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Research Article

Effects of partial dehydration methods on high-moisture forages chemical composition

Nicole S.A. Lima [a], Daiana F. Quirino [a], Marcia O. Franco [b], Edenio Detmann [a,*]

[a] Department of Animal Science, Universidade Federal de Viçosa, Viçosa, Brazil
[b] Natural Resources Institute Finland (Luke), Jokioinen, Finland

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ABSTRACT

This study evaluated whether partial dehydration methods alter the chemical composition of high-moisture forages. Two experiments were conducted. In Experiment 1, 20 maize silage samples were subjected to freeze-drying, forced-air oven drying, forced-air oven drying preceded by a microwave preheating step, or microwave drying. In Experiment 2, 28 forages (silages, fresh grasses, and fresh legumes) were evaluated using freeze-drying, forced-air oven drying, microwave drying, or air-fryer drying. Method effects were assessed based on water removal efficiency, nitrogenous and fibrous compound profiles, in vitro digestibility, indigestible fibre, and colorimetric properties. Microwave and air-fryer drying removed less water than freeze-drying and resulted in greater differences in nitrogenous compound concentrations and profiles, including apparent N volatilisation and greater concentrations of detergent-insoluble nitrogen. Heat application inconsistently affected fibrous components but consistently reduced in vitro dry matter digestibility without altering in vitro fibre digestibility, likely reflecting starch retrogradation and production of heat-derived artefacts soluble in neutral detergent. These modifications were most pronounced in fresh forages, indicating an interaction with their chemical matrix. Among heat-based methods, forced-air oven drying produced the least chemical alteration. Overall, heat application during partial dehydration modifies the chemical characteristics of high-moisture forages, and when freeze-drying is not feasible, forced-air oven drying best preserves the original sample composition.

1. Introduction

Forages constitute the main component of ruminant diets in tropical production systems (Silvestre and Millen, 2021; Associação Brasileira das Indústrias Exportadoras de Carne – ABIEC., 2024). Optimizing forage utilisation requires accurate characterisation of its chemical composition, which is the primary determinant of its potential and effectiveness in supplying energy and nutrients for animal product synthesis. However, the physical characteristics of fresh forages and silages preclude their direct evaluation by most chemical

Abbreviations: ADF, acid detergent fibre; ADFp, acid detergent fibre expressed exclusive of protein; ADIN, acid detergent insoluble nitrogen; ADL, acid detergent lignin; DM, dry matter; IN, insoluble nitrogen; iNDF, indigestible neutral detergent fibre; IVDMD, in vitro dry matter digestibility; IVNDFD, in vitro neutral detergent fibre digestibility; KL, Klason lignin; N, nitrogen; NDF, neutral detergent fibre; NDFp, neutral detergent fibre expressed exclusive of protein; NDIN, neutral detergent insoluble nitrogen; nNDIN, insoluble nitrogen not bound to the cell wall; NPN, non-protein nitrogen; PN, protein nitrogen; SN, soluble nitrogen; SPN, soluble protein nitrogen.

* Correspondence to: Departmento de Zootecnia, Universidade Federal de Viçosa, Viçosa, Minas Gerais 36570-900, Brazil.
E-mail address: detmann@ufv.br (E. Detmann).

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analytical procedures. Therefore, prior physical processing is required to convert the laboratory sample into an analytical sample suitable for chemical analysis.

Partial dehydration of laboratory samples from high-moisture forages is essential to facilitate handling, storage, and mechanical processing procedures (e.g., grinding) (Undersander et al., 1993; Pelletier et al., 2010; Association of American Feed Control Officials – AAFCO., 2018). Ideally, alterations resulting from physical processing should be restricted to the physical domain, thereby preserving the chemical integrity of the original material collected from the decision unit (Association of American Feed Control Officials – AAFCO., 2018; Detmann et al., 2025).

Partial dehydration can be achieved through cold or heat-based processes, each with distinct advantages and limitations. Freeze-drying is generally regarded as the method that yields analytical samples most similar to the original material, although complete elimination of volatilisation losses cannot be guaranteed (Smith, 1981; Morris et al., 2019, Evan et al., 2025). However, the high operational costs associated with specialized infrastructure and equipment limit its widespread adoption.

Heat-based methods are more accessible; however, in addition to volatilisation losses, heat application promotes protein denaturation and the formation of chemical artefacts. These alterations are intensified at elevated temperatures (i.e., >60°C). Artefacts generated by non-enzymatic heat-mediated reactions are predominantly quantified as components of the fibrous fraction, thereby modifying the chemical profile of the analytical samples and increasing their divergence from the characteristics of the original material (Van Soest, 1994; Eskin et al., 2013).

The most widely adopted heat-based methods for partial dehydration involve the use of forced-air ovens (50–60°C; Detmann et al., 2025), which allow accurate control of physical processing parameters and operate at temperatures considered less prone to protein denaturation and artefact formation. However, a slow heating ramp in forced-air ovens may promote undesirable metabolic processes (e.g., indigenous enzyme activities, protein degradation) and increase the likelihood of non-enzymatic reactions due to prolonged exposure times (Deinum and Maassen, 1994). Therefore, it has been hypothesized that a preheating step using microwave ovens may reduce biases in chemical composition estimates, accelerating the achievement of target drying temperatures (Hofman and Belgium, 1965; Pelletier et al., 2010).

Alternative heat-based methods, such as using microwave ovens and air fryers, offer faster and more cost-effective options compared to conventional laboratory equipment and are widely available. However, these devices offer limited control over the physical conditions, being characterized by irregular heating patterns and difficulties in temperature regulation during dehydration (Brodie et al., 2019), which may result in overheating and, consequently, an increase in undesirable chemical modifications.

Methods characterized by lower cost and greater accessibility are attractive alternatives and, therefore, tend to be adopted more frequently in analytical laboratories. Nevertheless, their appropriate adoption and application require a comprehensive evaluation of their effects on dry matter estimation and analyte integrity. Although several dehydration methods have been described for forage sample preparation, there is still limited scientific evidence comparing their effects on dry matter estimates and chemical composition, particularly for recently adopted alternatives such as domestic air-fryer drying. The increasing use of these practical and low-cost methods in routine laboratory activities has outpaced their scientific validation and standardization.

The use of partial dehydration methods without a solid scientific basis or adequate standardization may introduce systematic biases in estimates of forage chemical composition, potentially compromising forage quality assessment, nutritional evaluation, and the decisions based on these analyses in production systems.

Therefore, our objective was to comprehensively evaluate, through two independent experiments, the effects of different partial dehydration methods on dry matter quantification and on multiple analytical characteristics of high-moisture forages, including chemical composition, nitrogen fractionation, fibre characterization, digestibility, and colorimetric properties. To ensure that the results would be applicable to a wide range of practical situations, the study encompassed diverse forage materials, including silages, fresh grasses, and fresh legumes.

2. Material and methods

Two experiments were conducted at the Animal Nutrition Laboratory of the Department of Animal Science at the Universidade Federal de Viçosa, Viçosa, Minas Gerais, Brazil. The animal care and handling procedures were approved by the Ethics Committee on the Use of Production Animals of the Universidade Federal de Viçosa (protocol number 066/2023).

2.1. Experiment 1

Twenty primary samples of maize silage were collected in the Viçosa region, Minas Gerais, Brazil. Immediately after collection, each primary sample (1 kg) was quartered to obtain four subsamples (250 g) and frozen (-20°C). Each subsample obtained from a given primary sample was subsequently subjected to a distinct partial dehydration method.

2.1.1. Partial dehydration methods

The following partial dehydration methods were evaluated: freeze-drying, forced-air oven drying, microwave oven drying, and forced-air oven drying preceded by an initial microwave heating step.

After thawing at room temperature, all subsamples were placed on pre-weighed trays and arranged in a thin layer (maximum height of 2 cm).

Freeze-drying procedure followed the standard method of the National Institute of Science and Technology in Animal Science (INCT-CA; method G-002/2; Detmann et al., 2025). Subsamples were refrozen in an ultra-freezer (-80°C for 12 h; CL374–89, ColdLab,

Piracicaba, São Paulo, Brazil). After freezing, the subsamples were transferred to a freeze-dryer and processed for 72 h (working temperature −35°C; vacuum −218 μHg; LP510, Liobras, São Carlos, São Paulo, Brazil). Afterward, subsamples were left at room temperature for 40 min and then weighed.

Forced-air oven drying followed the INCT-CA standard method (method G-001/2; Detmann et al., 2025). Subsamples were placed in a forced-air oven (55°C; 171 A, Fabbe Primar, São Paulo, São Paulo, Brazil) for 48 h. After drying, the subsamples were left at room temperature for 40 min and then weighed.

Microwave drying followed the National Forage Testing Association protocol (NFTA; method 2.2.1.2; Undersander et al., 1993). Each subsample was placed in a microwave oven (MEF28, Electrolux, Manaus, Amazonas, Brazil) at maximum power for 3 min, removed, and manually stirred; this 3-min cycle was repeated thrice. If the subsample was still damp to the touch, additional 1-min cycles at 50% power were applied until the material was sufficiently dry to the touch. After this process, the material was left at room temperature for 40 min and then weighed.

The method proposed by NFTA (Undersander et al., 1993) requires the use of a microwave oven with a minimum power of 600 W. Therefore, prior the drying process, the actual power output of the equipment was measured according to the procedures described by Souza et al. (2002). The estimated power output of the microwave oven operating at 100% power was 729 W.

Drying in a forced-air oven preceded by an initial microwave heating step was conducted similarly to the forced-air oven-drying method previously described. However, subsamples were previously heated in a microwave oven at maximum power for 1 min (Pelletier et al., 2010) and then immediately transferred to the forced-air oven (no cooling period was allowed).

Immediately after each heat-based partial dehydration process, the subsample temperature was measured using an infrared thermometer (TD-965, ICEL Manaus, Manaus, Amazonas, Brazil). Measurements were performed assuming an emissivity of 0.9 (wood emissivity), with the thermometer positioned approximately 20 cm from the centre of the material.

2.1.2. Chemical and biological analyses

The amount of water presumably extracted from the subsamples during the partial dehydration processes was estimated based on its mass loss, according to the following equations:

$$WPE = \frac{(NMw - ADSw)}{NM} \times 1000 \quad (1)$$

$$ADS = \frac{ADSw}{NMw} \times 1000 \quad (2)$$

where: WPE = water presumably extracted during the partial dehydration procedure (g/kg as-is), NMw = subsample or natural matter weight (g), ADSw = air-dried sample weight (g), and ADS = concentration of air-dried sample (g/kg as-is).

Immediately after partial dehydration and weighing, all subsamples were ground in a knife mill (TE-680, Tecnal, Piracicaba, São Paulo, Brazil) to pass through a 2-mm screen sieve. Subsequently, subsamples were quartered, and half of each subsample was reground to pass through a 1-mm screen sieve. The material ground through the 2-mm sieve was used for the in situ incubation procedures, whereas all other analyses described below were performed using the material ground through the 1-mm sieve. The ground materials were stored in sealed polyethylene containers and opened only when test portions were removed for chemical and digestibility analysis.

The residual water concentration after the partial dehydration procedures was quantified by loss on drying (105°C for 16 h). Thus, the total water and dry matter (DM) contents were calculated according to the following equations:

$$RW = \frac{(ADSw - ODSw)}{ADSw} \times 1000 \quad (3)$$

$$TW = WPE + \left(\frac{ADS \times RW}{1000}\right) \quad (4)$$

$$DM = 1000 - TW \quad (5)$$

where: RW = residual water concentration in the sample (g/kg of air-dried material), ADSw = air-dried sample weight (g), ODSw = oven-dried sample weight (i.e., after drying at 105°C; g), TW = total water concentration (g/kg as-is); and DM = dry matter concentration (g/kg as-is). The remaining terms were defined previously.

Chemical analyses were organised into two distinct groups: fibrous and nitrogenous compounds. For the first group, the subsamples were analysed for neutral detergent fibre (NDF; using thermostable α-amylase and omitting sodium sulphite; method F-013/2), acid detergent fibre (ADF; sequential procedure; method F-015/2), and Klason lignin (KL; method F-007/3). All fibrous compounds analyses were performed using a reflux system (model MA450/6; Marconi Equipamentos para Laboratórios, Piracicaba, São Paulo, Brazil) and Gooch crucibles according to the procedures described by Detmann et al. (2025).

For nitrogenous compounds evaluations, the subsamples were analysed for nitrogen content (N; method N-001/3), neutral detergent insoluble nitrogen (NDIN; methods F-013/2 and N-004/3), and acid detergent insoluble nitrogen (ADIN; methods F-015/2 and N-005/3), according to methods described by Detmann et al. (2025). Additionally, soluble nitrogen (SN) and insoluble nitrogen (IN) concentrations were evaluated based on N solubility in a borate-phosphate buffer (Licitra et al., 1996). All N quantifications were performed based on Kjeldahl method. The concentration of insoluble nitrogen not bound to the cell wall (nNDIN) was obtained by the

difference between IN and NDIN (Sniffen et al., 1992).

Indigestible NDF (iNDF) was quantified by a 288-h in situ ruminal incubation procedure (Valente et al., 2011). Additionally, in vitro DM digestibility (IVDMD) was evaluated using non-woven textile filter bags (100 g/m^2) in a 48-h incubation procedure conducted in an incubator (NT 720, Novatécnica, Piracicaba, São Paulo, Brazil), as described by Camacho et al. (2022). In vitro NDF digestibility (IVNDFD) was estimated by extracting the incubation residues with neutral-detergent solution using a fibre analyser (model 200, Ankom Technology, Macedon, NY, USA; method F-001/3; Detmann et al., 2025).

2.1.3. Statistical analysis

The data were analysed according to the following model:

$$Y_{ij} = \mu + S_i + M_j + \varepsilon_{ij} \quad (6)$$

where: Y_{ij} = observed response for the primary sample i subjected to partial dehydration method j; μ = general constant; S_i = random effect of the i-th primary maize silage sample, assumed to be independent and identically distributed as N (0, σ^2_S); M_j = fixed effect of the j-th partial dehydration method; and ε_{ij} = random error, assumed to be independent and identically distributed as N (0, σ^2_ε).

When appropriate, means were compared using Tukey's test. All analyses were performed using the GLIMMIX procedure of SAS 9.4 ($\alpha = 0.05$).

2.2. Experiment 2

Twenty-eight primary forage samples were used and organised into three groups: fresh grasses, fresh legumes, and silages. The fresh grasses group (n = 10) comprised primary samples of Urochloa decumbens cv. Basilisk, U. brizantha cv. Marandu, U. humidicola, Megathyrsus maximus cv. BRS Zuri, M. maximus cv. Mombaça, M. maximus cv. Massai, Cynodon sp. Tifton 85, C. dactylon, Cenchrus purpureus, and Saccharum sp. The fresh legumes group (n = 8) comprised primary samples of Arachis pintoi, Cajanus cajan, Leucaena leucocephala, Gliricidia sepium, Lablab purpureus, Pueraria phaseoloides, Stylosanthes guianensis cv. Mineirão, and Neonotonia wightii. The silage group (n = 10) was composed of maize silage (n = 4), sugarcane silage (n = 2), sorghum silage (n = 2), and elephant grass silage (n = 2).

Fresh forage samples were collected by cutting the plants at approximately 15 cm above ground level in plots or pastures in the Viçosa region, Minas Gerais, Brazil. Silage samples were collected from silos in the same region. With the exception of Cenchrus purpureus and Saccharum sp., which were mechanically processed in a disintegrator immediately after collection, the fresh forage samples were sectioned with scissors to reduce particle size to approximately 2 cm. Each primary sample (1 kg) was quartered to obtain four subsamples (250 g) and frozen (-20°C). Each subsample obtained from a given primary sample was subsequently subjected to a distinct partial dehydration method.

2.2.1. Partial dehydration methods

The following partial dehydration methods were evaluated: freeze-drying, forced-air oven during (55°C), microwave oven drying, and air-fryer drying. After thawing at room temperature, all subsamples were placed on pre-weighed trays and arranged in a thin layer (maximum height of 2 cm). Freeze-drying, forced-air oven drying, and microwave oven drying followed the same procedures described for Experiment 1. However, for this experiment, a different microwave oven was used (NN-ST27LWRU, Panasonic, Manaus, Amazonas, Brazil), for which the calibration procedure indicated an effective power output of 635 W.

Air-fryer drying was based on protocols described by Severson (2019) and Wallau and Vendramini (2019). Subsamples were placed

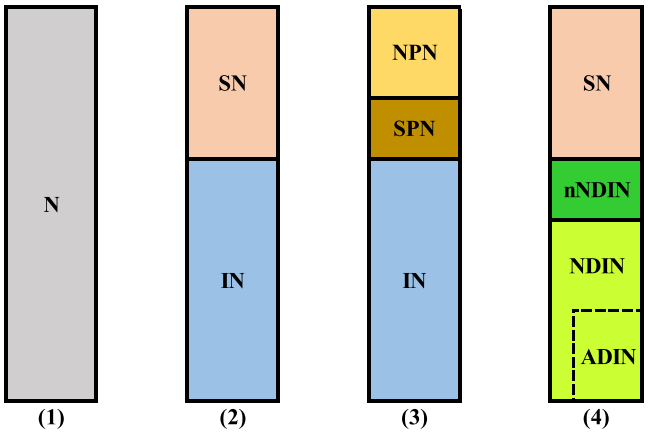

Fig. 1. Schematic representation of the fractionation procedures of nitrogen compounds: (1) N, total nitrogen; (2) solubility-based fractionation: SN, soluble N; IN, insoluble N; (3) soluble fraction: NPN, non-protein N; SPN, soluble protein N; (4) insoluble fraction: nNDIN, insoluble N not bound to the cell wall; NDIN, neutral detergent insoluble N; ADIN, acid detergent insoluble N.

in an air fryer (AF-35-BF, Mondial, China) set at 121°C for 30 min. After 15 min of drying, the subsamples were manually stirred. If the subsamples were still damp to the touch, additional 5-minute drying cycles at 121°C were applied until the material was considered sufficiently dry to the touch. Subsequently, the material was left at room temperature for 40 min and then weighed.

At the end of all partial dehydration procedures, the subsample temperature was measured using an infrared thermometer, as previously described. Sample grinding and handling procedures followed those described for Experiment 1.

2.2.2. Chemical, biological, and physical analyses

The chemical analyses followed the procedures previously described, with three modifications. The residual water concentration after the partial dehydration procedures was evaluated using an updated loss-on-drying method (105°C for 3 h; Quirino et al., 2023). Additionally, KL was replaced by acid detergent lignin (ADL; method F-005/3; Detmann et al., 2025). Nitrogenous compounds were further fractionated into protein nitrogen (PN) and non-protein nitrogen (NPN) by solubilization in trichloroacetic acid (method N-002/3; Detmann et al., 2025). The soluble protein nitrogen (SPN) fraction was calculated as the difference between SN and NPN (Sniffen et al., 1992). A schematic representation of N compound analyses is presented in Fig. 1.

The iNDF analysis was conducted as previously described. However, the IVDMD assessment was performed in individual flasks, instead of using filter bags and an incubator. In this case, the procedures described by Gomes et al. (2011) were applied.

The colour pattern of the material after partial dehydration was evaluated by adapting the procedures described by Quirino et al. (2023) using a colorimeter (4500 L, Hunter Associates Laboratory Inc., Reston, Virginia, United States) and the CIE coordinate space (Commission Internationale de l'Éclairage; L*, a*, and b* coordinates).

After partial dehydration and weighing, each subsample was evenly spread in a white plastic tray (40 × 20 cm) to completely cover the bottom surface. Six readings were then obtained at randomly selected locations on the surface of each subsample. The values obtained from the six readings were averaged for each subsample and used for the statistical analyses. The same procedure was performed before partial dehydration. In this case, the average value across all subsamples from each primary sample was calculated to estimate the colour pattern of the fresh material, thereby enabling valid comparisons between the fresh material and the different partial dehydration methods.

Three different approaches were used. The first approach assessed the lightness coefficient (L*), which ranges from black (i.e., L* = 0) to white (i.e., L* = 100).

The second approach was based on the hue angle (h°), which represents the angular position in the a*–b* plane and is defined as the angle between the hypotenuse and the axis corresponding to the a* coordinate, calculated as follows:

$$h^\circ = \frac{arctg\left(\frac{a^*}{b^*}\right)}{2\pi} \times 360 \; \forall \; a* > 0 \; and b* > 0 \tag{7}$$

$$h^\circ = \left[\frac{arctg\left(\frac{a^*}{b^*}\right)}{2\pi} \times 360\right] + 180 \; \forall \; a* < 0 and \; b* > 0, or \; b* < 0 \tag{8}$$

The third approach consisted of converting the CIE coordinates into the RGB system (red, green, and blue) using the Coloroid Professional Color Plan Designer software. These data were compiled into a Microsoft Excel spreadsheet, in which each cell was filled with the corresponding solid colour generated by RGB values. This procedure was adopted to provide a descriptive visual pattern for the materials evaluated.

2.2.3. Statistical analyses

The data were analysed according to the following model:

$$Y_{ijk} = \mu + G_i + A_{(i)j} + M_k + GM_{ik} + \varepsilon_{ijk} \tag{9}$$

where: Y_{ijk} = response obtained for forage j belonging to group i and processed using dehydration method k; μ = general constant; G_i = fixed effect of the i-th forage group; $A_{(i)j}$ = random effect of the j-th forage nested within the i-th forage group, assumed to be independent and identically distributed as N (0, $\sigma^2_{F/G}$); M_k = fixed effect of the k-th partial dehydration method; GM_{ik} = fixed effect of interaction between forage group and partial dehydration method; and ε_{ij} = random error, assumed to be independent and identically distributed as N (0, σ^2_ε).

Statistical analyses were performed using the GLIMMIX procedure of SAS 9.4 (α = 0.05). When an interaction between forage group and partial dehydration method was detected (P < 0.05), the effect of partial dehydration method within each forage group was examined using the SLICE and SLICEDIFF options of the GLIMMIX procedure. When appropriate, means were compared using Tukey's test.

3. Results

3.1. Experiment 1

Effects of partial dehydration methods were observed for all variables related to water extraction from maize silage samples ($P < 0.01$, Table 1). The amount of water presumably extracted during partial dehydration was greater ($P < 0.05$) for freeze-drying and for forced-air oven drying preceded by a microwave heating step, which did not differ from each other ($P > 0.05$). In contrast, the lowest water extraction values were obtained using microwave drying ($P < 0.05$), whereas forced-air oven drying alone showed intermediate values. Residual water content after partial dehydration exhibited an inverse pattern, with the highest value observed for microwave drying ($P < 0.05$) and the lowest value for freeze-drying ($P < 0.05$).

There was difference among methods ($P < 0.01$) regarding total water and DM concentrations, with microwave drying and forced-air oven drying preceded by a microwave heating step representing the extremes of the multiple-comparison profile. However, the overlapping means did not allow a clear differentiation among methods. This pattens suggests that the amount of water presumably extracted during partial dehydration was, to some extent, compensated for by residual water during the definitive drying at 105°C (Table 1).

The microwave drying method resulted in higher subsample temperatures at the end of the partial dehydration process compared to forced-air oven drying (Table 1).

All variables associated with N compounds were affected by the partial dehydration methods ($P < 0.03$, Table 2). Lower N concentrations were observed when heat-based methods were applied ($P < 0.05$). Among these, methods involving microwave at any stage resulted in the lowest N contents. The lower total N concentrations were reflected in the SN concentration ($P < 0.01$), with heat-based methods reducing SN ($P < 0.05$); the lowest SN values were observed with microwave drying. Microwave drying also increased IN compared with freeze-drying ($P < 0.05$). Part of this increase associated with microwave drying was related to a greater concentration of nNDIN ($P < 0.05$), whereas no differences were observed for NDIN contents ($P > 0.05$). The ADIN concentration was higher for all heat-based methods compared with freeze-drying ($P < 0.05$).

The partial dehydration methods did not differ regarding NDF and NDF expressed exclusive of protein (NDFp, $P \geq 0.20$, Table 3). However, ADF differed among methods ($P < 0.01$), with higher values observed for forced-air oven drying compared to freeze-drying ($P < 0.05$). Regarding ADF expressed exclusive of protein (ADFp), microwave drying did not differ from freeze-drying ($P > 0.05$). Regardless of the method, heat application increased KL concentration ($P < 0.05$).

Although multiple comparisons did not reveal a consistent pattern, heat application reduced IVDMD ($P < 0.05$), with the most pronounced decrease observed for microwave drying (Table 3). No differences were observed for IVNDFD ($P > 0.24$). The iNDF concentration differed between freeze-drying and forced-air oven drying ($P < 0.05$), whereas the remaining methods overlapped with both. Overall, this pattern suggests that heat consistently increased iNDF concentration.

3.2. Experiment 2

Despite the inclusion of different forage groups in Experiment 2 and their inclusion in the statistical model (Eq. 9), direct comparisons among forages were not among our objectives. The inclusion of forage groups aimed to broaden the scope of results and the range of inferences. Accordingly, we chose not to explicitly discuss simple differences among forage groups, as the groups were assumed to be inherently different. Differences among forage groups are presented in Tables 4–8 and Figs. 2–4. However, the presentation and discussion of our results herein focus on the effects of the partial dehydration methods. Therefore, only the differences among forages that contribute to the understanding of method-related differences were presented and discussed.

Dehydration methods affected the amount of water presumably extracted during partial dehydration ($P < 0.01$), regardless of forage group ($P > 0.08$, Table 4). Freeze-drying and forced-air oven drying resulted in the greatest amount of water presumably extracted and did not differ from each other ($P > 0.05$), whereas air-fryer drying extracted the least amount of water ($P < 0.05$). In contrast, the effects of partial dehydration methods on residual water content depended on the forage group ($P < 0.02$, Table 4).

Table 1
Water removed, dry matter concentration, and final temperature of heat-based methods in maize silage samples (n = 20) subjected to different partial dehydration methods.

	Partial dehydration method[a, b]					
Item[c]	FD	OD	M + OD	M	SEM	P-value
WPR[d]	644a	623b	641a	598c	6.9	< 0.001
RW[e]	70.8c	116.5a	93.0b	129.3a	4.23	< 0.001
TW[d]	669ab	667ab	674a	650b	6.1	< 0.001
DM[d]	331ab	333ab	326b	350a	6.1	< 0.001
FT[f]	-	52.9 ± 0.5	56.9 ± 0.3	97.2 ± 0.5	-	-
IT[f]	-	-	44.6 ± 1.4	-	-	-

[a] FD, freeze-drying; OD, forced-air oven drying; M + OD, forced-air oven drying preceded by an initial microwave heating step; M, microwave drying. [b] Means in a row followed by different letters differ at $P < 0.05$. [c] WPR, water presum ably removed during partial dehydration; RW, residual water; TW, total water; DM, dry matter; FT, final sample temperature at the end of the partial dehydration process; IT, intermediate sample temperature after the initial microwave heating step. [d] g/kg as-is. [e] g/kg air-dried sample. [f] °C.

Table 2
Concentration of nitrogenous compounds (g/kg dry matter) in maize silage samples (n = 20) subjected to different partial dehydration methods.

	Partial dehydration method[a, b]					
Item[c]	FD	OD	M + OD	M	SEM	P-value
N	11.6a	10.8b	10.0c	9.9c	0.33	< 0.001
SN	7.28a	6.13b	5.49c	4.93d	0.379	< 0.001
IN	4.27c	4.64b	4.54b	4.94a	0.090	< 0.001
nNDIN	3.26b	3.49b	3.47b	3.79a	0.094	< 0.001
NDIN	1.02a	1.15a	1.07a	1.15a	0.041	0.029
ADIN	0.230b	0.366a	0.354a	0.386a	0.0202	< 0.001

[a] FD, freeze-drying; OD, forced-air oven drying; M + OD, forced-air oven drying preceded by an initial microwave heating step; M, microwave drying. [b] Means in a row followed by different letters differ at P < 0.05. [c] N, nitrogen; SN, soluble N; IN, insoluble N; nNDIN, insoluble N not bound to the cell wall; NDIN, neutral detergent insoluble N; ADIN, acid detergent insoluble N.

Table 3
Concentration of fibrous compounds (g/kg of dry matter) and in vitro digestibility (g/kg) in maize silage samples (n = 20) subjected to different partial dehydration methods.

	Partial dehydration method[a, b]					
Item[c, d]	FD	OD	M + OD	M	SEM	P-value
NDF	402	421	413	409	7.6	0.20
NDFp	396	414	407	402	7.5	0.22
ADF	207b	223a	213ab	212ab	4.7	0.003
ADFp	205b	220a	211ab	209b	4.6	< 0.001
KL	82.6b	97.1a	95.8a	90.9a	1.84	< 0.001
IVDMD	764a	735bc	756ab	725c	7.6	< 0.001
IVNDFD	563	562	580	543	13.9	0.24
iNDF	143b	158a	146ab	149ab	3.7	0.013

[a] FD, freeze-drying; OD, forced-air oven drying; M + OD, forced-air oven drying preceded by an initial microwave heating step; M, microwave drying. [b] Means in a row followed by different letters differ at P < 0.05. [c] NDF, neutral detergent fibre; NDFp, neutral detergent fibre expressed exclusive of protein; ADF, acid detergent fibre; ADFp, acid detergent fibre expressed exclusive of protein; KL, Klason lignin; IVDMD, in vitro dry matter digestibility; IVNDFD, in vitro NDF digestibility; iNDF, indigestible NDF. [d] Concentrations of NDF and ADF expressed exclusive of protein were obtained by subtracting the concentrations of NDIN and ADIN multiplied by the factor 6.25, respectively.

Differences were observed only for fresh grasses (P < 0.01), for which air-fryer drying resulted in the highest residual water content (P < 0.05).

Total water content was affected by dehydration method (P < 0.01), independently of forage group (P > 0.21). The highest water contents were observed with freeze-drying and forced-air oven drying, whereas microwave and air-fryer drying resulted in the lowest values (Table 4). Accordingly, DM content exhibited an inverse pattern.

The temperature of the subsamples at the end of the partial dehydration process was affected by the dehydration methods (P < 0.01), independently of forage group (P > 0.62). All methods differed from each other (P < 0.05), in ascending order: freeze-drying, forced-air oven drying, microwave drying, and air-fryer drying (Table 4).

For total N concentration, the effect of partial dehydration method depended on forage group (P < 0.03, Table 5). In fresh grasses and legumes, freeze-drying resulted in the highest N concentrations (P < 0.05). In contrast, using air-fryer drying for grasses and microwave drying for legumes produced the lowest N concentrations (P < 0.05), respectively. In the silage group, N concentration was not affected by the partial dehydration method (P > 0.55). A method × forage group interaction was also observed for SN (P < 0.01), with patterns in fresh grasses and legumes similar to those observed for total N. In silages, freeze-drying and air-fryer drying resulted in the highest and lowest SN concentrations (P < 0.05), respectively. Conversely, the effect of partial dehydration methods on IN was independent of forage group (P > 0.51), despite of a significant method effect (P < 0.01). Microwave and freeze-drying yielded the highest and the lowest IN concentrations, respectively.

The NPN concentration was affected by the partial dehydration methods (P < 0.01), with no interaction with forage group (P > 0.19, Table 5). On average, freeze-drying and forced-air oven drying resulted in higher NPN concentrations (P < 0.05). No effect of partial dehydration method (P ≥ 0.11) was observed on PN concentration.

An interaction between partial dehydration method and forage group was observed for nNDIN concentration (P < 0.01, Table 5). In fresh grasses, nNDIN was highest for freeze-drying and forced-air oven drying, lowest for air-fryer drying, and intermediate for microwave drying. In fresh legumes, freeze-drying resulted in the highest values, followed by forced-air oven drying, whereas microwave and air-fryer drying produced similar and lower concentrations (P > 0.05). In silages, nNDIN did not differ among methods (P > 0.98).

The SPN concentration was affected by the partial dehydration method (P < 0.01), independently of forage group (P > 0.17, Table 5). Freeze-drying resulted in the highest SPN concentration, whereas microwave drying produced the lowest values; forced-air oven and air-fryer drying showed intermediate concentrations.

A partial dehydration method × forage group interaction was detected for NDIN concentrations (P < 0.01, Table 5). In both fresh

Table 4
Water removed, dry matter concentration, and final temperature in forage samples of different groups subjected to different partial dehydration methods.

		Partial dehydration method[a, f]							P-value[d]		
Variable[b]	Group[c]	FD	OD	M	AF	P-value[e]	Mean[g]	SEM	G	M	G × M
WPR[h]	FG	758	756	749	726	-	747AB	14.8	0.028	0.001	0.088
	FL	786	793	776	785	-	785 A	16.6			
	SIL	730	723	720	713	-	722B	14.8			
	Mean[g]	758 A	758 A	748AB	741B	-	-	-			
	SEM	9.3				-	-	-			
RW[i]	FG	103b	103b	100b	118a	0,002	-	4.0	0.072	0.008	0.012
	FL	117a	113a	103a	104a	0,036	-	4.5			
	SIL	103	105	93	101	0,11	-	4.0			
	Mean	-	-	-	-	-	-	-			
	SEM	-									
TW[h]	FG	783	781	774	759	-	774AB	13.3	0.023	< 0.001	0.21
	FL	811	817	799	808	-	809 A	14.9			
	SIL	758	753	746	742	-	750B	13.3			
	Mean[g]	784 A	783 A	773B	770B	-	-	-			
	SEM	8.3				-	-	-			
DM[h]	FG	217	219	226	241	-	226AB	13.3	0.023	< 0.001	0.21
	FL	189	183	201	192	-	191B	14.9			
	SIL	242	247	254	258	-	250 A	13.3			
	Mean[g]	216B	217B	227 A	230 A	-	-	-			
	SEM	8.3				-	-	-			
FT[j]	FG	31.6	51.3	80.1	102.9	-	-	-	0.94	< 0.001	0.62
	FL	30.2	49.4	86.1	102.0	-	-	-			
	SIL	31.7	51.3	87.0	98.8	-	-	-			
	Mean[g]	31.1D	50.7 C	84.4B	101.2 A	-	-	-			
	SEM	1.77				-	-	-			

[a] FD, freeze-drying; OD, forced-air oven drying; M, microwave drying; AF, air-fryer drying. [b] WPR, water presumably removed during partial dehydration; RW, residual water; TW, total water; DM, dry matter; FT, final sample temperature at the end of the partial dehydration process. [c] FG, fresh grasses (n = 10); FL, fresh legumes (n = 8); SIL, silages (n = 10). [d] G, M, and G × M, effects of the forage group, partial dehydration method, and their interaction, respectively. [e] P-values refer to the comparison between partial dehydration methods nested within each forage group (it was applied in case of significant interaction). [f] Means in the line, within each forage group, followed by different lowercase letters differ at $P < 0.05$. [g] Estimated marginal means in a line or column followed by different uppercase letters differ at $P < 0.05$. [h] g/kg as-is. [i] g/kg air-dried sample. [j] °C.

grasses and legumes, freeze-drying and forced-air oven drying resulted in lower NDIN concentrations, whereas microwave and air-fryer drying produced higher values ($P < 0.05$). No differences among methods were observed for NDIN in silages ($P > 0.57$). A partial dehydration method × forage group interaction was also detected ($P < 0.01$) for ADIN concentrations. However, method effects were observed only for fresh legumes ($P < 0.01$), in which air-fryer drying increased ADIN concentrations ($P < 0.05$).

The NDF concentrations exhibited a partial dehydration method × forage group interaction ($P < 0.01$, Table 6). In fresh grass, freeze-drying and air-fryer drying resulted in the lowest and highest NDF concentrations, respectively ($P < 0.05$). Forced-air oven drying and microwave drying produced intermediate values, with forced-air oven drying resembling freeze-drying, and microwave drying resembling air-fryer drying. In fresh legumes, heat application increased NDF concentrations compared with freeze-drying ($P < 0.05$), following an ascending order of forced-air oven drying, microwave drying, and air-fryer drying, with the latter two not differing from each other ($P > 0.05$). The concentration of NDFp was affected by dehydration method ($P < 0.01$), with no interaction with forage group ($P > 0.94$). The highest NDFp concentration was obtained with air-fryer drying ($P < 0.05$), whereas the other methods did not differ from each other.

Effects of partial dehydration methods on ADF concentration depended on forage group ($P < 0.01$, Table 6). Differences among methods were observed for fresh legumes only, in which air-fryer drying increased ADF concentration ($P < 0.05$). A method × forage group interaction ($P < 0.01$) was also observed for ADFp. In fresh legumes, microwave drying resulted in the lowest ADFp concentration ($P < 0.05$). Interaction between partial dehydration method and forage group was also detected for ADL concentration ($P < 0.01$). For both fresh grasses and legumes, higher ADL concentrations ($P < 0.05$) were obtained with air-fryer drying.

The iNDF concentration was not affected by partial dehydration method or by the method × forage group interaction ($P \geq 0.10$, Table 6). A partial dehydration method × forage group interaction was detected for IVDMD ($P < 0.01$). In fresh legumes, IVDMD was highest for freeze-drying and decreased with forced-air oven drying and air-fryer drying, whereas microwave drying resulted in intermediate values. In vitro NDF digestibility was not affected by partial dehydration method or its interaction with forage group ($P \geq 0.18$).

A partial dehydration method × forage group interaction was observed for lightness (L*; $P < 0.01$, Table 7). In fresh grasses, L* was lowest in the original material, highest with freeze-drying, and intermediate for all heat-based methods. In fresh legumes, a similar pattern was observed, although air-fryer drying resulted in intermediate values. In silages, freeze-drying and forced-air oven drying produced the highest L*, followed by microwave and air-fryer drying, whereas the original material exhibited the lowest L*. A partial dehydration method × forage group interaction was observed for hue angle ($P < 0.01$). In fresh forages, original samples exhibited the

Table 5
Concentration of nitrogenous compounds (g/kg of dry matter) in forage samples of different groups subjected to different partial dehydration methods.

		Partial dehydration method[a, f]							P-value[d]		
Variable[b]	Group[c]	FD	OD	M	AF	P-value[e]	Mean[g]	SEM	G	M	G × M
N	FG	12.8a	12.3ab	12.2ab	11.3b	0.007	-	1.61	< 0.001	< 0.001	0.024
	FL	32.9a	32.2ab	30.3c	31.1bc	< 0.001	-	1.80			
	SIL	10.2	9.9	9.7	9.7	0.55	-	1.61			
	Mean	-	-	-	-	-	-	-			
	SEM	-				-	-	-			
SN	FG	5.71a	4.77ab	3.54bc	3.17c	< 0.001	-	0.885	0.001	< 0.001	0.003
	FL	11.78a	10.30b	7.33d	8.82c	< 0.001	-	0.989			
	SIL	6.96a	6.37ab	5.70ab	5.52b	0.019	-	0.885			
	Mean	-	-	-	-	-	-	-			
	SEM	-				-	-	-			
IN	FG	7.15	7.47	8.62	8.10	-	7.84B	0.962	< 0.001	< 0.001	0.51
	FL	21.12	21.92	23.08	22.32	-	22.10 A	1.08			
	SIL	3.25	3.55	3.95	4.13	-	3.73 C	0.962			
	Mean[g]	10.51 C	10.98BC	11.86 A	11.52AB	-	-	-			
	SEM	0.595				-	-	-			
NPN	FG	4.62	3.95	3.06	2.40	-	3.50B	0.834	0.060	< 0.001	0/19
	FL	9.34	8.53	6.83	6.85	-	7.89 A	0.931			
	SIL	5.89	5.01	4.98	4.80	-	5.30AB	0.834			
	Mean[g]	6.61 A	5.98 A	4.96B	4.69B	-	-	-			
	SEM	0.539				-	-	-			
PN	FG	8.26	8.30	9.10	8.85	-	8.62B	1.397	< 0.001	0.11	0.77
	FL	23.55	23.71	23.50	24.29	-	23.76 A	1.317			
	SIL	4.32	4.43	4.67	4.83	-	4.56 C	1.397			
	Mean	12.04	12.14	12.14	12.66	-	-	-			
	SEM	0.539				-	-	-			
nNDIN	FG	4.14a	4.04a	2.63ab	1.68b	0.009	-	0.769	< 0.001	< 0.001	< 0.001
	FL	12.88a	8.05b	4.81c	4.12c	< 0.001	-	0.860			
	SIL	1.87	1.95	1.72	1.64	0.98	-	0.769			
	Mean	-	-	-	-	-	-	-			
	SEM	-									
SPN	FG	1.10	0.82	0.47	0.75	-	1.71 A	0.176	0.003	< 0.001	0.17
	FL	2.43	1.78	0.66	1.97	-	0.79B	0.198			
	SIL	1.07	0.89	0.72	0.71	-	0.86B	0.176			
	Mean[g]	1.53 A	1.17AB	0.62B	1.14AB	-	-	-			
	SEM	0.165				-	-	-			
NDIN	FG	3.01b	3.44b	6.00a	6.43a	< 0.001	-	0.826	< 0.001	< 0.001	0.004
	FL	8.24c	13.87b	18.19a	18.18a	< 0.001	-	0.932			
	SIL	1.38	1.60	2.24	2.50	0.57	-	0.826			
	Mean	-	-	-	-	-	-	-			
	SEM	-				-	-	-			
ADIN	FG	0.74	0.88	1.22	1.18	0.88	-	0.560	< 0.001	< 0.001	< 0.001
	FL	2.37b	3.70b	4.40b	8.11a	< 0.001	-	0.626			
	SIL	0.69	0.75	0.94	0.86	0.98	-	0.560			
	Mean	-	-	-	-	-	-	-			
	SEM	-				-	-	-			

[a] FD, freeze-drying; OD, forced-air drying oven; M, microwave drying; AF, air-fryer drying. [b] N, nitrogen; NS, soluble N; IN, insoluble N; NPN, non-protein N; PN, protein N; nNDIN, insoluble N not bound to the cell wall; SPN, soluble protein N; NDIN, neutral detergent insoluble N; ADIN, acid detergent insoluble N. [c] FG, fresh grasses (n = 10); FL, fresh legumes (n = 8); SIL, silages (n = 10). [d] G, M, and G × M, effects of the group, partial dehydration method, and their interaction, respectively. [e] P-values refer to the comparison between partial dehydration methods nested within each forage group (applied in case of significant interaction). [f] Means in the line, within each forage group, followed by different lowercase letters differ at $P < 0.05$. [g] Estimated marginal means in a line or column followed by different uppercase letters differ at $P < 0.05$.

highest hue values, which were, on average, reduced after partial dehydration, particularly when heat-based methods were applied. In silages, hue angle was not affected by partial dehydration method ($P > 0.05$). The colour patterns of all evaluated materials are presented in Table 8.

4. Discussion

Before interpreting the results, it is important to clarify the basis of the chemical and digestibility measurements. Because all analyses were performed after partial dehydration, the statistical comparisons presented here test differences in the measured chemical composition and digestibility of the resulting DM among dehydration methods. Thus, these results demonstrate changes in the analytical characteristics of the obtained DM, but they do not by themselves quantify the absolute recovery or loss of individual chemical constituents from the original fresh material. Such quantification would require direct analyses of the fresh material or a

Table 6
Concentration of fibrous compounds (g/kg of dry matter) and in vitro digestibility (g/kg) in forage samples of different groups subjected to different partial dehydration methods.

		Partial dehydration method[a, f]							P-value[d]		
Variable[b]	Group[c]	FD	OD	M	AF	P-value[e]	Mean[g]	SEM	G	M	G × M
NDF	FG	680c	684bc	715ab	750a	< 0.001	-	33.7	0.006	< 0.001	< 0.001
	FL	467c	520b	562a	590a	< 0.001	-	37.6			
	SIL	617	605	620	638	0.063	-	33.7			
	Mean	-	-	-	-	-	-	-			
	SEM	-				-	-	-			
NDFp	FG	661	662	678	709	-	678 A	47.6	< 0.001	< 0.001	0.94
	FL	416	433	448	475	-	443B	44.9			
	SIL	608	595	606	622	-	608 A	47.6			
	Mean	562B	563B	577B	602 A	-	-	-			
	SEM	19.4					-	-			
ADF	FG	384	384	381	395	0.64	-	24.9	0.50	< 0.001	0.001
	FL	334b	341b	310b	393a	< 0.001	-	27.9			
	SIL	370	370	380	384	0.51	-	24.9			
	Mean	-	-	-	-	-	-	-			
	SEM	-				-	-	-			
ADFp	FG	380	378	373	387	0.50	-	24.1	0.17	< 0.001	0.006
	FL	319a	318a	283b	342a	< 0.001	-	27.0			
	SIL	365	366	374	379	0.40	-	24.1			
	Mean	-	-	-	-	-	-	-			
	SEM	-				-	-	-			
ADL	FG	38.7b	39.6b	43.7b	52.8a	< 0.001	-	6.28	< 0.001	< 0.001	< 0.001
	FL	85.4b	93.3b	87.5b	114.2a	< 0.001	-	7.03			
	SIL	54.5	56.3	59.9	63.2	0.054	-	6.28			
	Mean	-	-	-	-	-	-	-			
	SEM	-				-	-	-			
iNDF	FG	192	198	203	194	-	197	23.9	0.20	0.10	0.20
	FL	241	254	244	264	-	251	26.7			
	SIL	248	259	247	251	-	251	23.9			
	Mean	227	237	232	237	-	-	-			
	SEM	4.6				-	-	-			
IVDMD	FG	591a	583a	557a	555a	0.027	-	25.4	0.70	< 0.001	< 0.001
	FL	602a	546b	507bc	501c	< 0.001	-	28.4			
	SIL	565	560	560	560	0.97	-	25.4			
	Mean	-	-	-	-	-	-	-			
	SEM	-				-	-	-			
IVNDFD	FG	508	495	492	495	-	498 A	25.3	0.005	0.18	0.20
	FL	379	328	379	372	-	364B	28.3			
	SIL	413	412	395	418	-	409AB	25.3			
	Mean	434	412	422	428	-	-	-			
	SEM	16.4				-	-	-			

[a] FD, freeze-drying; OD, forced-air drying oven; M, microwave drying; AF, air-fryer drying. [b] NDF, neutral detergent fibre; NDFp, neutral detergent fibre expressed excusive of protein; ADF, acid detergent fibre; ADFp, acid detergent fibre expressed exclusive of protein; ADL, acid detergent lignin; IVDMD, in vitro dry matter digestibility; IVNDFD, in vitro NDF digestibility; iNDF, indigestible neutral detergent fibre. [c] FG, fresh grasses (n = 10); FL, fresh legumes (n = 8); SIL, silages(n = 10). [d] G, M, and G × M, effects of the group, partial dehydration method, and their interaction, respectively. [e] P-values refer to the comparison between partial dehydration methods nested within each forage group (applied in case of significant interaction). [f] Means in the line, within each forage group, followed by different lowercase letters differ at $P < 0.05$. [g] Estimated marginal means in a line or column followed by different uppercase letters differ at $P < 0.05$.

mass-balance approach, which was not feasible for the analytical procedures employed in the present study. Accordingly, the interpretations below focus on changes in the analytical characteristics of the resulting DM and their implications for forage evaluation rather than on absolute recovery of individual constituents.

Overall, microwave drying resulted in lower water extraction from samples compared with freeze-drying. A similar pattern was observed when using air-fryer drying. Considering the DM estimate obtained with the freeze-drying method as a standard value, the DM content was, on average, overestimated by 5.1–5.7% using microwave drying, and by 6.5% using air-fryer drying, which indicates a reduced water extraction during the partial dehydration processes. This lower water extraction may be attributed to difficulties of these methods in removing structural water from feed materials (e.g., water adsorbed to macromolecules), which present greater quantification challenges (Khan and Karim, 2017; Joardder et al., 2017). Conversely, greater water extraction by freeze-drying compared to microwave and air-fryer drying may be attributed to its higher efficiency in water removal through sublimation relative to water removal through evaporation (Bhatta et al., 2020; Joardder et al., 2017). However, even though the amount of residual water extracted was greater for microwave drying (Experiment 1) and air-fryer drying (fresh legumes, Experiment 2), we did not observe a perfectly balanced final water extraction, which resulted in higher DM concentrations when partial dehydration was performed using microwave or air-fryer drying. Despite the differences observed in Experiment 1, we observed, on average, a closer

Table 7
Colorimetric evaluation of forage samples of different groups subjected to different partial dehydration methods.

		Partial dehydration method[a, f]								P-value[d]		
Variable[b]	Group[c]	F	FD	OD	M	AF	P-value[e]	Mean	SEM	G	M	G × M
L*	FG	36.5c	55.7a	49.7b	47.2b	47.3b	< 0.001	-	1.49	< 0.001	< 0.001	< 0.001
	FL	27.4c	39.1a	31.2b	28.3b	28.0bc	< 0.001	-	1.67			
	SIL	37.8c	51.8a	49.4a	45.6b	44.7b	< 0.001	-	1.49			
	Mean	-	-	-	-	-	-	-	-			
	SEM	-					-	-	-			
h°	FG	103.8a	99.7ab	96.3b	89.3c	88.2c	< 0.001	-	2.33	< 0.001	< 0.001	< 0.001
	FL	100.8a	95.8b	84.9c	80.7c	81.5c	< 0.001	-	2.61			
	SIL	75.9a	79.1a	78.7a	76.3a	75.0a	0.030	-	2.33			
	Mean	-	-	-	-	-	-	-	-			
	SEM	-					-	-	-			

[a] F, fresh material; FD, freeze-drying; OD, forced-air drying oven; M, microwave drying; AF, air-fryer drying. [b] L*, lightness coefficient; h°, hue angle. [c] FG, fresh grasses (n = 10); FL, fresh legumes (n = 8); SIL, silages (n = 10). [d] G, M, and G × M, effects of the group, partial dehydration method, and their interaction, respectively. [e] P-values refer to the comparison between partial dehydration methods nested within each forage group (applied in case of significant interaction). [f] Means in the line, within each forage group, followed by different lowercase letters differ at P < 0.05.

similarity in total water extracted when partial dehydration was carried out by freeze-drying and by the exclusive use of forced-air oven drying.

Nevertheless, the pattern of water extraction alone may not fully explain the observed differences in DM estimates, as the components removed during partial dehydration are not exclusively water, and the outcomes must be interpreted in conjunction with the other response variables evaluated in this study. Previous research has demonstrated that the application of heat during partial dehydration can promote losses of volatile substances and water formation through undesirable non-enzymatic reactions (Van Soest and Mason, 1991; Van Soest, 1994; Bradley, 2010; Mauer and Bradley, 2017; Evan et al., 2025). We emphasize that volatilisation is not restricted to N compounds; losses of several organic constituents may also occur, even during freeze-drying, potentially leading to biased estimates of DM content (Evan et al., 2025). Collectively, these mechanisms support the lack of a perfect balance between the amount of water presumably extracted during partial dehydration and the residual water recovered by loss-on-drying (a procedure common to all evaluated methods), thereby explaining the differences observed in total water and DM estimates.

An indirect evidence that supports the occurrence of chemical side reactions during partial dehydration can be obtained by comparing maize silage subjected to freeze-drying with samples drying in a forced-air oven preceded by an initial microwave heating step (Experiment 1). Although both methods resulted in similar amounts of water presumably removed during partial dehydration, residual water content was greater when drying involved the force-air oven with prior microwave heating. Because the second drying stage (i.e., loss-on-drying) was identical for both methods, the higher residual water associated with the heat-based approach indicates that part of the water removed during partial dehydration consisted of water generated during heat-mediated non-enzymatic reactions. In such reactions, intermediate compounds, such as Schiff's bases, are formed with concomitant water release (Van Soest, 1994; Eskin et al., 2013). Accordingly, this pattern observed in Experiment 1 can be extended to other heat-based methods and raises concerns regarding the actual capacity for water removal during partial dehydration when excessive heat is applied. Consequently, a direct relationship appears to exist between the temperature applied during partial dehydration, the magnitude of the differences observed in the measured chemical characteristics, and the divergence in total water extracted. Notably, the highest temperatures were achieved when microwave and air-fryer were used for partial dehydration.

The most pronounced differences observed in our study were associated with N compounds. Initially, heat reduced the total N concentration in both experiments compared with freeze-drying. Although this effect was not detected for silages in Experiment 2, it was clearly observed in the exclusive evaluation of maize silage in Experiment 1. Therefore, we consider it appropriate to conclude that heating forage samples during partial dehydration results in lower measured N concentrations and may promote N volatilisation.

However, it is worth noting that volatilisation is not the only modification affecting N compounds under excessive heat. Such modifications may also include reductions in solubility (Deinum and Maassen, 1994; Van Soest, 1994) and in potential of ruminal degradation (Sadeghi and Shawarang, 2006; Doiron et al., 2009). In our study, chemical evaluation of original material was not performed due to extensive handling and processing difficulties. Although freeze-drying is not entirely free of N losses (Morris et al., 2019), it is widely recognized as the partial dehydration method that results in the least loss of analyte integrity (Smith, 1981; Morris et al., 2019). Therefore, due to numerous interactions observed in Experiment 2 and the need for integration of both experiments, we summarised N compound modification data graphically in Figs. 2 and 3.

Volatilisation losses of N compounds may contribute to reductions in total N concentration in the study material. However, as previously highlighted, changes promoted by excessive heat encompass both volatilisation and insolubilisation processes, which may be captured by evaluating the total pool of soluble N compounds. In this context, we observed a clear association between heating intensity during partial dehydration and the magnitude of the differences observed in N compound concentrations and profiles, regardless of experiment and forage type (Fig. 2). In Experiment 1, the reduction in total N concentration was more pronounced, which appears to reflect the specific characteristics of the forage evaluated (i.e., maize silage). Although NPN was not evaluated in Experiment 1, results from Experiment 2 indicated that reductions in NPN concentration numerically mirrored the reduction in total N concentration, with no changes observed in the protein N pool. These findings suggest that the apparent reduction in total N concentration may have involved preferential losses of N from the NPN pool. Among the heat-based partial dehydration methods, forced-

Table 8
Coloration pattern of forage samples from different groups subjected to different partial dehydration methods.

	Method[a,b]				
Sample[c]	F	FD	OD	M	AF
1	103,81,49	155,130,94	139,118,84	128,105,71	119,97,68
2	106,83,52	128,109,80	131,109,78	126,98,64	126,101,69
3	94,73,45	136,110,75	130,108,76	113,90,59	126,102,71
4	103,78,43	137,117,86	133,110,78	122,96,62	122,99,69
5	132,103,67	164,139,106	150,126,98	158,132,102	125,124,91
6	125,98,62	143,124,97	134,114,87	123,99,72	134,107,77
7	118,98,64	151,134,106	148,132,103	150,130,97	132,111,81
8	117,94,58	139,120,89	140,121,90	133,111,77	129,107,76
9	89,68,47	117,93,70	115,91,65	107,83,58	109,83,60
10	90,68,48	130,106,79	116,92,66	107,83,58	91,69,50
11	92,87,57	135,126,97	124,113,86	121,106,80	112,96,70
12	124,104,71	169,145,107	149,126,95	145,120,89	140,114,81
13	97,94,66	142,137,107	144,131,104	147,134,103	150,130,97
14	74,77,50	128,127,100	111,109,84	101,91,68	108,98,71
15	87,88,59	128,127,100	124,117,85	121,108,77	132,118,83
16	87,85,51	133,123,94	111,102,77	106,92,66	108,96,68
17	69,73,45	136,136,107	114,114,87	112,105,78	121,115,83
18	81,88,52	136,136,105	133,130,99	124,116,87	127,116,83
19	88,85,52	130,118,86	116,109,82	119,107,79	117,101,72
20	89,88,52	143,140,106	115,109,81	122,111,82	121,109,80
21	80,71,51	94,80,58	83,68,48	82,66,49	81,66,46
22	70,71,53	110,108,84	100,88,68	95,86,69	101,93,74
23	56,56,46	81,76,52	60,54,41	66,51,35	53,44,36
24	69,74,54	104,105,79	83,77,56	60,55,40	86,77,54
25	71,70,48	96,93,68	88,77,56	83,70,49	79,64,45
26	63,57,42	100,89,64	72,60,45	73,59,42	68,58,42
27	68,57,40	101,84,61	84,71,51	82,67,48	77,61,43
28	72,71,49	97,94,67	94,84,59	84,72,50	69,62,43

[a] F, fresh material; FD, freeze-drying; OD, forced-air drying oven; M, microwave drying; AF, air-fryer drying. [b] The values inside each cell correspond to the RGB coordinates. [c] 1–4, maize silage; 5–6, sugarcane silage; 7–8, elephant grass silage; 9–10, sorghum silage; 11, Tifton 85; 12 Saccharum sp.; 13 P. purpureum; 14, U. decumbens; 15, U. brizantha cv. Marandu; 16. C. dactylon; 17, M. maximus cv. BRS Zuri; 18, M. maximus cv Mombaça; 19, U. humidicola; 20, M. maximus cv Massai; 21, A. Pintoi; 22, C. canjan; 23, L. leucocephala; 24, G. sepium; 25, L. purpureus; 26, P. phaseoloides; 27, S. guanensis cv. Mineirão, and 28, N. wightii.

air oven drying resulted in the smallest apparent reduction in N concentration in both experiments (Fig. 2).

As highlighted earlier, part of the differences observed in N fractions may arise from insolubilisation processes. Unlike volatilisation, the intensity of heat-induced insolubilisation appears to be forage-type dependent. Minimal changes were observed in insoluble

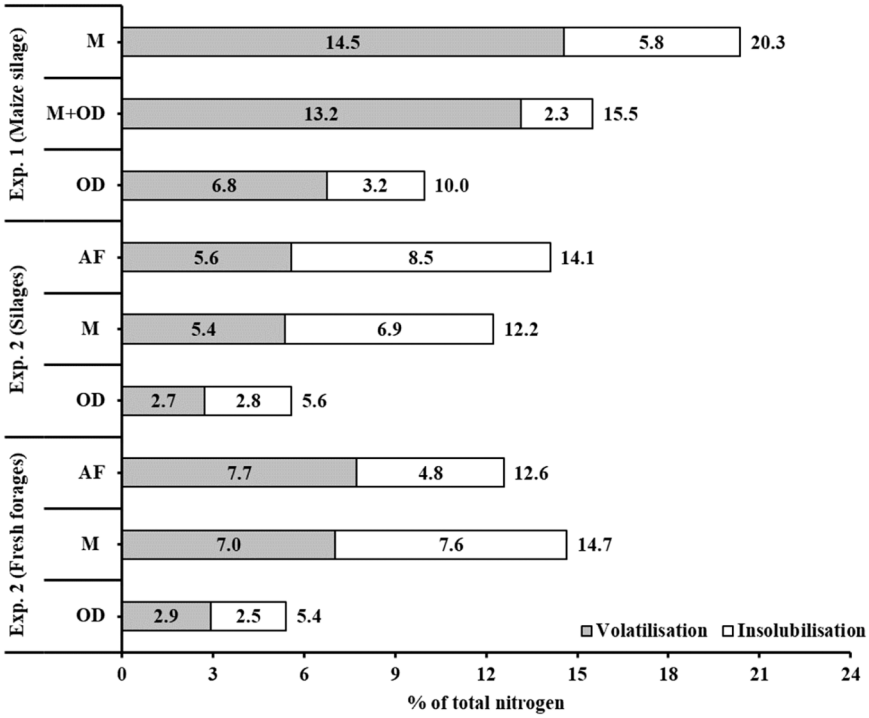

Fig. 2. Total modification of nitrogen compounds in forage samples subjected to heat-based partial dehydration methods. Calculations were based on the nitrogen contents obtained from the freeze-drying method (OD, oven-drying; M+OD forced-air oven drying preceded by an initial microwave heating step; M, microwave drying; and AF, air-fryer-drying).

N fractions in silages (Fig. 3), whereas the most pronounced differences in insoluble N fractions occurred in fresh forage samples (Fig. 3, Table 5). High temperature is the primary factor promoting these reactions, although additional characteristics, such as pH, may also play a relevant role (Eskin et al., 2013). For example, Lee et al. (1984) demonstrated that, even when samples are exposed to identical temperatures, the formation of non-enzymatic intermediates is reduced under acidic conditions, which may explain the lower intensity of modification in silage samples.

Our results highlight the complex nature of changes in N compound solubility as influenced by the time × temperature binomial during partial dehydration. The first modification to be addressed concerns the reduction in the soluble N pool (Fig. 2), which appears to affect the soluble protein N fraction, as observed in Experiment 2, rather than the NPN, as previously discussed. However, this modification apparently does not seem to arise solely from a decrease in solubility. If that were the case, a concomitant increase in the nNDIN pool would be expected, which was not observed. Instead, a simultaneous reduction in both SPN and nNDIN occurred, rendering these fractions analytically similar to cell wall-bound N compounds, as evidenced by the increase in NDIN fraction (Fig. 3).

Thus, excessive heat during partial dehydration, particularly in fresh forages, likely promotes non-enzymatic polymerization reactions involving both soluble protein and nNDIN (Van Soest, 1994; Pelletier et al., 2010), thereby potentially increasing the proportion of N analytically recovered as cell-wall-associated N. However, the most pronounced differences were characterized by increases in NDIN concentration, with comparatively smaller increases in ADIN concentration, indicating that a substantial proportion of the N analytically recovered as NDIN was not insoluble in acid detergent. Two aspects of this dynamic must be emphasised: (1) the magnitude of these differences is greater in fresh forages than in silages; and (2) among the heat-based methods, the forced-air oven drying consistently resulted in the smallest differences in the measured N characteristics.

The hypothesis of artefact formation through non-enzymatic reactions is reinforced by the colourimetric characteristics of the samples. Overall, colour evaluation may provide a practical and rapid complementary indicator of heat damage in feed samples because it allows visual detection of colour shifts towards brown and/or black within a specific study material, which are characteristic of non-enzymatic browning reactions (Quirino et al., 2023). As such, in our study, colour evaluation provided valuable complementary evidence and can be interpreted in two stages. First, by comparing fresh and freeze-dried materials: fresh samples showed a more intense green coloration (Table 8; samples 11–28, except sugarcane). This was corroborated by their higher hue angle values, which, when exceeding 90°, indicate a shift towards green (180°; Wrolstad and Smith, 2017). Fresh samples also exhibited lower lightness values, resulting in a visually darker appearance. After freeze-drying, the samples became lighter, with increased lightness and hue values approaching 90°, which denotes a yellowish tone. This pattern likely reflects pigment degradation promoted by the freeze-drying process.

It seems logical to presume that pigment degradation also occurs with heat use in partial dehydration. However, in these cases, the colour pattern differed from that observed with freeze-drying. With heat application, the hue angles shifted away from yellow (i.e.,

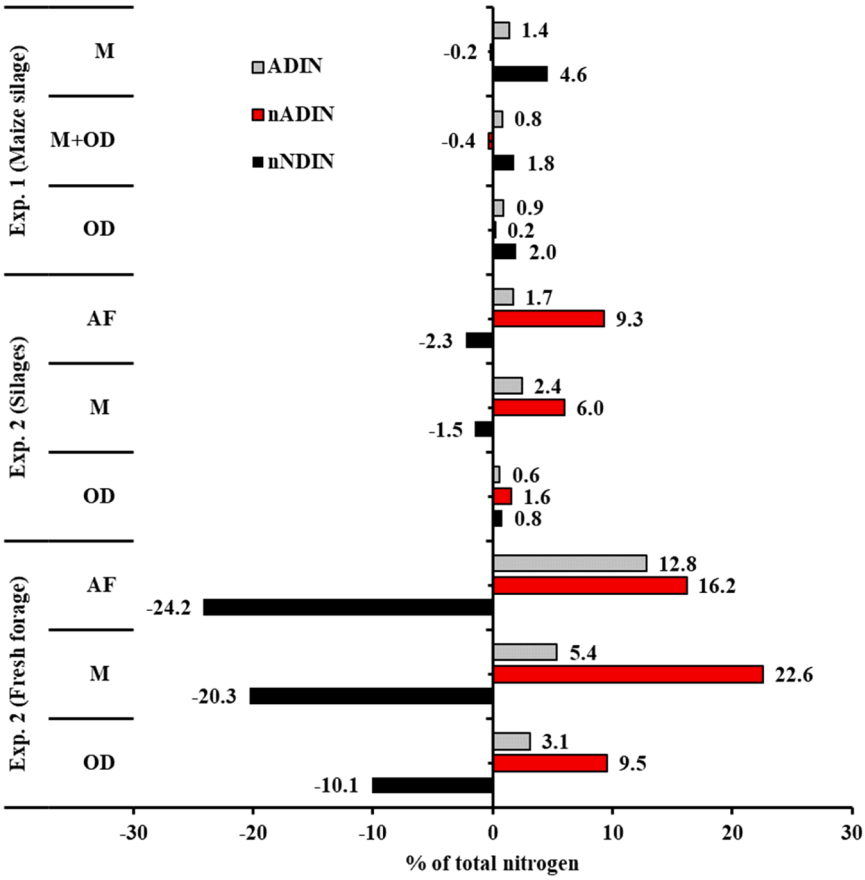

Fig. 3. Total modifications of insoluble nitrogenous compound fractions in forage samples subjected to heat-based partial dehydration methods. Calculations were based on the nitrogen contents obtained from the freeze-drying method (OD, oven-drying; M+OD forced-air oven drying preceded by an initial microwave heating step; M, microwave drying; AF, air-fryer-drying; nNDIN, insoluble N not bound to the cell wall; nADIN, N insoluble in neutral detergent but soluble in acid detergent; ADIN, acid detergent insoluble N).

90°) towards red (i.e., 0°), an effect more pronounced when microwave and air-fryer methods were applied. Additionally, in comparison with freeze-drying, the use of heat led to a reduction in lightness values, which tended to shift towards black (i.e., L = 0). The combination of yellow with an increased contribution of red and black results in brown colour manifestation (Quirino et al., 2023), a coloration characteristically associated with artefacts generated by non-enzymatic reactions (Zhang et al., 2018). Thus, a clear confluence emerges between the modifications in N compound profiles and colorimetric patterns, indicating a clear divergence in the measured chemical characteristics associated with excessive heat during the partial dehydration process. Once again, among heat-based methods, forced-air oven drying showed the smallest differences in the measured chemical characteristics relative to freeze-drying.

The increase in IN contents and their concomitant chemical modifications may contribute to differences in fibre concentrations. However, extensive overlap in the multiple comparison profiles hindered a clear establishment of these associations. In silages from both experiments, no differences were detected in NDF concentrations, except for a marked increase in the overall mean concentration of NDFp when air-fryer drying was applied.

In fresh forages, NDF concentrations increased with the heat-based methods, although this effect was mitigated when NDF concentrations were expressed with proper correction for contaminating protein. In this case, we again emphasize the deleterious effect of air-fryer drying. As exposure to elevated temperatures increased, so did the occurrence of non-enzymatic reactions (Feather and Nelson, 1984). The insoluble compounds formed during these reactions contain N in their structure (Van Soest, 1965; Zhang et al., 2018), which could lead to more pronounced differences among methods when NDF is expressed without proper protein correction, thereby supporting the pattern observed for fresh forages. It is important to note that fresh forages are generally richer in amino acid compounds (Rooke and Hatfield, 2003), which serve as substrates for non-enzymatic polymerisation reactions (Van Soest, 1994; Zhang et al., 2018). In contrast, beyond the acidic pH resulting from fermentation, ensiling conditions also promote the conversion of protein nitrogen into ammonia nitrogen. Consequently, because silages contain lower concentrations of amino acid compounds, the artificial increase in fibre concentration tends to be less pronounced, which corroborates the results here observed.

To better contextualize these effects, the ratio between NDF values obtained without and with correction for contaminating protein

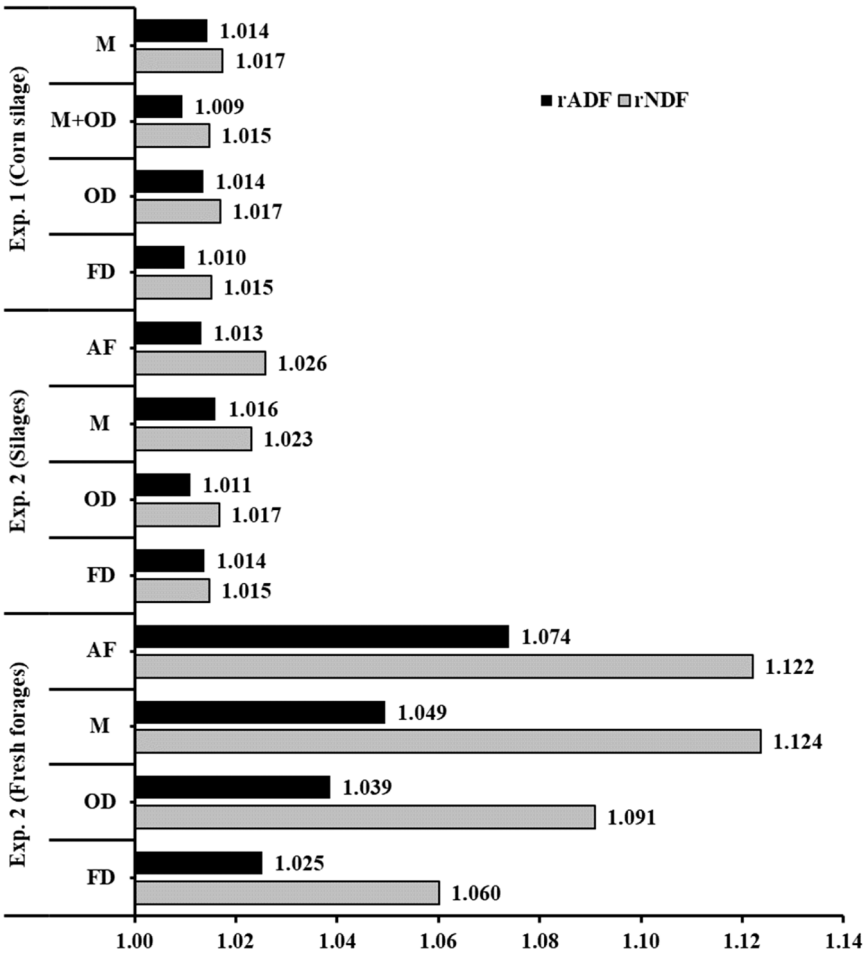

Fig. 4. Ratio between neutral detergent fibre (NDF) and acid detergent fibre (ADF) concentrations with and without correction for contaminant protein in forages subjected to different partial dehydration methods (FD, freeze-drying; OD, oven-drying; M+OD forced-air oven drying preceded by an initial microwave heating step; M, microwave drying; and AF, air-fryer-drying).

was calculated. For silage samples, this ratio remained relatively constant and close to unity, indicating minimal influence of partial dehydration methods on NDF estimates (Fig. 4). In contrast, markedly higher ratios were observed for fresh forages, increasing proportionally with the intensity of heat applied to the samples. This pattern reinforces the interpretation that heat-based partial dehydration methods promote modifications in nitrogenous compounds, which, in turn, affect fibre measurements.

The ADF concentrations in Experiment 1 and in fresh legumes from Experiment 2 were also affected by the partial dehydration methods. However, once more, the overlap among means limited a clear identification of consistent method-related pattern. Despite this, one specific result merits attention. In fresh legumes from Experiment 2, microwave drying resulted in a marked decrease in ADFp. One possible explanation is the disruption of lignocellulosic structures by microwave radiation (Brodie, 2007). Nonetheless, this mechanism alone does not appear sufficient to explain a response restricted to a single forage type. An alternative explanation may be associated with the higher tannin concentrations typically found in legumes. In these materials, heat may favour the formation of artefacts, particularly tannin–protein complexes, which are retained in the ADF residue (Parissi et al., 2005; Kaplan et al., 2025). Although correction for contaminating N contributed to greater homogeneity among methods, the ratio between uncorrected and corrected ADF values revealed a pattern analogous to that described for NDF, with an important distinction (Fig. 4). When fresh forages were considered, the effects of heating on these ratios were more pronounced for NDF than for ADF. This observation supports the interpretation that N insolubilisation in fresh forages predominantly increases the NDIN pool, without a proportional increase in ADIN pool. Consistent with other results, forced-air oven drying was the heat-based method associated with the lowest degree of alteration in fibrous components.

Despite the well-documented indigestibility of artefacts created by non-enzymatic reactions (Deinum and Maassen, 1994; Van Soest, 1994; Pelletier et al., 2010; Kaplan et al., 2025), no effect on IVNDFD was detected in our study. This finding contrasts with the results reported by Pelletier et al. (2010), who observed a decrease in IVNDFD when forage samples were partially dehydrated using heat-based methods. Conversely, some authors have suggested that microwave radiation may enhance digestibility. Owing to rapid heating under conditions of high moisture, microwave exposure may promote the formation of vapor bubbles within plant cells,

leading to cell rupture and improved enzymatic accessibility to the interior of cell (Chen et al., 2012; Joardder et al., 2017; Brodie et al., 2019). Nevertheless, such mechanisms were not supported by the results obtained in the present study. Although an increase in lignin concentration was observed with the use of heat-based methods in maize silage samples (Experiment 1) and with the use of air-fryer drying for fresh forages (Experiment 2), these changes did not align with the pattern observed for IVNDFD. A roughly similar pattern was verified for iNDF concentration. Despite the overlap observed in the multiple comparison tests, heat application increased iNDF concentration in maize silage samples in Experiment 1, whereas no such alteration was detected in the Experiment 2. Although not associated with changes in IVNDFD, heat-associated alterations in iNDF concentration may represent a concern when this component is used as an internal marker in digestion trials with ruminant animals.

However, IVDMD was consistently reduced when maize silage (Experiment 1) and fresh legumes (Experiment 2) were subjected to heat-based drying methods. It is important to emphasise that the most pronounced IVDMD reductions were observed with microwave and air-fryer drying. As no corresponding alterations were detected in IVNDFD, the decrease in IVDMD appears to be primarily associated with changes in cell-content components rather than in the fibrous fraction.

Specifically, in the case of maize silage, we hypothesize that starch retrogradation may have occurred as a result of heat exposure. Heat application during partial dehydration procedure can promote starch gelatinisation, which involves disruption of the crystalline structure of starch granules (Sajilata et al., 2006). Upon heating, water is absorbed by the granules, leading to their swelling and partial solubilisation of starch components. However, during subsequent cooling, a syneresis process occurs, accompanied by the formation of new double-helix structures that differ from the native configuration and exhibit greater resistance to enzymatic digestion. Amylose retrogradation can take place within hours after cooling (Sajilata et al., 2006). Moreover, microwave irradiation has been reported to decrease starch solubility and alter its crystalline structure, rendering it less ordered and more amorphous, which may facilitate subsequent retrogradation (Jiang et al., 2017). Therefore, we hypothesised that the heating-cooling sequence experienced by maize silage samples during partial dehydration promoted starch retrogradation, thereby contributing to the reduction in IVDMD observed in the present study.

With regard to fresh legumes, their inherently higher protein contents provide a greater availability of substrates for non-enzymatic reactions. The cell contents of forage legumes include sucrose, galactans, and starch (Van Soest, 1994). Therefore, the higher abundance of proteins and amino acids may have favoured a greater intensity of heat-induced non-enzymatic reactions, involving these carbohydrates. Hemicellulose and soluble sugars (e.g., sucrose) are among the carbohydrates most susceptible to thermal damage. Upon degradation, their monomeric units may serve as substrates for non-enzymatic reactions (Van Soest and Mason, 1991). However, non-enzymatic reactions comprise multiple stages and involve the formation of diverse intermediate compounds. One artefact derived from sugar monomers is furfural, which is essentially indigestible. However, due to its solubility in the detergent solutions, it is not quantified in fibre residues (Van Soest, 1994). Moreover, although SPN and nNDIN concentrations were reduced by heat application, the analytical procedures employed in our study do not allow a detailed qualitative characterisation of these compounds. Therefore, it is plausible that a fraction of the cell-content protein remaining after heat drying corresponds to indigestible artefacts that are not necessarily insoluble in detergent solutions. This hypothesis may help explain the observed reduction in IVDMD, while also accounting for the absence of detectable changes in IVNDFD in fresh legumes.

Overall, the results clearly indicate that the magnitude of chemical alterations in the samples was proportional to the intensity of heat applied during partial dehydration. This pattern helps explain the lower level of modification observed with forced-air oven drying, as this method operates at lower temperatures compared with the other heat-based procedures. Theoretically, coupling an initial microwave heating step with subsequent forced-air oven drying could interrupt some detrimental metabolic processes in fresh forages (e.g., respiration, indigenous enzyme activities, protein degradation). This strategy would rely on a rapid temperature increase to inactivate biological process, thereby avoiding or attenuating undesirable modifications that might occur under the slow heating curve characteristic of forced-air ovens (Hofman and Belgium, 1965; Karn, 1991; Deinum and Maassen, 1994; Pelletier et al., 2010). However, no benefit was observed with this combined approach. On the contrary, the results suggest that the negative effects observed with exclusive microwave drying were also present when microwave heating was used as an initial heating step.

Microwave-assisted partial dehydration has been proposed as a strategy to reduce the time required for forced-air oven drying (Joardder et al., 2017). However, limited heating uniformity and difficulties in temperature control during the dehydration process represent important constraints to its practical application (Jiang et al., 2017; Brodie et al., 2019). In our study, we verified that microwave drying resulted in final temperatures exceeding 80°C, thereby increasing the likelihood of non-enzymatic reactions, which

Fig. 5. Examples of blunders caused by the use of an air-fryer equipment during the partial dehydration process of forage samples.

are known to intensify at temperatures above 60°C (Van Soest, 1965).

The operating principle of the air fryer resembles that of a forced-air oven, as drying is achieved through the circulation of heated air, promoting relatively uniform and rapid heating of the sample (Lee and Lee, 2024). However, the airflow generated by the air fryer is typically more intense than of conventional laboratory ovens. When finely disintegrated samples are subjected to this process, particles my become suspended and dispersed within the equipment (Fig. 5), compromising complete material recovery and introducing potential analytical errors due to incomplete mass recovery (Association of American Feed Control Officials – AAFCO., 2018). In addition, the temperature recommended for partial dehydration using an air fryer (121°C; Severson, 2019) may further intensify non-enzymatic reactions, as previously discussed. An additional concern relates to the internal configuration of the equipment, particularly the heating element, which is positioned in the upper portion and separated from the sample only by a metal grid. The strong upward airflow can cause fine particles to become lodged against this grid, where direct exposure to the heating element may result in localised carbonisation of the material (Fig. 5).

In summary, despite the various problems associated with the use of heat during partial dehydration, these were less pronounced when forced-air oven drying was applied. Although this method entails practical limitations due to the longer time required for sample drying, it resulted in smaller differences in the measured chemical characteristics when compared with freeze-drying as a reference. Clearly, freeze-drying cannot yet be considered a widely adoptable method for partial dehydration, given the economic and logistical barriers associated with implementing lyophilisation systems in routine laboratory settings. Therefore, considering the comparatively smaller differences in the measured chemical characteristics, forced-air oven drying emerges as the most viable option when the objective is to maximise preservation of the measured characteristics of the forage samples under study in ruminant nutrition evaluations.

Alternative heat-based methods, such as microwave drying and air-fryer drying, should not be used for the partial dehydration of high-moisture samples intended for ruminant nutrition evaluation because their higher operating temperatures, relative to freeze-drying and forced-air oven drying, can lead to greater bias in estimates of chemical composition and digestibility. However, when used solely to estimate DM content, the associated bias appears to be relatively small compared with the differences observed in the measured chemical characteristics of the samples. Therefore, despite the limitations discussed above, these methods may still have practical applications for consultants and farmers who require rapid, approximate estimates of DM content, such as for adjusting feedlot diets or estimating forage mass availability in grazing systems.

5. Conclusions

The application of heat during partial dehydration results in differences in the measured chemical characteristics of high-moisture forage samples. These differences are more pronounced in fresh forages than in silages and tend to increase with the intensity of heat applied to the sample. Among the heat-based methods evaluated, forced-air oven drying results in the smallest differences in the measured chemical characteristics and is therefore recommended when freeze-drying is not feasible.

Ethics declaration

This study was conducted in accordance with the following guidelines for animal welfare and/or reporting: CONCEA (Brazil). This study was approved by the Ethics Committee on the Use of Production Animals of the Universidade Federal de Viçosa. (Approval No. 066/2023)

CRediT authorship contribution statement

Nicole S.A. Lima: Writing – original draft, Investigation. **Daiana F. Quirino:** Writing – review & editing, Investigation. **Marcia O. Franco:** Writing – review & editing. **Edenio Detmann:** Supervision, Project administration, Funding acquisition, Formal analysis, Conceptualization, Writing – review & editing.

Declaration of Generative AI and AI-assisted technologies in the writing process

During the preparation of this work, the authors used ChatGPT-5.5 (OpenAI) to improve the language and grammar of the text and to produce the graphical abstract. After using this tool, the authors reviewed and edited the content as needed and take full responsibility for the content of the published article.

Declaration of Competing Interest

The authors report no declarations of competing interest.

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