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Maize bran gum: Extraction, characterization and functional properties

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Abstract

A water-soluble maize bran gum (MBG) was alkali-extracted under mild conditions from maize bran. MBG presented an arabinoxylan (AX) content of 74% (w/w), a ferulic acid content of 0.34 μg/mg MBG, an *A*/*X* ratio of 0.85 and an intrinsic viscosity of 208 ml/g. Gelling and emulsifying capabilities of MBG at different concentrations were investigated. Gels were obtained from this MBG by laccase covalent cross-linking of ferulic acid leading to the formation of diferulic (di-FA) and triferulic (tri-FA) acid. Gel hardness increased from 0.32 to 0.81 N as the MBG concentration changed from 1 to 2% (w/v) while the di-FA and tri-FA contents remained constant (0.030 and 0.015 μg/mg MBG, respectively). The emulsion stability index (ESI) of oil-in-water containing different MBG concentrations was also investigated. ESI ranged from 0.01 to 0.20 as the MBG content in the mixture increased from 0% to 1% (w/v).

Keywords: Maize bran gum; Arabinoxylan; Ferulic acid; Gels; Emulsion

1. Introduction

Maize bran is a by-product of the commercial maize dry milling process. Because of the increase in maize white flour production for the tortilla industry in Mexico, maize bran residue is becoming a potential source of added-value biomolecules as hydrocolloids for the food industry. Maize bran contains heteroxylans (approximately 50%), cellulose (approximately 20%) and phenolic acids (approximately 4%, mainly ferulic and diferulic acid) (Saulnier, Vigouroux, & Thibault, 1995a). Starch (9–23%), proteins (10–13%), lipids (2–3%) and ash (2%) are also present in maize bran (Hespell, 1998). The heteroxylans portion of maize bran can be extracted with alkaline (Saulnier, Marot, Chanliaud, & Thibault, 1995b; Whistler, 1993) or acid solutions (Saulnier et al., 1995a) to produce water-soluble maize bran gum (MBG).

MBG has been reported to be composed of D-xylose (48–54%), L-arabinose (33–35%), galactose (5–11%) and D-glucuronic acid (3–6%) (Suguwara, Suzuki, Totsuka, Takeuchi, & Ueki, 1994; Whistler & BeMiller, 1956). MBG gum structure is highly branched with a β-(1 → 4)-xylopyranose backbone and α-L-arabinofuranose residues as side chains on O3 and O2 and O3 (Saulnier et al., 1995b). Most of the D-glucuronic acid residues are linked to the O-2 position of xylose residues of the main xylan backbone (Montgomery & Smith, 1957). Galactose and some xylose residues are attached to the arabinofuranosyl branches (Whistler & Corbett, 1955). Ferulic acid (FA) and dehydrodimers of ferulic acid (di-FA) (5–5′, 8–5′, 8-*O*-4′ structures) have been detected in saponified extracts of maize bran (Saulnier et al., 1999). Di-FA structures were first identified in grass cell walls by Ralph, Quideau, Grabber, and Hatfield (1994), they may serve to cross-link cell-wall polymers and contribute to the mesh-like network of the cell wall (Iiyama, Lam, & Stone, 1994). Di-FA cross-linking structures have been reported to be formed through oxidative gelation of

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feruloylated sugar beet pectins (Micard, Grabber, Ralph, Renard, & Thibault, 1997), feruloylated wheat arabinoxylans (Figueroa-Espinoza & Rouau, 1998) and feruloylated maize heteroxylans (Lapierre, Pollet, Ralet, & Saulnier, 2001; Ng, Greenshields, & Waldron, 1997). The formation of a tri-FA structure in laccase-induced wheat arabinoxylan gels has been recently reported by Carvajal-Millán, Guigliarelli, Belle, Rouau, and Micard (2005a). These di-FA and tri-FA cross-linked polysaccharide gels have an interesting technological potential as they are mostly stabilized by covalent linkages which make them stable upon heating and exhibit no syneresis after long storage times (Izydorczyk & Biliaderis, 1995; Vansteenkiste, Babot, Rouau, & Micard, 2004).

MBG could be used as an adhesive, thickener, stabilizer (Wolf, MacMasters, Cannon, Rosewell, & Rist, 1953), film former, emulsifier (Whistler, 1993; Woo, 2001) and a gel former (Lapierre et al., 2001; Ng et al., 1997). Nevertheless, the detailed evaluation of MBG functional properties has not been accomplished. Peroxidase/H_2O_2-mediated MBG gelation has been reported by Ng et al. (1997) and Lapierre et al. (2001) but laccase/O_2 oxidative cross-linking of MBG has not been reported before.

In this study, laccase-induced gelation of a MBG extracted under mild alkaline conditions has been investigated and the gel hardness and covalent cross-linking content have been determinated. In addition, the emulsifying capacity of MBG was evaluated using Mexican oregano oil. Mexican oregano (*Lippia berlandieri* Schauer) belongs to the Verbenaceae family and is quite distinct from its European counterparts. Mexican oregano has a much stronger and robust flavour (Turgut & Silva, 2005). Thymol and carvacrol are the two major compounds in the essential oil fraction of Mexican oregano. These compounds are of special interest due to their antioxidant and antimicrobial properties. Some of the properties of Mexican oregano oil are being currently studied due to the growing interest for substituting synthetic additives commonly found in foods (Arcila-Lozano, Loarca-Piña, Lecona-Uribe, & González de Mejía, 2004). These characteristics are of interest for the food industry as they can improve the safety and stability of foods. Because of the low oregano oil concentration used (0.1–0.2% v/v), oregano oil-in-water emulsions need to be prepared. Therefore, in this study, MBG was evaluated as a natural emulsifier for the emulsification of Mexican oregano oil.

2. Experimental

2.1. Materials and methods

Maize bran was kindly provided by a commercial milling industry in Northern Mexico. Laccase (benzenediol:oxygen oxidoreductase, E.C.1.10.3.2) from *Trametes versicolor*, sodium hydroxide, citric acid, sodium phosphate dibasic, chlorydric acid and sulfuric acid were purchased from Sigma Chemical Co. (St. Louis, MO, USA).

2.2. MBG extraction

Maize bran was milled down to 0.84 mm particle size using a M20 Universal Mill (IKA®, Werke Staufen, Germany). Maize bran (500 g) was suspended in ethanol (2500 ml) and agitated on a rotatory shaker (100 rpm) for 12 h at 25 °C to remove lipophilic components. The ethanol treated bran was then filtered through 2.7 μm pore size filters (Millipore) and subjected to starch gelatinization and enzymes inactivation (boiling for 30 min in 3500 ml of water). After boiling, maize bran was recovered by filtration through 2.7 μm pore size filters (Millipore) and treated with 2500 ml of 0.5 N NaOH solution at 25 °C in darkness for 8 h under shake (100 rpm). Residual solids were then eliminated by filtration through 2.7 μm pore size filters (Millipore) and the filtrate was centrifuged (12,096*g*, 20 °C, 15 min). Supernatant was acidified to pH 4 with HCl 3 N. This acidified liquid was centrifuged (12,096*g*, 20 °C, 15 min) and supernatant was then recuperated and precipitated in 65% (v/v) ethanol for 4 h at 4 °C. Precipitate was recovered and dried by solvent exchange (80% (v/v) ethanol, absolute ethanol and acetone) to give MBG.

2.3. Neutral sugars

Neutral sugars content in MBG was determined after hydrolysis of the gum with 2 N trifluoroacetic acid at 120 °C for 2 h. The reaction was stopped on ice and the extracts were evaporated under air at 40 °C, rinsed twice with 200 μl of water. The evaporated extract was solubilized in 500 μl of water. Sorbitol was used as internal standard. Samples were filtered through 0.45 μm (Whatman) and analyzed by high-performance liquid chromatography (HPLC) using a MetaCarb H Plus column (7.8 × 300 mm; Varian, St. Helens, Australia) eluted with water (filtered 0.2 μm, Whatman) at 0.6 ml/min and 80 °C. A refractive index detector Star 9040 (Varian, St. Helens, Australia) was used.

2.4. Ferulic acid (FA), dimers of ferulic acid (di-FA) and trimer of ferulic acid (tri-FA) in MBG and MBG gels

FA, di-FA and tri-FA contents in MBG and MBG gels were quantified by high-performance liquid chromatography (HPLC) after deesterification step as described by Vansteenkiste et al. (2004). Tri-FA (4-*O*-8′,5′-5″-dehydrotriferulic acid) levels were quantified as described by Rouau et al. (2003). An Alltima C_{18} column (250 × 4.6 mm) (Alltech associates, Inc., Deerfield, IL) and a photodiode array detector Waters 996 (Millipore Co., Milford, MA) were used. Detection was by UV absorbance at 320 nm.

2.5. Proteins

Protein content in MBG was determined according to the Bradford method (Bradford, 1976).

2.6. Ash

Ash content was determined according to the AACC methods (AACC, 1998).

2.7. Viscosity determinations

Specific viscosity (η_{sp}) of MBG solutions was measured with an AVS 400 capillary viscosimeter (Schott Geräte, Hofheim, Germany), equipped with an Oswald capillary tube (flow water time 75.15 s). The η_{sp} was related to the MBG concentration (η_{sp}/C) to obtain their reduced viscosity η_{red} (ml/g) according to Rao (1993). The intrinsic viscosity [η] was determined by the Mead, Kraemer and Fouss method (Kraemer, 1938; Mead & Fouss, 1942).

2.8. MBG gel preparation

MBG solutions (1% and 2% w/v) were prepared in 0.05 M citrate phosphate buffer pH 5. Laccase (1.675 nkat per mg MBG) was added to MBG solution. Gels were allowed to form for 2 h at 25 °C.

2.9. Rheology

The hardness of 1% and 2% (w/v) MBG gels, freshly made (2 h) in 6 ml glass flasks of 30 mm height and 25 mm internal diameter was analyzed with a TA.XT2i Texture Analyzer (RHEO Stable Micro Systems, Godalming, England). The gels were deformed by compression at a constant speed of 1.0 mm/s to a distance of 4 mm from the gel surface using a cylindrical plunger (diameter 15 mm). The peak height at 4 mm compression was called gel hardness (Carvajal-Millán et al., 2005a).

2.10. Emulsion stability index (ESI)

ESI of Mexican oregano oil-in-water at 0.2 (v/v) containing MBG at different concentrations varying from 0% to 1% (w/v) was determined by a turbidimetric method as described by Adapa, Schmidt, and Toledo (1997). Emulsions were placed in a 1-cm pathlength cuvette and absorbance was measured using a UV–vis spectrophotometer Cary IE (Varian, St. Helens, Australia) at a wavelength of 500 nm. ESI was expressed as a function of absorbance.

3. Results and discussion

3.1. MBG extraction and characterization

Table 1 shows the yields on MBG for the different NaOH incubation times. MBG yields were dependent on the time of extraction with values ranging from 0% up to 66% (w MBG/w maize bran total AX content). However, the gelling capability of MBG was greatly affected by time of extraction and after 8 h of 0.5 N NaOH incubation, MBG did not form gels. The latter can be related to a reduction in FA content and MBG intrinsic viscosity in MBG samples (Table 1), as a result of the severity of the extraction procedure. Therefore, 8 h of maize bran alkali-extraction was selected in this study, as this condition provided the maximal yield (29% w MBG/w maize bran total AX content) on gel forming MBG.

Table 1
Yield, FA content and intrinsic viscosity ([η]) of MBG obtained at different incubation time in 0.5 M NaOH at 25 °C and 100 rpm in darkness

Incubation time (h)	Yield % (w MBG/w total AX content in maize bran)	FA (μg/mg MBG)	[η] (ml/g)
0	0.0	–	–
2	5.5	1.6	275
4	13.0	0.6	240
8	28.5	0.34	208
12	38.5	0.22	190
16	52.0	0.15	183
24	66.0	0.1	170

All results are obtained from triplicates.

Composition of this MBG is presented in Table 2. Arabinoxylan (AX) represented 74% dry basis (db) of the recovered MBG. This value was estimated from the sum of xylose + arabinose. The ratio arabinose-to-xylose was high ($A/X = 0.85$) indicating a moderately branched structure, similar to that reported by Singh, Doner, Johnston, Hicks, and Eckhoff (2000) in maize bran arabinoxylans. Residues of glucose, galactose, mannose, proteins and ash were also detected in this MBG. The ferulic acid (FA) content (0.34 μg/mg MBG) was similar to that obtained by Lapierre et al. (2001). Di-FA and tri-FA were also detected in MBG (0.77 and 0.39 μg/mg MBG, respectively) suggesting that some arabinoxylan chains might be inter and/or intra cross-linked. The di-FA and tri-FA contents in this MBG were higher than FA content. Lapierre et al. (2001) reported similar results in a maize bran heteroxylan fraction extracted under similar alkaline conditions with FA and di-FA content of 0.32 and 0.60 μg/mg heteroxylan fraction, respectively. The maize bran sample used by these authors contained a FA and di-FA content of 28.2 and 6.8 μg/mg, respectively, the 8–5′ and 5–5′ structures being the main diferulates. In the maize bran sample used in our study FA and di-FA represented 37.0 and 6.5 μg/mg, respectively, the most prominent being the 5–5′ diferulate structure. Accord-

Table 2
Composition of maize bran gum

Arabinose[a]	34.00 ± 0.90
Xylose[a]	40.00 ± 1.60
Glucose[a]	5.10 ± 0.20
Galactose[a]	3.20 ± 0.10
Mannose[a]	0.40 ± 0.02
Protein[a]	2.50 ± 0.10
Ash[a]	4.10 ± 0.11
Ferulic acid[b]	0.34 ± 0.01
Diferulic acids[b]	0.77 ± 0.01
Triferulic acid[b]	0.39 ± 0.01

All results are obtained from duplicates.
[a] Results are expressed in g/100 g MBG matter.
[b] Phenolics are expressed in μg/mg MBG.

ingly with results of Lapierre et al. (2001), in our study, a very small proportion of ester-linked ferulic acid (about 1%) survived the alkaline extraction used for the isolation of MBG while a higher proportion of di-FA (about 10%) survive the extraction process. The relative percentages of each di-FA structure were: 16%, 21% and 63% for the 8–5′ (mainly in the benzofuran form), 8-*O*-4′ and 5–5′ structures, respectively. The predominance of 5–5′ dimer structure has not been previously reported in MBG. Lapierre et al. (2001) reported the 8–8′ di-FA structure as the main diferulate in a maize heteroxylan fraction recuperated under different alkaline extraction conditions. Nevertheless, the 8–8′ di-FA structure was not detected in our study. It has been reported that pericarp of tropical maize varieties resistant to storage pest has a higher concentration of phenolic acids in the cell wall (García-Lara et al., 2004). Arnason et al. (1994) demonstrated that storage pest resistant maize genotypes have higher concentrations of total di-FA. García-Lara et al. (2004) reported a higher concentration of 5–5′ di-FA structure in tropical maize genotypes resistant to a storage pest. It has been reported that di-FA linkages within the pericarp cell wall of maize provide a biochemical mechanism for controlling the mechanical properties of the cell wall (Waldron, Parr, Ng, & Ralph, 1996) and they limit the biodegradation of cell wall polysaccharides (Ishii, 1997). The latter could explain the insect resistance in tropical maize genotypes. Thus, the predominance of 5–5′ di-FA structure in our MBG could be attributed to the higher content of this diferulate in our maize bran sample. The intrinsic viscosity ([η]) value of MBG was 208 ml/g, which is higher to that reported for other MBG (Chanliaud, Saulnier, & Thibault, 1995).

3.2. MBG functional properties

3.2.1. MBG gel hardnes and cross-linking content

MBG solutions at 1% and 2% (w/v) produced firm and brittle gels in presence of laccase. The hardness and di- and tri-FA cross-linking content of MBG gels at 1% and 2% (w/v) are reported in Table 3. Gel rheological properties were greatly affected by MBG concentration. Sixty percent increase in the gel hardness was recorded by increasing the MBG concentration from 1% to 2% (w/v). This increase in gel hardness can not be explained by the di- and tri-FA content, which was similar. At the end of gelation, 79% and 76% of the FA initially present in 1% and 2% (w/v) MBG solutions were oxidized while only 17% was recovered as di and tri-FA. As a matter of fact, the di- and tri-FA content in MBG did not increase after laccase-induced gelation, they rather decreased from 0.77 to 0.03 and from 0.390 to 0.015 μg/mg MBG, respectively. In a similar study, Lapierre et al. (2001) reported a similar di-FA content (0.06% w/w or 0.6 μg/mg MBG) in MBG, which did not increased after peroxidase gelation, in spite of a decrease in FA content. These authors atributed this result to the formation of ferulate cross-linking structures which can not be released by mild alkaline hydrolysis and/or to the participation of lignin residues in the formation of the peroxidase-induced MBG gel. Grabber, Ralph, and Hatfield (1988) have reported the propensity of ferulate esters to copolymerize with lignin units. Lapierre et al. (2001) suggested that cross-linking of maize heteroxylan chains may not exclusively involve ferulic acid, but also lignin units.

Table 3
Hardness and FA, di-FA and tri-FA content in MBG gels at different concentrations

	MBG concentration (% w/v)	
	1	2
Hardness, H (N)	0.32 ± 0.01	0.81 ± 0.01
FA (μg/mg MBG)	0.071 ± 0.002	0.081 ± 0.002
Di-FA (μg/mg MBG)	0.030 ± 0.001	0.030 ± 0.001
Tri-FA (μg/mg MBG)	0.015 ± 0.001	0.015 ± 0.001

All results are obtained from triplicates.

This decrease in FA content without a proportional formation of di- and tri-FA structures has been recently reported by Carvajal-Millán et al. (2005a), Carvajal-Millán et al. (2005b) and Carvajal-Millán et al. (2005c) in laccase-induced wheat arabinoxylan gels. These results were related to the formation of non-detected di- and tri-FA or higher ferulate structures and/or to physical interactions between arabinoxylan chains. In our study, the relative percentages of di-FA structures found in MBG gels were different to those found in the MBG as only the 5–5′ and 8-*O*-4′ di-FA structures were detected and at relative percentages of 50%. Lapierre et al. (2001), reported that the di-FA profile of a MBG was not altered by peroxidase gelation, the 8–8′ structure being predominant.

3.2.2. MBG emulsion stability index (ESI)

The ESI of 0.2% (v/v) oregano oil-in-water containing MBG at different concentrations is showed in Fig. 1. It was found that ESI increased from 0.01 to 0.20 as the MBG concentration increased from 0% to 1% (w/v). In general, it is considered that hydrocolloids stabilize emulsions

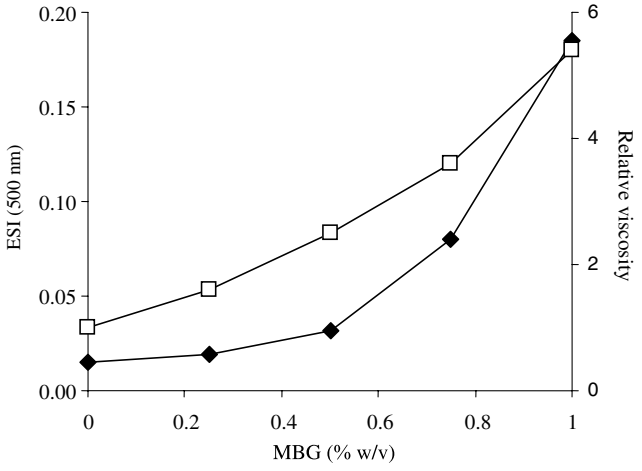

Fig. 1. Emulsion stability index (ESI) of Mexican oregan oil-in-water containing MBG at different concentrations (◆). The relative viscosity of MBG solutions in water at the same concentrations is indicated (□).

primarily by increasing the viscosity on the continuous phase (Cui, 2001). In Fig. 1 is also showed the relative viscosity (η_{rel}) of MBG in water as a function of gum concentration. The η_{rel} values varied from 1.0 to 5.4 as the MBG concentration increased from 0% to 1.0% (w/v). This increase in η_{rel} could then explain the improvement in ESI of oregano oil-in-water as a function of MBG concentration in the mixture.

4. Conclusions

MBG with a high arabinoxylan content can be recovered from a maize bran industry milling residue. Under the extraction conditions used in this study, the MBG recovered presented a ferulic acid content high enough to allow the formation of a firm laccase-induced covalent gel. The covalent cross-linking structures (di- and tri-FA) quantified in MBG gels did not increase with the gum concentration (1% and 2% w/v) even when the gels hardness is highly improved. These results suggest that covalent linkages through di- and tri-FA quantified structures are only partially involved in the MBG gel formation. Other covalent cross-links might result from the strong oxidizing treatment (eg.: lignin compounds) and/or physical interactions participate in the MBG gel structure.

MBG can improve the ESI of oregano oil-in-water mixtures by a 95% at a MBG concentration of 1% (w/v). This ESI capacity is related to the high viscosity of MBG in water, which is probably originates from a moderately branched arabinoxylan structure as suggest the arabinose to xylose ratio. The MBG presented in this study could be a potential candidate as encapsulation and emulsification agent in the food industry. Recuperation of this MBG from a low-value maize milling by-product could represent a commercial advantage face to other gums commonly used in the food industry.

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