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Extraction and characterization of sunflower pectin

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Abstract

Chemical composition and rheological properties of sunflower pectin, including gel-point temperature (T_g) were determined. Pectin from sunflower head residues was characterized and compared with low methoxyl commercial pectins. Pectin was isolated using sodium hexametaphosphate extraction followed by either acidified alcohol (KIM) or HCl solution (LIN) precipitation. LIN pectin resulted with a higher ash content (\approx 30%).The low methoxyl pectin extracted from sunflower heads had 11% degree of esterification, high anhydrogalacturonic acid content (77–85%) and low acetyl content (2.3–2.6%) on an ash-free basis. Viscosity average molecular weight ranged from 39,500 to 52,000. Sunflower pectin formed gels at high temperatures almost instantly in the presence of calcium ions.

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1. Introduction

Pectin has been used as a gelling agents since practically the last two centuries (Oakenfull & Scott, 1984). Pectins are bio-polymers consisting almost entirely of D-galacturonic acid (AGA) and galacturonic acid methyl ester residues. Some pectins, like pectin obtained from sunflower, shown to be also acetylated at some degree. Pectins with degree of methylation (DM) higher than 50%, named high methoxyl pectin (HMP) form gel after heating in sugar solutions at concentration higher than 55% and pH lower than 3.5. On the other hand, formation of gel with a low methoxyl pectin (LMP; DM < 50%) requires the presence of calcium ions, extending the use of this gelling agent to a broader range of foods. LMP may be used as a gelling agent in lowsugar products, such low-calorie jams and jellies, confectionery jelly products, and other foods applications. The heat reversibility of LMP gels can be utilized in bakery jams and jellies for glazing, retorting, microwaving, baking, and sterilizing or pasteurizing.

Although some LMP occurs in plants, they are usually manufactured from HMP. There are four methods of demethylation according to the agents used: acids, alkalis, enzymes and ammonia in alcohol. Sunflower pectin, obtained from heads and stalks which remain in the field after seed removal, is a naturally occurring LM pectin. Papers on sunflower pectin extraction methods (Kim, Sosulski, & Campbell, 1978a, 1978b); chemical composition (Miyamoto & Chang, 1992) and gelling properties (Li & Chang, 1997) are available. However, it is known that cultivar, location and processing methods affect yield and quality of sunflower pectin (Chang, Dhurandhar, You, & Miyamoto, 1994). Particularly, information on the pectin from sunflower variety TC 3004-ATAR (Argentina) is not available.

The gelation of LMP was described as quite a complex process (Grosso & Rao, 1998). This gel point may also be defined as the stage at which a liquid begins to exhibit pseudoplastic properties. This stage may conveniently be observed from the inflection point on a viscosity time plot. After some time, a stable gel is reached (Doublier, Launay, & Cuvelier, 1992). The rates of gelstructure development and gel strength are affected by type of pectin, pH, solute and temperature. It is very difficult to predict the temperature of gelation (T_g) , which must be determined experimentally. A small amplitude oscillatory rheological test (SAOT) has been used for studying the gelling mechanism (Doublier et al., 1992). Characterization of the storage (G') and the loss (G'') modulus, as well as T_{g} can be obtained using a rheometer either with controlled step displacements or

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with dynamic oscillatory deformations (da Silva & Gonçalves, 1994) and if fracture at the tool surfaces can be prevented, the yield stress can be determined also. The transition from viscoelastic liquid to viscoelastic solid is a gradual one. There is no discontinuity in the rheological behavior. At least three conditions must be satisfied by any gelling point determination method: (i) it must be non-destructive; (ii) gels must be deformed within the linear viscoelastic range; and (iii) results should be expressed in fundamental terms.

By using low strain amplitude (<1°) and measuring the phase angle, it is possible to study gelation with minimal risk of damage to the gel structure. The elastic modulus (G') and the viscous modulus (G'') as a function of frequency (ω) have been used to define the gelling temperature (T_g) and setting time (t_g) (da Silva & Rao, 1999).

The main objectives of this work were (1) to extract pectin from sunflower head residues; to (2) chemically characterize the obtained pectin; and (3) to study the gelling capability of this product. Most of studied gel properties will be compared with that of commercial demethylated citrus pectin.

2. Materials and methods

2.1. Pectin isolation

Pectin was extracted from sun dried heads (without seeds) of sunflower (*Heliantrus annuuus* L., variety TC 3004-ATAR) harvested in Cnel. Pringles (Buenos Aires, Argentina). The extraction of pectin was based in the method proposed by Kim et al. (1978a, 1978b); including milling the sunflower heads in a Fitzmill Comminutor (The Fitzpatrick Co., USA); ground heads were extracted with water (25 l/kg solids) at 75 °C for 15 min mainly to remove pigments; filtration; and the solids treated with a solution (0.75%) of sodium hexamethaphosphate (SHMP) at 75 °C for 1 h. Solid:liquid ratio was 20:1 and a pH = 3.5 was obtained by adding HCl (6 N).

After extraction, the pectin solution was filtered through a Büchner funnel with a Whatman No. 4 filter paper and diatomaceous earth pre-coat. One half of the filtrate was precipitated with HCl (1 N) (Lin, Humbert, & Sosulski, 1976) and was identified as LIN sample; and the other half precipitated with 95% acidified ethanol (Kim et al., 1978a, 1978b) and identified as KIM sample. LIN sample was concentrated under vacuum (55 °C) in a Büchi Rotovaporator (Büchi Co., Zurich, Switzerland), volume ratio 2:1. Concentrate pectin solution was precipitated with acidified ethanol (96%). Formed gel was allowed to precipitate for 19 h at 3 °C. KIM sample was precipitated by mixing with HCl (1 N) in a 1:3.2 acid to extract ratio. More details on gel precipitation, washing and drying are given in Fig. 1.

Dry LIN and KIM pectin were finally ground to pass a 100 mesh sieve. Other pectin samples used were citric pectins Sigma P9311 and P9436, with 31% and 67%DM, respectively (Sigma Chemical Co., St. Louis, USA), and the commercial citric pectins Genupectin LM104AS (DM = 31%) and LM101AS (DM = 36%) (Hercules, Copenhagen Pectin, Denmark).

2.2. Pectin characterization

Ash content was determined according to the method proposed by McCready (1970). Pectin sample (1 g) was incinerated in a furnace at 600 °C for 4 h; cooled and stored in a desiccator with PO_5 until weighting.

Standard pectin gels were prepared as suggested by the National Research Council (1972) Method, after modifications proposed by Chang and Miyamoto (1992). A standard pectin gel contains 1% pectin; 30% sucrose; 25 mg Ca²⁺/g pectin (pH = 2.0). Selected pH (2.0–2.8) was adjusted in the hot pectin solution (Orion pH meter with high temperature electrode) with the addition of citric acid and sodium citrate solutions. Soluble solids were determined with a bench top Abbe refractometer. All the other reagents were analytical grade and used without further purification.

2.3. Determination of AGA content (% AGA)

Anhydrogalacturonic acid (AGA) was qualitatively determined by the colorimetric phenyl-phenol method (Blumenkrantz & Asboe-Hansen, 1973) with an UV/ VIS. Perkin–Elmer Lambda 3 Spectrophotometer, at 520 nm (Abs₅₂₀) in a 1 cm quartz cell. Washing water used during pectin extraction was also assayed for AGA content.

2.4. Determination of the degree of methylation of pectin (DM)

DM was determined by gas chromatography, based in the method proposed by Lee, Acree, and Butts (1975). A stock solution of pectin was prepared at a concentration of 8.33 mg/ml in distilled water. Pectin methyl esters were hydrolyzed as follows: 1 ml of NaOH 1 N was added to 12 ml pectin solution. This solution was incubated during 30 min at room temperature, and finally diluted with distilled water to give 20 g. Aliquots of the hydrolyzed pectin were analyzed for methanol. A Varian 3700 gas chromatograph with a 2 m×2 mm ID stainless steel filled column (Porapak N 80/100 mesh) was used. The oven temperature was fixed to 115 °C and the injector and detector (FID) temperatures were 210

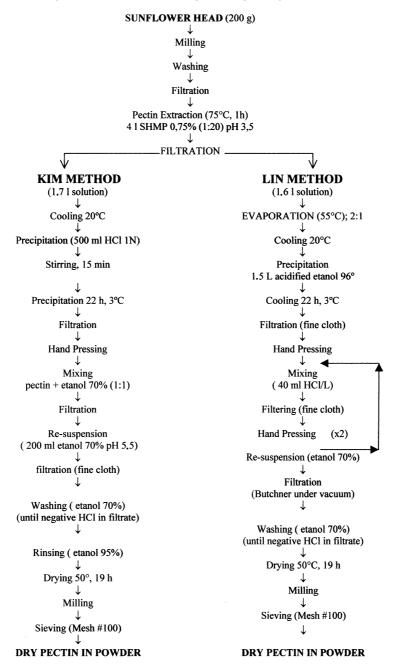


Fig. 1. Flow diagram of pectin extraction procedures.

and 220 °C, respectively. The carrier gas (N₂) flow rate and sample volumes were 20 ml/min and 2 μ l, respectively. Standard curve was prepared with known amounts of methanol in distilled water. Fitting the calibration curve resulted in a straight line through the origin with a regression coefficient $r^2 = 0.9975$. DM was calculated as the ratio molar of methanol to AGA, expressed as a percentage.

The DM value was calculated through the following equation:

$$DM = Meq. ECG/Meq. TCG$$
(1)

where ECG are the esterified carboxylic groups (generated methanol) and TCG are the total carboxylic groups calculated from the galacturonic acid content.

2.5. Determination of the acetyl group

Acetyl ester content was determined by an enzymatic method (Boehringer Mannheim, 1997) based on the colorimetric determination of NADH. The acetyl content was estimated after hydrolysis of pectin by KOH (1 N) for 30 min at room temperature, using a standard curve prepared with known amounts of acetic acid.

2.6. Determination of molecular weight (M_w)

Sunflower pectin molecular weight was estimated by applying the Mark Houwink–Sakurada equation, relating $[\eta]$ with M_w (Arslan, 1995):

$$[\eta] = k[M_{\rm w}]^a \tag{2}$$

where k and a are constants and $[\eta]$ is the intrinsic viscosity defined as:

$$[\eta] = \lim_{C \to 0} \left(\frac{\eta_{\rm r} - 1}{C} \right) \tag{3}$$

where η_r is the relative viscosity (solution to solvent) and *C* is the pectin concentration. Both *k* and *a* depend on temperature and solute and solvent characteristics. A large number of models have been used to deduce $[\eta]-M_w$ relationships (da Silva & Rao, 1992). In this work the following values were assumed $k = 9.55 \times 10^{-4}$ and a = 0.73 (Anger & Berth, 1986). The two equations commonly employed for determining $[\eta]$ of food gums; Huggins (Eq. (4)) and Kraemer (Eq. (5)):

$$\frac{\eta_{\rm sp}}{c} = \left[\eta\right] + k_{\rm h} \left[\eta\right]^2 c \tag{4}$$

where $\eta_{sp} = \text{specific}$ viscosity; c = concentration; $\eta_{sp}/c = \text{reduced}$ viscosity and $k_h = \text{Huggins constant}$.

$$\frac{\ln \eta_{\rm r}}{c} = [\eta] + k_{\rm k} [\eta]^2 c \tag{5}$$

where $k_k =$ Kraemer's constant.

Determination was made in two ranges of pectin concentration (0.0035–0.012 and 0.006–0.020 g/l) by extrapolation to zero concentration (Billmeyer, 1984; Rao, 1999). Viscosity measurements were performed at 25 °C with a Bohlin RVO stress controlled rheometer using concentric cylinders geometry (C25). Constant shear stress ($\sigma = 0.1$ Pa) was selected.

2.7. Gel evaluation

The Ridgelimeter is the most commonly used device to standardize highly methoxyl pectins for commercial use (Cox & Higby, 1944). This empirical Sag-test is a one-point, non-destructive measurement. Although sag measurements are not a sufficient quality criterion (Endreß, Bamberger, & Dengler, 1995) the method shows excellent reproducibility and easy handling. Therefore, the gel forming ability of sunflower pectin was also quantified by the IFT SAG method (IFT, 1959) after the modification proposed by Black and Smit (1972) for low-ester pectins. The reading on the Ridgelimeter was corrected for temperature after conversion to %sag. Readings were taken on duplicate gels.

Black and Smit (1972) defined the low methoxyl pectin firmness (LMPF) as the weight of the gel divided by the weight of a pectin sample required to yield a gel of standard firmness having a sag of 10.5 mm at 11 °C.

Furthermore, the low methoxyl pectin strength (LMPS) was defined as National Research Council (1972) as:

$$LMPS = \frac{100}{w} \left[\frac{2.0 - (\%sag + 4.5)}{25.0} \right]$$
(6)

where w is the weight of gel batch and %sag was corrected to 25 °C. Gel pH and soluble solids were determined with a Cole-Parmer DigipHase (Cole-Parmer, USA) and a Reichert Abbe Mark II Refractometer (Reichert Scientific Instrument, Buffalo, NY, USA), respectively.

2.8. Small amplitude oscillatory test

Rheological properties, including gel-point temperature (T_g) were determined with a Bohlin CVO (Bohlin Instruments Ltd., London, UK) rheometer with cone (4 cm dia.; 4°) and plate system. A solvent trap was placed on top of the cone to minimize moisture loss (Arenaz & Lozano, 1998). Three methods were used to test pectin solutions dynamically:

- (1) Controlled cooling: The hot pectin dispersion was poured on the rheometer plate, set at 99 °C and the cone lowered to the selected gap (150 µm). Dynamic test was immediately done (0.1 Hz; 5% strain) while temperature sweeps over 99–20 °C (cooling rate, 3 °C/min). Magnitudes G', G'', complex viscosity (η^*) and phase angle (δ) as a function of temperature were acquired.
- (2) Controlled heating: Gel formed in between cone and plate after cooling was heated from 20 to 95 °C at a rate of 3 °C/min. Magnitudes G', G", and phase angle (δ) as a function of temperature were obtained.
- (3) Frequency sweep: In this case rheological tests were performed in the range 0.005–1.0 Hz (initial strain 0.03 Pa) on pectin gels at temperatures in the range 5–30 °C. Formerly, an amplitude sweep to ascertain the linear viscoelastic range was performed on selected samples. In food gels, the temperature where the total phase angle variation is halved $(\Delta \delta_0/2)$ can be regarded as rather a good criterion to define the gel point (T_g) which is in accordance with the temperature at which G' = G'' (Barfod & Pedersen, 1990).

3. Results and discussion

Sunflower head residues contained 13.9% (±0.8) moisture. While sunflower head pectin obtained by acidified ethanol precipitation (KIM) had an ash level of 6.1%, similar to that values reported by Sosulski, Lin, and Humbert (1978), LIN pectin obtained by precipitation with HCl had approximately 30% (±1.2) ash. Difference in level of ash was attributed to pH during

Table 1 Galacturonic acid (AGA) content of pectin

Pectin	AGA ^a g/100 g	Standard error	AGA (free ash basis)
LM 104-AS	46.46	2.71	_
Sigma P-9436	77.65	3.25	_
LIN 76 m	52.93	3.11	77.05%
KIM 78 m	79.92	2.54	85.12%
Heads washing water	1.197 g/l	0.044	_

^a Determinations were made in triplicate.

extraction (Lin et al., 1976). Pectin yield resulted 11.6 ± 0.5 g (wt.%) and 7.4 ± 0.3 g (wt.%) for LIN and KIM pectin, respectively. These values are higher than the obtained from traditional pectin sources, like citrus and apple (Alarcão & Silva, 1990).

Calculated degree of acetylation was $13.71 \pm 0.98\%$ and $10.82 \pm 0.85\%$ for LIN and KIM pectin, respectively. The low methoxyl pectin extracted from sunflower heads had $11 \pm 0.16\%$ degree of esterification, high AGA content (77-85.1%) and low acetyl content $(2.3 \pm 0.16\% - 2.6 \pm 0.09\%)$ on an ash-free basis. AGA levels were similar to that reported for sunflower pectin (Marechal & Rigal, 1999; Miyamoto & Chang, 1992; Sosulski et al., 1978) and higher to that of demethylated citrus pectin (Table 1). It was also found that, in addition to undesirable pigments, washing water also removed up to 2.9% from the total sunflower head AGA content. Acetyl content was lower than 4% (2.6% in the case of LIN pectin and 2.25% for KIM pectin), a value considered to be the critical limit for gel formation (Pilnik & Voragen, 1992) and similar to the acetyl level found in sunflower pectin (Alarcão & Silva, 1990; Miyamoto & Chang, 1992).

3.1. Intrinsic viscosity and molecular weight

The viscosity of sunflower pectin in NaCl 0.1 M solution and intrinsic viscosity (by extrapolation to zero concentration) obtained by Billmeyer (1984) and Rao (1999) procedures were studied. Fig. 2 shows a typical result obtained after extrapolation of Huggins (Eq. (4)) and Kraemer (Eq. (5)) equations. It can be observed that relative viscosity (η_r) values varied with the concentration criteria-Rao or Billmeyer-applied for calculation. The viscosity average molecular weight was calculated by applying Eq. (2) with constants proposed by Anger and Berth (1986). Table 2 shows intrinsic viscosity and average molecular weight of sunflower pectin solutions, extracted from head pomace. Viscosity average molecular weight was in the range 39,500-52,500 and 35,300-48,000 on an ash-free basis, for LIN and KIM pectin, respectively. These values were considerably lower than those obtained by Kim et al. (1978a, 1978b) ($M_w \cong 110,000$) and Alarcão and Silva (1990) ($M_{\rm w} \cong 105,000$).

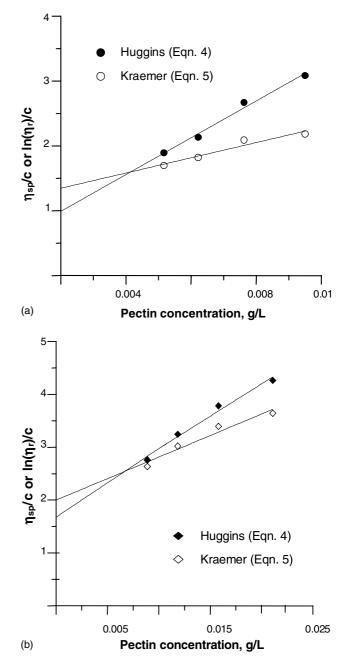


Fig. 2. Intrinsic viscosity of LIN sunflower pectins in NaCl 0.1 M solution, by extrapolation to zero concentration with Huggins (Eq. (4)) & Kraemer (Eq. (5)) equations. Relative viscosity (η_r) values varied with the concentration criteria—Billmeyer (Fig. 2a) or Rao (Fig. 2b)— applied for calculation.

3.2. Gel evaluation

Determined %sag; LMPF; and LMPS of gels from different pectins were listed in Table 3. LMPF value for sunflower KIM pectin was around 126. The gels of commercial citric and LIN pectin did not stay raised by themselves during sag assay.

Results indicated that KIM pectin gave a gel firmness comparable to the commercial standardized Genupectin

Parameters	Extrapolation	$[\eta] (dl/g)^a$		$M_{ m v}{}^{ m b}$	
		LIN 76	KIM 78	LIN 76	KIM 78
Rao ($\eta_{\rm r} = 1.2 - 2.0$)	Huggins	2.325	2.110	43,555 (±830)	38,103 (±994)
	Kraemer	3.019	2.870	62,265 (±1200)	58,099 (±1150)
	Average	2.672 ^c	2.489 ^c	52,685	47,818
Billmeyer	Huggins	2.040	1.864	36,397 (±760)	32,173 (±557)
$(\eta_{\rm r} = 1.1 - 1.5)$	Kraemer	2.294	2.128	42,746 (±921)	38,561 (±1018)
	Average	2.167 ^c	1.996°	39,537	35,328

Intrinsic viscosity $[\eta]$ and average molecular weight $(M_{\rm v})$ of pectin solutions, extracted from sunflower head pomace (ash-free basis)

^a Determination were made in duplicate.

^b Molecular weight was estimated by applying Eq. (3) with constants proposed by Anger and Berth (1986).

^cHuggins and Kraemer's extrapolations average.

Table 3 Determined %sag; low methoxyl pectin firmness (LMPF); and low methoxyl pectin strength (LMPS) of gels from different pectins

Pectin	% sag (25 °C)	LMPF	LMPS
KIM	17.68	126	111.26
LIN	63.59	_	_
LM101AS	17.64	126.5	113.43
LM104AS	16.37	>126.5	116.51

Gel composition: 1% pectin, 25 mg Ca²⁺/g pectin, pH \approx 3.

LM101AS gel and slightly inferior to the LM104AS gel. LIN sunflower pectin did not gel suitably. For that reason the extraction with the method by Kim et al. (1978a, 1978b), is recommended. If Lin et al. (1976) precipitation method with acidified ethanol was employed during pectin production, a later washing with HCl is advisable to improve gel strength.

Kim et al. (1978a, 1978b) evaluated the gel firmness of sunflower pectin and found that the optimal conditions were 1% pectin, 22.5 mg Ca²⁺/g pectin, pH 4.3% and 30% sucrose. Sosulski et al. (1978) used the same method of extraction and precipitation of sunflower pectin and obtained firmer gels with 30–40% of sugar and 25–30 mg/g calcium levels. These last authors prepared the gel according to National Research Council (1972) and determined LMPF value, using the method of Cox and Higby (1944) with standard glasses at 25 °C, was 139. In our case, obtained gel firmness value at 11 °C and applying the factor of correction of temperature proposed by Black and Smit (1972) was LMPF = 125.1.

Chang and Miyamoto (1992), using also the sag method and standard procedure, obtained average gel power of 111.1 and 119.7 for sunflower and citric commercial LM104AS pectins respectively. These values are similar to the obtained in this work: 111.3 and 116.5 (Table 3) with the same method of calculation, but in cylindrical glasses, previously cured at 11 °C and later corrected to 25 °C according to Black and Smit (1972).

3.3. Sol-gel transition results

Fig. 3 shows a typical plot of G', G'' and δ versus temperature, obtained from small amplitude oscillatory measurements at constant frequency during controlled cooling. Fig. 3 shows the formation of a critically Cross-Linked network as characterized by the temperature dependence of G' and G'' on cooling. The rheological response shows that after a period of instability at elevated temperatures, the pectin changes during cooling from predominantly liquid-like solution, to a typically gel-like structure with G'' < G. The same Fig. 3 also shows the variation of δ during cooling of sunflower pectin solution. Temperature at which $\delta = 45^{\circ}$ was also assumed as the gelling temperature T_g .

Results indicated that $T_{\rm g}$ for a gel formed with 0.5% sunflower pectin, 25 mg Ca²⁺/100 g, 30% sugar and

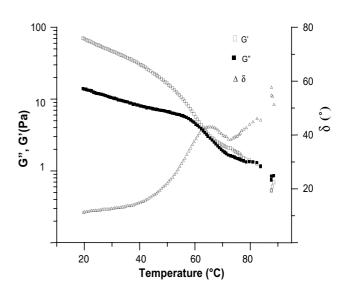


Fig. 3. Semilog plot of *G'*, *G''* and δ related to temperature, obtained at constant frequency (1 Hz) during controlled cooling 3 °C/min of sunflower pectin sample LIN 0.5% (0.34% ash free), 30% sugar, 25 mg Ca²⁺/g pectin, pH 2.7.

Table 2

pH \cong 3 was around 80 °C. In the case of a more concentrated pectin solution (1%) T_g resulted higher than 90 °C. Temperature of gelling was difficult to determine in low methoxy pectin due to rheometer limitations (particularly to keep sample temperature near boiling point) and assay conditions (LMP gels almost instantaneously at elevated temperatures).

In the case of sunflower pectin the variation of modulus G' and G'' with temperature followed the same rule applicable for any polysaccharide gel, characterized with a G' "plateau" during an extended frequency range. This behavior reflects the existence of a tri-dimensional network depending on the molecular weight (Endreß et al., 1995).

The analysis of G' and G'' values shows that in absolute value the firmness of the gel is, from greater to minor: $KIM'' \gg LM104AS > LIN \gg Sigma P9311$. In other words, the gel processed with KIM pectin has a firmness similar to that of the commercial citric pectin LM104AS. The lower the degree of methoxylation of pectin, the greater the similarity with AGA and greater it is the reactivity with calcium, as it was reflected in the elevated gelling temperatures observed. Gelling occurs by the addition of calcium ions, and frequently pregelling happens practically simultaneously with the addition of calcium chloride to the pectin solution.

3.4. Study of gel properties by frequency sweep

 $T = 30^{\circ}C$

□ G'; 50 mg Ca/g

G"; 50 mg Ca/g

G'; 25 mg Ca/g
G"; 25 mg Ca/g

1.0

1000

100

10

1

0.1

G, G" (Pa)

The log-log representation of the elastic and dynamic modules—G' and G''—(Fig. 4) as a function of the an-

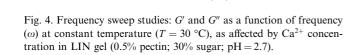
0

100.0

gular velocity ω shows the characteristic spectrum of the pectin gel. It was observed that the absolute values of both modules were greater in the gel with greater calcium content, and follows a practically linear behavior. The gel with lower calcium content exhibited a greater dependency of the modules with frequency, and a smaller ratio G'/G'', which indicates the gel was weaker. Therefore, the higher the calcium content available in the solution, the greater the formation of a three-dimensional network, resulting in stronger gels.

On the other hand, sunflower pectin precipitated with acidified ethanol (LIN) showed a strength similar to the commercial citric Sigma. The elastic to viscous module ratio (G'/G'') of a viscoelastic gel leads to an objective description of the sensorial properties of the gel and represents a base for pectin standardization (Endreß et al., 1995). It is known that this ratio affects the texture: a very large elastic module and a very small viscous module results in a high G'/G'' ratio, indicating a brittle gel with a high breaking strength. The gel is sensible to mechanical processing and shows tendency to syneresis.

Gels with low G'/G'' values are soft and show better extensibility. They are more stable during mechanical processing and show less syneresis. The value of G' is between 6 and 8 times greater than G'', depending on the type of gel (Fig. 5). This observation indicates a predominantly elastic behavior in gels of LMP with calcium, with a quite permanent character of the zones of union, with no evidence of a reordering in the structure of the network (Axelos, Lefebvre, Qiu, & Rao, 1991).



10.0

ω (Hz)

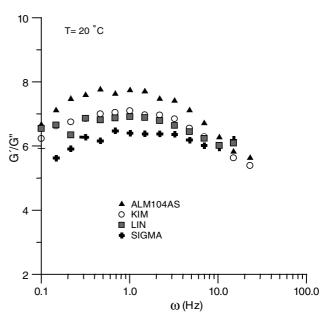


Fig. 5. Frequency dependence of G'/G'' at 20 °C. Gels composition: 1% pectin; 30% sugar; (25 mg Ca²⁺/g).

3.5. Gel melting results

During the heating process of gels, the values of G' was much greater than those of G''. The magnitude of G' diminished with the increase of the temperature and reached a minimum when approaching the temperature of melting ($T_{\rm m}$). The melting data were analyzed according to the method of Fu and Rao (1999). The determination coefficient (R^2) was >0.998 for the studied samples. $T_{\rm m}$ was determined as the first value of the temperature in which dG'/dT = 0. Obtained temperature of fusion was $T_{\rm m} = 56$ °C for the 0.5% LIN pectin gel with 50 mg Ca²⁺/g, and 30% sugar (pH 2.8) (warming up rate: 4 °C/min). The temperature of melting is important in the manufacturing of fruit products stable during heating.

4. Conclusions

From a practical point of view, precipitation method during sunflower pectin extraction affected both the ash content degree of esterification and the degree of polymerization (molecular weight) of extracted pectin. It must be remarked that the gelling temperature of this type of gels is a complex function of several parameters, including number of polymeric chains, solvent interaction and attractive and repulsive forces involved, among others (da Silva & Rao, 1999). The lower the degree of methylation the higher the reactivity with calcium, which was reflected as an increase in T_g . Moreover, pregelling may occurs during the adding of Ca²⁺ even under boiling conditions.

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