules to escape from the ASD during the early stages of vacuum storage.

According to previous reports on flavor inclusion using CDs (Yildiz et al., 2019), the encapsulation capacity of CDs for cinnamaldehyde molecules—corresponding to the q_m value in this study—is approximately 1.5 to 1.8 mmol per gram of CDs. This capacity equates to the inclusion of two cinnamaldehyde molecules within a single β - or γ -CD molecule, which appears plausible based on the cavity volumes of the CD molecules. Starch (Gao et al., 2023) and rice bran (Liu et al., 2023) have also been investigated as adsorption-based flavor carriers, demonstrating cinnamaldehyde encapsulation capacities of 0.1–0.5 mmol/g and approximately 0.3 mmol/g, respectively. On the other hand, as shown in Fig. 2(a), one gram of the PVP carrier matrix is capable of encapsulating up to 4 mmol of cinnamaldehyde. The temporal profiles of cinnamaldehyde retention (Fig. 3) suggest the presence of an apparently irreversible mode of cinnamaldehyde encapsulation in the ASD, with an amount of approximately 1 mmol per gram of PVP. According to these findings, PVP is inferred to have a high capacity for molecular encapsulation of flavor during drying processes, comparable to other reported carriers. However, it is essential to verify the potential effectiveness of PVP as an alternative flavor encapsulating carrier by considering additional factors, such as stability against chemical degradation and hygroscopicity. The maximum q_m value ($\sim 4\text{ mmol/g-PVP}$) and the apparently irreversible encapsulation amount of cinnamaldehyde ($\sim 1\text{ mmol/g-PVP}$) imply that each cinnamaldehyde

molecule within the ASD are associated with approximately two or eight PVP monomer units, respectively. This interpretation of the flavor encapsulation capacity of PVP seems plausible, considering the relative molecular sizes of the PVP monomer unit and the cinnamaldehyde molecule.

PVP has been suggested to form coil structures capable of encapsulating substances with molecular weights of a few hundred (Zelkó & Süvegh, 2005). These coil structures might possibly serve as flavor-retentive pockets, contributing to the flavor encapsulation capacity of PVP. However, further investigation is necessary to determine whether PVP forms such structures in alcohols and whether these structures are maintained following the spray-drying process.

This study employed an under-vacuum condition for storing the ASD samples to exclusively evaluate the encapsulation ability of spray-dried PVP during storage, thereby eliminating other environmental factors. However, flavor powder deterioration can also result from water vapor sorption from atmospheric air, the associated plasticization of the ASD structure, and the oxidation of unsaturated bonds in the flavor molecules. Therefore, to comprehensively assess the effectiveness of the ASD-based flavor encapsulation technique, future studies will evaluate storage stability under various conditions, including different relative humidities, oxygen presence, and temperatures.

5. Conclusion

The amorphous solid dispersion (ASD), composed exclusively of polyvinylpyrrolidone (PVP) and the volatile flavor compound cinnamaldehyde, was prepared via spray-drying under various solution conditions. These conditions included variations in the concentrations of PVP and cinnamaldehyde, differences in PVP molecular weights, and the use of different solvents (methanol or ethanol). Subsequently, the flavor retention properties of the resulting ASD samples were evaluated. It was found that the rate of flavor retention, relative to the initially loaded cinnamaldehyde, increased as the cinnamaldehyde-to-PVP ratio decreased, consistent with expectations. In contrast, the capacity of PVP to retain cinnamaldehyde during spray-drying reached a maximum of approximately 4 mmol/g-PVP, corresponding to a ratio of one encapsulated cinnamaldehyde molecule per approximately two PVP monomer units. Based on the supplementary IR spectroscopic and thermal analyses of the ASD samples, the flavor encapsulation mechanism was inferred to involve the noninteractive entrapment of flavor molecules within the inter- and intramolecular “pockets” of the carrier PVP during the drying process. The number of the “entrapment pockets” is hypothesized to reach a maximum at PVP concentrations slightly above its overlap concentration. Furthermore, the dissipation of cinnamaldehyde from the ASD samples during storage under vacuum was examined, indicating an apparently irreversible flavor encapsulation mechanism.

CRediT authorship contribution statement

Haruna Sato: Writing – original draft, Validation, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Naohonda:** Visualization, Validation, Resources, Investigation, Data curation. **Takato Hasegawa:** Validation, Software, Investigation, Data curation. **Chika Araga:** Supervision, Methodology, Data curation, Conceptualization. **Hiroyuki Imanaka:** Validation, Supervision, Investigation, Conceptualization. **Naoyuki Ishida:** Writing – review & editing, Supervision, Project administration, Methodology, Investigation, Conceptualization. **Koreyoshi Imamura:** Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Conceptualization.

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Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.foodchem.2026.150819>.

Data availability

Data will be made available on request.

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