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ORIGINAL ARTICLE

Spray drying egg using either maltodextrin or nopal mucilage as stabilizer agents

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Abstract In this work, a comparative study between spray drying (SD) of fresh egg by either maltodextrin (MD) or nopal-mucilage (MN) as stabilizing vectors was made. The powders obtained were characterized for drying performance, moisture content, chemical proximate analysis, thermal analysis (TGA), chemical composition (FTIR), microscopy (SEM) and rheology (viscoelasticity and steady state simple shear viscosity). Infrared analysis showed that MN has the effect of a thickening agent rather than an encapsulating one. Results indicated that SD egg with MN produced a high thermal and mechanical stable product and rendered the highest drying performance, producing a more uniform and defined sphere-shaped morphology in comparison to egg SD either alone and with MD.

Keywords Spray drying · Egg · Maltodextrin · Nopal mucilage · Stabilizer agents

List of symbols

a_w	Water activity
SD	Spray drying
MD	Maltodextrin, MD10
MN	Nopal (*Opuntia ficus-indica*) mucilage
Hv	Egg
RS	Drying performance
Hv-SD	Spray-dried egg powders
60Hv-40MD	SD-eggs with maltodextrins (relation 40/60)
60Hv-40MN	SD-egg with nopal mucilage (relation 40/60)
TGA	Thermogravimetric analysis
FTIR	Fourier transform infrared spectroscopy
SEM	Scanning electron microscopy
G'	Elastic modulus
G''	Viscous modulus
ω	Frequency
η	Viscosity

Introduction

Egg is a product with a high nutrient content and high bioavailability of iron and zinc. It is also an important source of essential saturated and unsaturated fatty acids (i.e., oleic acid, linoleic acid), in addition to minerals and vitamins required in the daily intake (Watkins 1995). It is widely consumed as both food and a food additive. Mexico is the 5th largest egg producer with 2360 ton year^{-1} (FAOSTAT data 2015), with a production that represents an important segment of the global food industry. Currently, processing technologies allow multiple presentations of the product, in either liquid, frozen, separated egg yolks, or in dried form (full, white or egg yolk). Recently, dried egg products have gained popularity (Koç et al. 2011). Prasad et al. (2004), suggesting that the biscuits with

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spray-dried (SD) egg in powder form may be a good fresh egg substitute for consumption in remote areas or where transportation is scarce. Egg storage (natural or processed) is problematic due to progressive deterioration caused by its high-water activity ($a_w \approx 1$) at storage conditions. Degradation of egg products depends on the type of egg and additives such as flavorings, colorings, and anti-oxidizers used in products stored for long periods (Campbell et al. 1979). SD-egg products sometimes present negative properties such as discoloration, loss of flavor and degradation of the nutrients. Food dehydration by open cycle spray-drying (SD) is one of the most widely-used process in industry, as it is an economic and effective method for protecting temperature sensitive materials (Risen and Reineccius 1988; I Ré 1998; Marreto et al. 2006; Vaidya et al. 2006). In the SD technique, the exposure of the product to high temperatures (> 150 °C) is relative short (< 5 s) and the product is sprayed into small water drops which protect the core product with a continuously evaporating water layer. In this regard, SD is an appropriate drying technique for temperature sensitive materials (Deasy 1984). Spray drying allows the preparation of stable and functional powder products (Koç et al. 2010; Liu and Liu 2009; Re 2006, Sagar and Suresh-Kumar 2010) improving mechanical stability by extending the shelf life of dried products and maintaining the nutritional properties of fresh egg (Yáñez et al. 2002). For the preparation of egg powder, the carbohydrates contained in the egg, mainly glucose (~ 98%) (Mine 1997) should be protected. Combined SD with high temperature produce degradation of glucose and reduction of proteins quality (i.e., albumin) of reconstituted eggs, in addition to loss of the nutritional quality of final products as they are rehydrated and incorporated into foodstuffs. Stabilizing agents for spray-drying are then necessary, which include mixtures of maltodextrin and starches. Maltodextrins are composed of polysaccharides and oligosaccharides of various molecular weights. They are water-soluble and provide consistency to solutions as a gelling and thickener agent. They also prevent crystallizations and promote dispersion. An interesting and promising alternative to maltodextrins is the use of nopal mucilage (*Opuntia ficus indica*). Nopal mucilage possesses similar properties to those of maltodextrins as emulsifier, thickener and efficient edible coating agent (Koocheki et al. 2009). It has potential benefits as additive in the food industry to enhance the shelf life of food products, as reported elsewhere (Medina-Torres et al. 2000, 2013; Del-Valle et al. 2005). Stable, low hygroscopic spray-dried products using nopal mucilage have been obtained, possessing sphere morphology with collapsed fragments and agglomerate structures (León-Martínez et al. 2010). Previous studies have revealed that the nopal mucilage (*Opuntia ficus indica*) is a complex mixture of polysaccharides (hetero-polysaccharide) with high average molecular weight (2.3×10^4 g mol^{-1}) and interesting physico-mechanical properties, such as the ability to form ions in water solutions, with stable flow properties (Medina-Torres et al. 2000, 2013).

In this study, maltodextrins and nopal mucilage are used to stabilize egg powders in the spray-drying operation. The powders obtained are characterized to disclose their macro and micro structural behavior, including chemical composition (proximate analysis). One of the objectives of this work is the analysis of potential benefits of these materials as stabilizers of the egg products, including information provided by previous works on process conditions of SD for similar food products (Medina-Torres et al. 2013, Santiago-Adame et al. 2015). This study includes the development of powder products with acceptable organoleptic and nutritional properties which need to be preserved during transportation and shelf life. Besides representing an alternative process to fresh eggs, egg powder can also be used as additive to prepare functional foods.

Materials and methods

Materials are labelled as follows:

Maltodextrin (MD), nopal-mucilage (MN), egg (Hv), spray drying process (SD), spray dried egg powders (Hv-SD), spray dried egg powders with maltodextrines (relation: 60Hv-40MD), and spray dried egg powders with nopal mucilage (relation: 60Hv-40MN).

The maltodextrin selected here is food-grade with 10DE of dextrinization (NIFRA Commercial S. A., Mexico) and possesses high stability according to previous studies reported in the literature (Santiago-Adame et al. 2015; Saavedra-Leos et al. 2015). Reagent-grade ammonium hydroxide from Sigma-Aldrich (NH_4OH, CAS: 1336-21-6) was used to stabilize the pH level. Fresh white eggs from poultry farms were stored in a cool and dark environment for 2 days. 6 months old mucilage was extracted from cladodes of *Opuntia ficus indica* from irrigated soil. Harvest time and size-controlled conditions are described by Medina-Torres et al. (2000, 2013). Nopal cladodes were collected from an area near Mexico City (Milpa Alta).

Methods

Extraction of nopal (Opuntia ficus indica) mucilage

The extraction procedure is described in Medina-Torres et al. 2000. Nopal cladodes were macerated (500 mL deionized water kg^{-1} of nopal) by using a Hamilton Beach juice extractor model Health-Smart with 350 W motor and two speeds for easy mucilage removal. The extracted material was filtered in a nylon mesh (149 microns) and centrifuged at 11,000 g (Dinac Clay, Becton–Dickinson, NJ, US). The mucilage was stored at 4 °C for subsequent use.

Egg solution pretreatment

The content of the eggs (mainly yolk and egg white, except the shell) were placed into a container and mixed under stirring with a propeller at 100 rpm until visual homogeneity of the sample. The solution was prepared by incorporating maltodextrin or nopal mucilage at 5 wt% in both cases, before the spray-drying process.

Spray drying (SD)

The spray drying process was carried out in a dryer (Niro–atomizer, Production Minor Spray Dryer, Niro Inc., Denmark) equipped with a rotary atomizer (TS-Menor, M02/A). Heating of the drying air was provided by electrical resistors and the feed flow was controlled with a peristaltic pump. Distilled water at room temperature (25–28 °C) was used in the initial feed to stabilize the process. The dispersion of pasteurized whole egg/mucilage or maltodextrin was accomplished by vertical atomization under co-current flow in the drying chamber. The feed was constant (1.4 L h^{-1}) with atomization speed of 20,000 rpm and pressure of 3 bar. During the dispersion process, temperature was kept at 10 °C. The inlet and outlet temperatures were 200 °C (± 5 °C) and 85 °C (± 5 °C) respectively, and the drying adiabatic process temperature was set at 77 °C. The powder was collected in a glass container under the cyclone action. Finally, the sample was vacuum-packed prior to analysis.

Drying performance (RS)

The RS is the ratio of the capsules mass obtained at the end of the drying process to the initial mass of the mixture (Eq. 1):

$$\%RS = \left[\frac{dried\ product\ weight\ (\mathrm{g})}{total\ mass\ of\ the\ initial\ mixture\ (\mathrm{g})}\right] \times 100 \quad (1)$$

Moisture content in spray-dried powders

The moisture content in the three samples: spray-dried egg powders (Hv-SD), SD-eggs with maltodextrins (relation 60Hv-40MD) and SD-egg with nopal mucilage (relation 60Hv-40MN) was determined by the AOAC 92510 method (2 g samples were dried in a hot-air oven at 103 °C for 1 h). The moisture loss was measured by weight difference in the sample after the drying process (AOAC International 2000).

Chemical proximate analysis of the products

Proximate analysis of the SD-egg samples (Hv-SD), SD-egg with maltodextrins (60Hv-40MD), and SD-egg with nopal mucilage (60Hv-40MN) respectively, was performed following the AOAC (AOAC 1995) procedures, based on 100 g of dry samples in triplicated sets (n = 3).

Thermogravimetric analysis (TGA)

Thermogravimetric analysis (TGA) was carried out in a TA-Instrument Q5000 IR Discovery. Characterization of the raw SD-materials reveals the effect of mucilage addition (MN) or maltodextrin (MD) on the egg, and the presence of proteins (i.e., ovalbumin) in the mucilage or in maltodextrin mixtures. Percentage variation of each ingredient by the thermal scan is detected.

Infrared spectrometry analysis (FTIR)

The FTIR analysis of raw materials and products after spray drying was performed on egg powders as a reference (Hv-SD), with maltodextrins (relation 60Hv-40MD) and SD-egg with nopal mucilage (relation 60Hv-40MN). The FTIR tests were performed with a Nicolet 6700 FTIR diamond tip (Thermo Fisher Scientific, USA) using the KBr method with 100 scans with resolution of 1 cm^{-1} from 4000 to 400 cm^{-1}. Similar conditions have been used elsewhere in a system of micro-particles with maltodextrin (Santiago-Adame et al. 2015).

Scanning electron microscopy (SEM)

Proximate analysis of SD-egg samples (Hv-SD), SD-egg with maltodextrins (60Hv-40MD) and SD-egg with nopal mucilage (60Hv-40MN) were performed. The sample was placed with copper parts in a conductive tape coated with gold at 10 mbar for 90 s (model Desk II, Denton Vacuum, NJ, USA), and examined in a scanning electron microscope (JEOL Mod. JSM6300 Jeol, Japan) at a voltage of 20 kV and 1000× magnification. Selected micrographs are representative of each sample.

Rheological behavior of egg spray-dried powder

The rheological characterization was carried out in the SD-egg sample (that shows the best properties) with nopal mucilage (relation 60Hv-40MN) in an emulsion at 6% (w/w) with 4.5% (w/w) vinegar, 0.6% (w/w) salt, 11.4 mL water and 77.5% (w/w) cooking oil in a final volume of 20 mL. Rheological tests were performed under simple shear and oscillatory flow using a controlled stress rheometer (AR-G2, TA Instruments) with a cone and plate geometry at a constant temperature of 25 °C. Viscosity [$\eta(\gamma)$] was measured under steady simple shear flow in a range from 0.1 to 200 s^{-1}. Oscillatory tests in the linear viscoelastic regime were performed to analyze the viscoelastic properties of the sample in the range of 0.1–100 rad s^{-1}. Experimental data were analyzed using the TA V.5.7.0 Rheology Advantage Data Analysis (TA Instrument Ltd., Crawley, UK) software.

Statistical analysis

Three replications were performed for each test. Data were analyzed by ANOVA with statistical significance set at $p < 0.05$.

Results and discussion

Results show clearly the effect of the SD on farm-egg (Hv-SD) using either maltodextrin (MD) or nopal mucilage (MN) as thickening agents. The spray-dried sample with nopal mucilage (60Hv-40MN) showed the highest yield with a drying performance of 58.49%, while in 60Hv-40MD the drying performance was only 56.27%. SD conditions employed in this study were similar to those reported in previous works dealing with the properties of SD-egg (Koç et al. 2011). The moisture content of the samples was 2.56 and 2.85 in the 60Hv-40MN and 60Hv-40MD systems, respectively. These values were in the range of those reported by Koç et al. (2011). During the preparation of raw materials, factors such as pH and °Brix were evaluated (pH was measured with a Thermo Scientific Orion pH Meter and °Brix were measured in a refractometer model RF-10-CAT). The egg mixture (before treatment alkalization) had a pH in the range of 7.2–7.6, and °Brix of 23.5–24.5.

Proximate chemical analysis of Hv-SD, Hv-MD and Hv-MN

In Table 1, results of the proximate chemical analysis of the SD-egg (Hv-SD), SD-egg with maltodextrins (60Hv-40MD), and SD-egg with nopal mucilage (60Hv-40MN) are shown. Spray drying is a technology that produces functional changes (chemical or physical) in the resulting powders. The moisture content in the samples ranged from 2.15 to 14.69%. The effect of the thickening material on moisture content (as compared with Hv-SD with a moisture content of 11.49%) is the reduction of 81.3% (2.15% moisture content) for the case of MN (60Hv-40MN). In the case of SD-egg with maltodextrins (60Hv-40MD), an increase of 21.7% (14.69% moisture content) was observed.

Protein content is very important due to its nutritional contribution. Fresh egg contains 9.7–12.6 wt% crude protein (USDA 2016) but in egg powder values up to 48.05, 31.88 g/100 g have been reported (USDA 2016, Ignário and Lannes 2007) which is a consequence of the nutrients concentration produced by the removal of water during the drying process. One of the most important effects of mucilage as a thickener material is the protection given to the egg proteins preserving more than 6 wt%. In this regard, the lipid content of the powder product changes from 9.08 to 15.32 wt%, as compared to that of the sole egg powder (USDA 2016). These changes in the lipid content in 60Hv-40MN may be associated with physical interactions involving the thickening agent (nopal mucilage) which provides a protective effect conferred by mucilaginous materials.

Thermogravimetric analysis (TGA)

The thermal stability of the materials was analyzed by measuring the thermal degradation of powders obtained by SD with MD or MN. Figure 1 shows the thermograms within a range from 0 to 300 °C. The Hv-SD sample shows a rapid degradation at 50 °C with a loss of 5 wt% of the sample. At 100 °C 20 wt% weight loss is shown, increasing up to 50 wt% at 150 °C. However, when either MD or MN are employed as stabilizers in the SD process, this degradation is efficiently avoided. The weight loss is drastically reduced up to 290 °C respect to the dried egg. However, at 290 °C an important change of the thermal behavior of the powders is observed, as maltodextrins (MD) show a higher degradation than that of nopal mucilage (MN), see Fig. 1b, c. This can be attributed to degradation of biopolymers in the egg mixture occurring at high temperatures (> 90 °C). Thus, the SD-egg powder with nopal mucilage showed more stability in the high temperature range, which was an indication of the protection provided by the mucilaginous material to the egg (Fig. 1c). Furthermore, according to the thermal degradation process of macromolecules, the increase in molecular mobility caused bond breaking, and thermal stability was related to degradation. Protection to the proteins provided by the mucilage prevents the partial denature action by heating or shearing (Kato et al. 1981).

Infrared analysis (FTIR)

This analysis provides information about the chemical composition of the egg (without adding thickening agents) and about interactions with the thickening agents in the SD

Table 1 Chemical composition of the samples: Hv-SD, 60Hv-40MD and 60Hv-40MN

Chemical constituents (%)	Samples		
	Hv-SD	60Hv-40MD	60Hv-40MN
Crude protein content	35.02 (± 0.58)	25.28 (± 1.24)	31.98 (± 0.46)
Crude lipid content	33.62 (± 1.13)	28.58 (1.16)	34.82 (± 1.03)
Crude fiber content	0	0	0
Nitrogen Free Extract	28.87 (± 1.0)	43.48 (± 1.1)	27.79 (± 1.1)
Ash content	2.49 (± 0.25)	2.66 (± 0.31)	3.26 (± 0.13)

Contained in 100 g of dry sample (Based on A.O.A.C procedures)

process: maltodextrin or nopal mucilage. Figure 2a, b show the spectra of the SD-egg sample (Hv-SD) with either maltodextrins (Fig. 2b), or with nopal mucilage (Fig. 2a). In Fig. 2a, the spectra of the egg alone and SD-egg with nopal mucilage are very similar, thus providing evidence that the mucilage is not an encapsulating agent but a thickener that stabilizes the egg protein by inducing a thermally-stable interconnected network, as shown in the TGA analysis. However, when MD is used, the spectra are not as similar as in the case of MN, which reveals that the SD-egg treated with MD is not as stable as that treated with MN and also is an evidence of the higher degradation suffered by the sample with maltodextrine during the spray drying process.

Microparticle morphology of Hv-MD and Hv-MN

In Fig. 3a–d, micrographs of MD alone (Fig. 3a), SD-egg with maltodextrins (Fig. 3b), MN alone (Fig. 3c) and SD-egg with nopal mucilage (Fig. 3d) are shown. Figure 3a presents micro-particles with deflated balloon-like morphology, associated with the process of rapid evaporation of water during spray drying. Figure 3b shows the egg dried with MD with similar morphology, but with contracted particles, evidencing degradation. In contrast, the morphologies with MN (Fig. 3c, d) present a more stable and uniform conformation of more defined spheres than that with MD (Fig. 3a, b) with apparently modal distribution, thus representing a more mechanically and thermally stable system. These results are similar to those reported by Ma et al. (2013) and Koç et al. (2011) for white egg powder dried at 165–205 °C, where amorphous particles appear larger.

Rheology of Hv-MD and Hv-MN powder rehydrated

The rheology of SD-egg (Hv-SD), SD-egg with maltodextrins (60Hv-40MD) and SD-egg with nopal mucilage (60Hv-40MN) is presented in this section. Figure 4 shows the mechanical spectrum in the linear viscoelastic regime of the egg powders obtained by SD (Hv-SD) rehydrated at 6% (w/v). Results are common to a gel-like system; both moduli almost independent of the applied frequency with a dominant elastic modulus ($G' > G''$) in the entire frequency range. Results are similar to those reported for fresh egg elsewhere (USDA 2016; Ignário and Lannes 2007; Miranda et al. 2002; Moros et al. 2002). These measurements reveal the effects of the high and low-density lipoproteins of the egg; the latter (LDL) are found mainly in the yolk and contain ~ 95% of total lipids. Additionally, it contains fractions of α and β-lipoproteins, as well as γ-livetins (Kiosseoglou and Sherman 1983). Moreover, the egg proteins are responsible for the structural arrangements in the egg dispersions with 6% (w/v).

The samples of SD-egg with either MN or MD did not show the gel like behavior of the sole SD-egg sample, attributed to the flow properties of MN and MD, as previously reported (Medina-Torres et al. 2000, 2013). This is the reason by which these systems where analyzed under simple shear flow tests. Figure 5 shows the flow curves of the rehydrated SD-egg powder samples with either MN or MD under simple shear flow. A typical shear-thinning and low viscosity behavior is depicted, which is beneficial to the processing of these systems since low viscosity systems are easier to handle than gels. The shear-thinning viscosity arises due to the presence of the associative structures among protein–polysaccharides (MN or MD) which are likely to form complexes, as reported in the literature (Orozco et al. 2007; Patino and Pilosof 2011; Medina-Torres et al. 2000; DeKruif and Tuinier 2001; Pal 1995; Franco et al. 1997, 1998; Moros et al. 2002). These associative structures may be easily aligned along the flow direction. It is necessary to mention that the spray-drying operation with fresh egg yolk has presented complications in the spray drier chamber. This problem is associated with the large amount of agglomerates and gel formation which interferes with the drying process. The drying yield is minimized because agglomeration of semi-dried droplets is fast and their adhesion to the walls induces the formation of

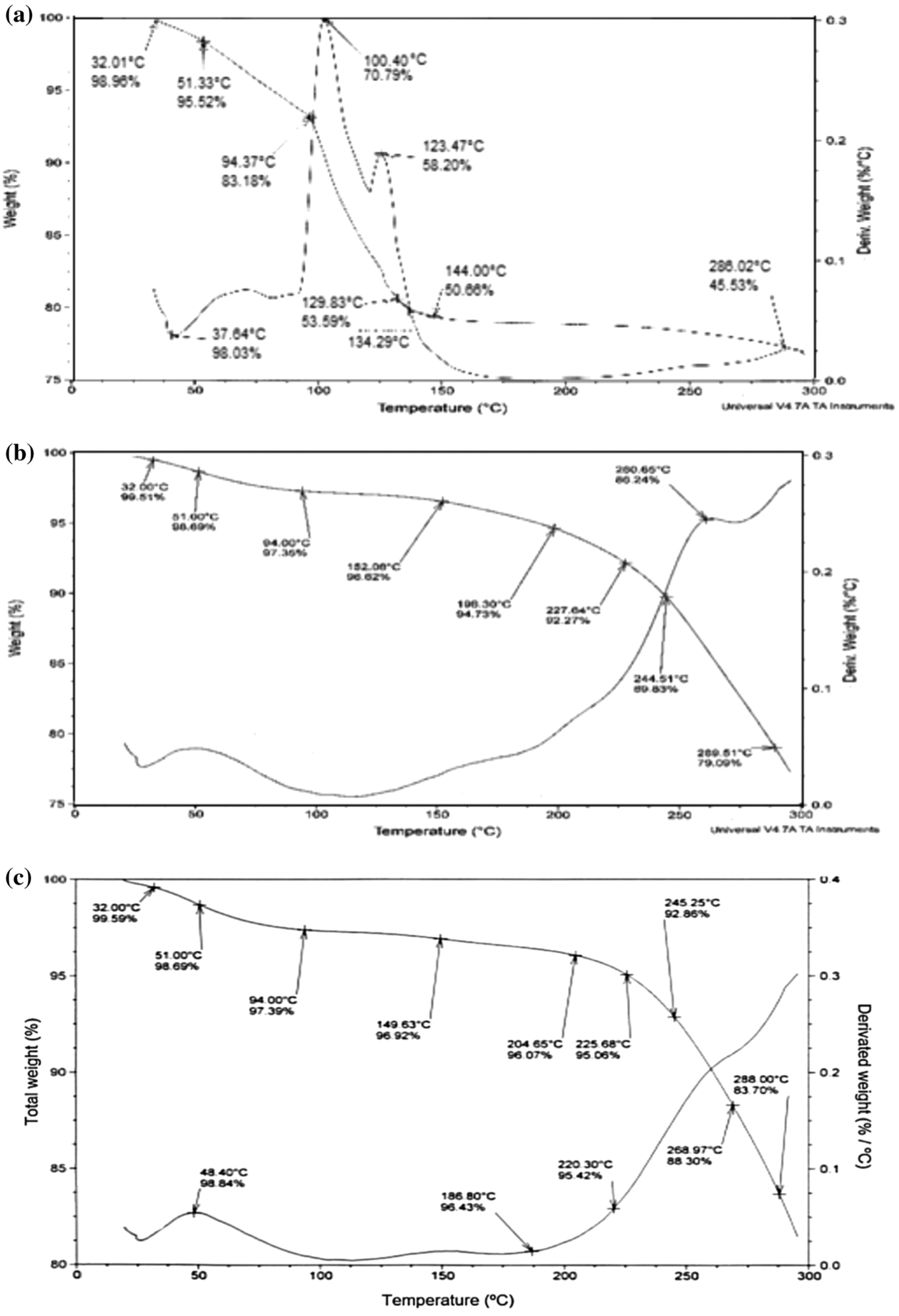

Fig. 1 TGA results for the three systems: **a** Hv-SD, **b** 60Hv-40MD, **c** 60Hv-40MN. Note: Temperatures indicating along the curves are for reference only

a thick layer during the drying process. The growing of these agglomerates is due to interactions between the protein molecules and the wall material, which can be overcome by dilution of the sample in the range of 25–50 wt% with water (DeKruif and Tuinier 2001; Ignário and Lannes 2007).

The sample 60Hv-40MD processed at the same temperature under SD displays lower viscosity than that of the sample 60Hv-40MN, attributed to thermal degradation (Moros et al. 2002) which shows consistency with the TGA and FTIR analyses.

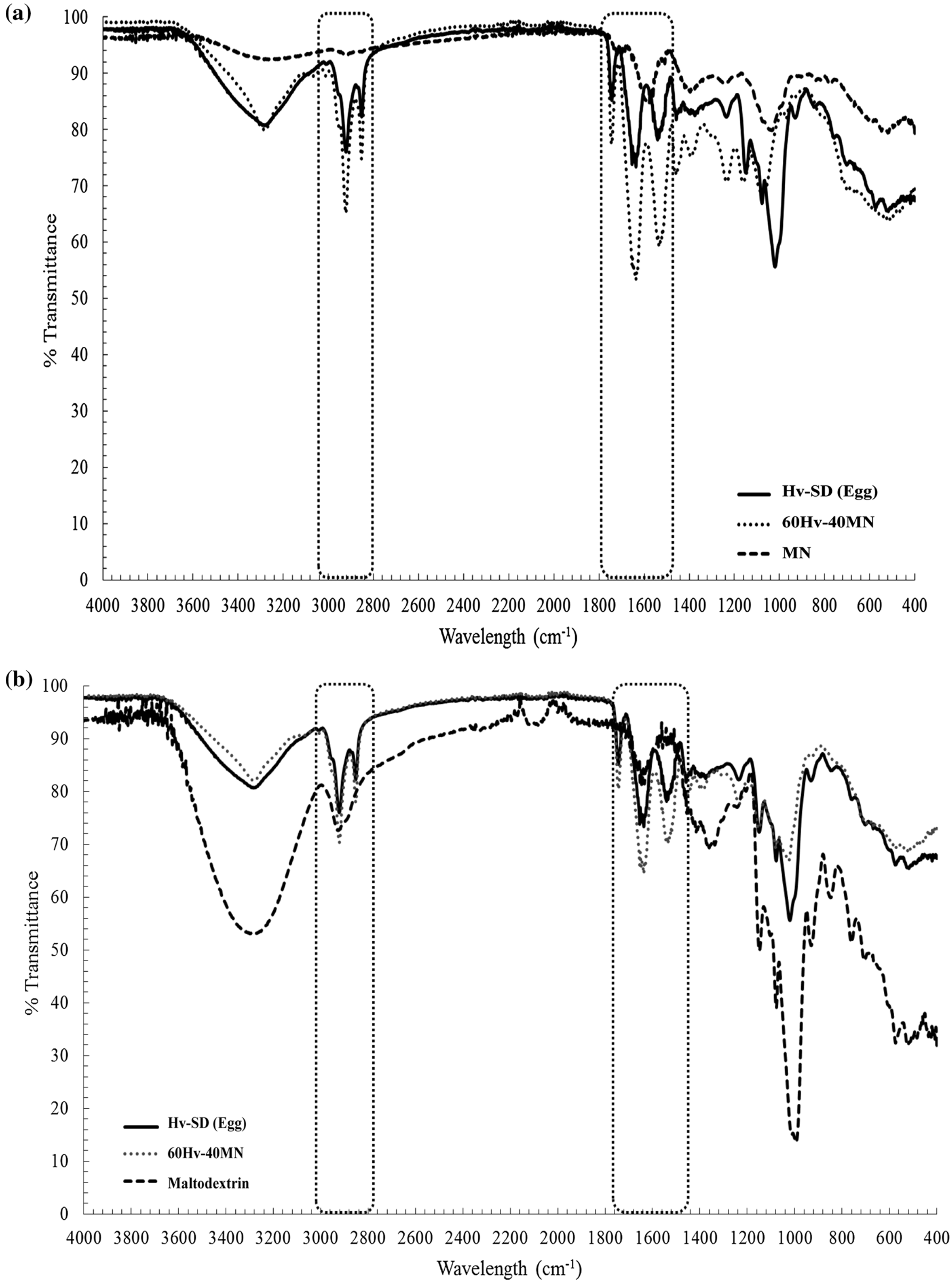

Fig. 2 Infrared spectrum: **a** whole egg SD with nopal mucilage (60Hv-40MN), and **b** SD-egg with maltodextrins (60Hv-40MD)

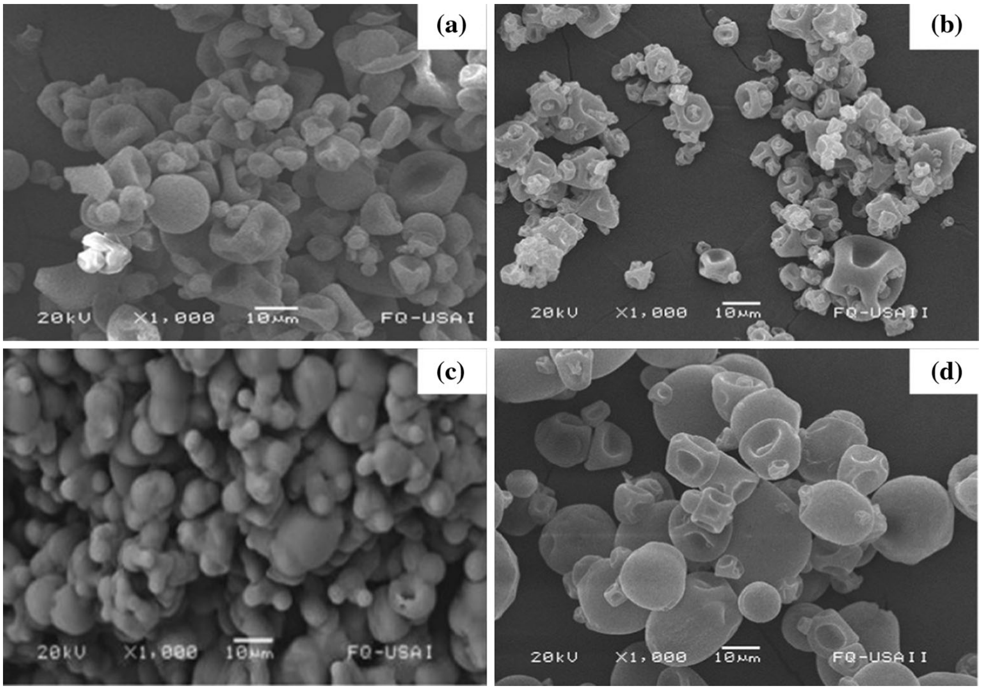

Fig. 3 Electronic Microscopy of: **a** MD, **b** 60 Hv-40MD, **c** MN and **d** 60Hv-40MN. ×1000

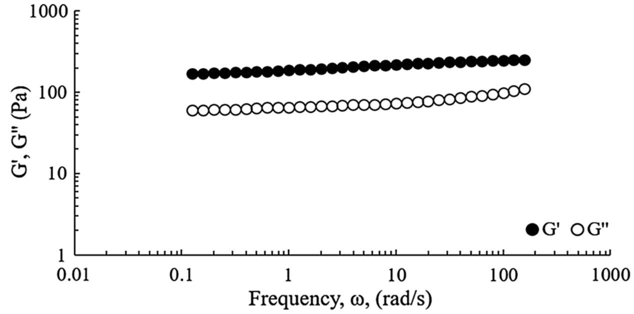

Fig. 4 Dynamic mechanical spectrum of the storage (G′) and loss (G″) modulus as a function of frequency. The emulsion of egg at 6 wt%

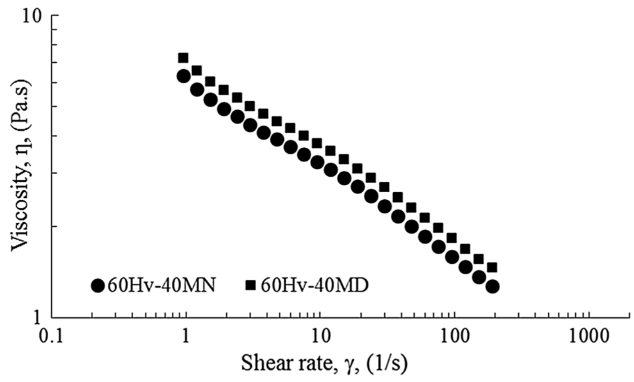

Fig. 5 Viscosity as a function of shear rate for 60 Hv-40MN and 60Hv-40MD at 6% wt%

Conclusion

Egg was spray-dried using stabilizing and thickening agents (Maltodextrin, MD or Nopal Mucilage, MN) to produce powdered egg. MN provided high thermal stability to the egg with the highest drying performance as compared to that of the MD sample. Infrared analysis evidenced that the nopal mucilage acts as a thickener agent and not as an encapsulating one.

The particle microstructure produced by SD revealed contracted-deflated balloon-like particle morphology in MD samples, associated with degradation by temperature, while for the case of MN, particles show uniform and defined sphere-shaped morphology.

Degradation of MD sample was confirmed by rheological measurements of rehydrated samples, where the MN system exhibited higher viscosity than that observed in the MD system.

Finally, egg powder can be stored for long times and possessed improved mechanical stability during flow operations.

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