

# Viscosity and Gelling Characteristics of Sunflower Pectin As Affected by Chemical and Physical Factors

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The effect of pH, buffer solution, buffer concentration, pectin concentration, and heating on the viscosity of sunflower pectin was studied. The sunflower pectin in all buffers and at all buffer concentrations had the highest viscosities at pH 3. Generally, the viscosity increased with the increase of pectin concentration. The effect of buffer concentration on viscosity was dependent upon the pH, pectin concentration, and heating. Sunflower pectin in sodium citrate buffer had a higher viscosity than pectin in other buffers. The effect of heating on viscosity was dependent upon the pH of the buffer. Gelling heat reversibility of the isolated sunflower pectin was determined and compared with that of two commercial citrus pectins. Jellies of sunflower pectin had good heat reversibility over a wide range of pH values (2.5–5.4). These results indicated that sunflower pectin has a high potential for producing low-calorie jellies.

**Keywords:** *Sunflower pectin; viscosity; gelling*

## INTRODUCTION

Sunflower heads are a good source of natural low-methoxyl (LM) pectin (Knowles, 1978) that can be extracted with solutions containing chelating agents, such as sodium hexametaphosphate (SHMP) (Sosulski et al., 1978). Sunflower pectin has multifunctional properties, including viscosity, gelation, film forming, ion-exchange capacity, and heavy metal chelating properties. Pectin provides beverage viscosity. The viscosity of pectin is used in formulated juice products to restore the mouthfeel of the fresh juice (Copenhagen Pectin, A/S, 1992).

Pectin viscosity and gelling properties are affected by many factors, which are molecular mass, degree of methoxylation (DM), temperature, concentration of solute, pH, and presence of salts (Nelson et al., 1977). Sunflower pectin is a good source of LM pectin. The viscosity and gelling properties of sunflower pectin have not been reported. In the food industry, pumping, which causes shearing, and reheating of food are frequently used. There are no reports on how shearing followed by reheating affects viscosity and gelling of sunflower LM pectin (SP).

Pectin is used as a gelling agent for jams, jellies, and fruit preparations. The heat reversibility of LM pectin gels can be utilized in bakery jams and jellies for glazing and for food applications such as retorting, microwaving, baking, sterilizing or pasteurizing, and freeze/thaw. Therefore, determining the viscosity and gelling properties of pectin may provide useful information for the application of sunflower pectin in the food industry.

The objectives of this study were (1) to investigate the effects of pH, buffer type, buffer concentration, pectin concentration, and heating on viscosity and to investigate the effects of shearing and repeated heating on the viscosity of sunflower pectin and (2) to investigate the

effects of shearing and repeated heat treatments on the gelling characteristics of sunflower pectin and to compare the results to two commercial LM pectins.

## MATERIALS AND METHODS

**Materials.** Sunflower head residues were obtained from Casselton, ND, after harvest October 20, 1992. After harvesting, the heads were dried in a walk-in, forced-air dryer at 55–60 °C containing 10% moisture and ground to pass a 60-mesh screen. The head residues were stored in a –25 °C freezer until use.

**Methods. Pectin Isolation.** Pectin was extracted from sunflower heads according to a modified method of Sosulski et al. (1978). Sunflower head residues were extracted with water (30:1, mL:g) at 75 °C for 15 min, and the extract was discarded. After water extraction, the residues were then extracted twice with 0.75% SHMP at pH 3.5 and temperature 75 °C for 1 h each time. The two SHMP extracts were combined and precipitated with 1 N nitric acid. After acid precipitation, the pectin was washed two times with 2 volumes of 0.1 N HNO<sub>3</sub> and three times with 1.5 volumes of 60–70% alcohol, dried in a vacuum oven at 60 °C overnight, and ground to pass a 60-mesh screen.

**Determination of Viscosity.** Sunflower pectin samples (0.33%, 0.67%, and 1%) were prepared in 0.01, 0.05, 0.1, and 0.2 M buffers (sodium citrate, SHMP, and sodium tripolyphosphate) at pH 2, 3, 4, 5, and 6. Before the viscosity was measured, samples were hydrated at 4 °C overnight, and the pH of the sample solutions was exactly adjusted with 0.1 N NaOH or 0.1 N HCl at pH 2–6. Viscosity was measured at 25 °C using a Brookfield cone/plate viscometer (Brookfield Inc., Model RTV, Houston, TX) at speeds from 0.3 to 30 rpm with cone cp-52 or cp-40. Viscosity of pectin, which has been heated at 100 °C for 5 min, sheared by hand after cooling to 25 °C using a spatula, and reheated at 100 °C for 2 min, was taken after cooling to 25 °C.

**Determination of Gelling Firmness and Heat Reversibility.** Jellies of SP, LM12, and LM104 were prepared, using procedures described by the National Research Council (1972) at pH 2.2 (unadjusted pH of the gel system) and pH 3.0 (adjusted with 0.1 NaOH) with 1.0% pectin, 30% sugar, and 25 mg of calcium/g of pectin and at pH 5.4 with 1% pectin, 20% sugar, and 45 mg of calcium/g of pectin (Chang and Miyamoto, 1992). The texture of gels of SP was determined by using a modification of the method of Freidman et al. (1963), using an Instron Universal testing machine. Two commercial LM pectin samples (LM12 and LM104) were used as references.

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**Table 1. Effect of pH Value, Pectin Concentration, and Heating on Viscosity of Sunflower Pectin in 0.1 M Sodium Citrate Buffer Solution**

| pectin concn (% w/v) |                   | viscosity (cP) at     |           |          |          |          |
|----------------------|-------------------|-----------------------|-----------|----------|----------|----------|
|                      |                   | pH 2                  | pH 3      | pH 4     | pH 5     | pH 6     |
| 0.33                 | no heat           | *1.44h <sup>a</sup>   | *77.6c    | 3.02h    | 2.34h    | 2.55h    |
|                      | heat <sup>c</sup> | 26.7(ef) <sup>b</sup> | 5.78(f)   | 2.01(f)  | 2.26(f)  | 3.21(f)  |
| 0.67                 | no heat           | *1.37h                | *4380.3b  | *15.6fhg | 6.81fhg  | 7.45fhg  |
|                      | heat              | 8931.5(b)             | 155.3(d)  | 4.31(f)  | 5.91(f)  | 5.21(f)  |
| 1                    | no heat           | *1.08h                | * >31000a | *47.8d   | 11.6fhg  | *14.6fhg |
|                      | heat              | 16000(a)              | 598.0(c)  | 26.7(ef) | 11.8(ef) | 6.55(f)  |

<sup>a</sup> Means followed by different letters differ at  $p < 0.05$ . <sup>b</sup> Means followed by different letters in parentheses differ at  $p < 0.05$ . <sup>c</sup> Heat treatment: 100 °C for 5 min. \*Significant difference between heat and no heat treatment at  $p < 0.05$ .

**Table 2. Effect of pH Value, Pectin Concentration, and Heating on Viscosity of Sunflower Pectin in 0.1 M SHMP Buffer Solution**

| pectin concn (% w/v) |                   | viscosity (cP) at      |           |          |           |           |
|----------------------|-------------------|------------------------|-----------|----------|-----------|-----------|
|                      |                   | pH 2                   | pH 3      | pH 4     | pH 5      | pH 6      |
| 0.33                 | no heat           | *1.23g <sup>a</sup>    | *51.3c    | 4.24gf   | 3.98gf    | *3.47g    |
|                      | heat <sup>c</sup> | 38.95(fg) <sup>b</sup> | 13.2(fg)  | 3.60(g)  | 3.21(g)   | 1.78(g)   |
| 0.67                 | no heat           | *1.34g                 | *3867.7b  | 9.84egf  | *9.32egf  | *8.61egf  |
|                      | heat              | 776.5(c)               | 590.3(d)  | 7.39(g)  | 7.65(g)   | 4.50(g)   |
| 1                    | no heat           | *1.80g                 | * >31000a | *22.9ed  | *21.9edf  | *18.2defg |
|                      | heat              | 7300.5(a)              | 2477.5(b) | 17.3(fg) | 15.65(fg) | 7.14(g)   |

<sup>a</sup> Means followed by different letters differ at  $p < 0.05$ . <sup>b</sup> Means followed by different letters in parentheses differ at  $p < 0.05$ . <sup>c</sup> Heat treatment: 100 °C for 5 min. \*Significant difference between heat and no heat treatment at  $p < 0.05$ .

To determine heat reversibility of gelling, gelled pectin was reheated with stirring at 100 °C for 2 min to remelt the content. The solution was allowed to set again. The texture of resulting gels was measured using the method described.

The textural profile of the jellies was determined by using a modification of the procedure of Friedman et al. (1963). A 5 kg weight beam was used. The plunger moved 1.5 cm and impinged 1.15 cm (75% of the gel depth) into the gels. The speed of the cross head in the Instron machine and chart speed were 20 mm/min.

**Statistical Analysis.** The effects of pH, buffer solution, buffer concentration, pectin concentration, and heating on the viscosity of sunflower pectin were analyzed by using the factorial design method, using the SAS program. Duncan's multiple range test was used to detect differences among all treatments and heat reversibility except the difference between the two treatments heated for 5 min and sheared and reheated for 2 min were organized using the Student *t*-test method. A 0.05 probability level was used to determine the significance of statistics. All experiments in this study were performed in duplicate.

## RESULTS AND DISCUSSION

**Effect of pH on Viscosity.** The viscosities of sunflower pectin at various pH values are shown in Tables 1–3. The pH significantly affected the viscosity of sunflower pectin. At pH 3, the viscosities of unheated sunflower pectin were higher than those of pectin at pH 2, 4, 5, and 6 in citrate and SHMP buffers at all three concentrations. The viscosities of unheated sunflower pectin were low at pH 2, 4, 5, and 6. It is not clear why SP had the highest viscosity at pH 3. After the pectin solution was heated at 100 °C for 5 min, the viscosity of pectin had a much greater change at pH 2 and 3 than at pH 4, 5, and 6. Generally, viscosities of sunflower pectin in citrate and SHMP buffers decreased with heating at pH 3 and increased at pH 2. The reasons

**Table 3. Effect of pH Value, Pectin Concentration, and Heating on Viscosity of Sunflower Pectin in 0.1 M Tripolyphosphate Buffer Solution**

| pectin concn (% w/v) |                   | viscosity (cP) at    |          |           |          |          |
|----------------------|-------------------|----------------------|----------|-----------|----------|----------|
|                      |                   | pH 2                 | pH 3     | pH 4      | pH 5     | pH 6     |
| 0.33                 | no heat           | *1.13g <sup>a</sup>  | *1.03g   | 3.86g     | 3.98gf   | 4.88gf   |
|                      | heat <sup>c</sup> | 7.20(e) <sup>b</sup> | 66.6(d)  | 3.47(e)   | 4.63(e)  | 3.80(e)  |
| 0.67                 | no heat           | 1.54g                | *3.47g   | *9.56efg  | *10.2efg | *10.8efg |
|                      | heat              | — <sup>d</sup>       | 570.9(b) | 6.75(e)   | 5.78(e)  | 5.14(e)  |
| 1                    | no heat           | *1.54g               | *80.0a   | *17.9defg | *54.5b   | *31.0c   |
|                      | heat              | 1126.0(a)            | 190.3(c) | 9.38(f)   | 10.7(de) | 6.94(e)  |

<sup>a</sup> Means followed by different letters differ at  $p < 0.05$ . <sup>b</sup> Means followed by different letters in parentheses differ at  $p < 0.05$ . <sup>c</sup> Heat treatment: 100 °C for 5 min. \*Significant difference between heat and no heat treatment at  $p < 0.05$ . <sup>d</sup> —, viscosity can not be measured because the pectin sample did not solubilize completely in the buffer.

for the increase at pH 2 might be due to the low solubility of pectin in pH 2 without heat. Heating helped solubilize sunflower pectin, whereas sunflower pectin was solubilized completely at pH 3. Heating at pH 3 might have caused a degradation of pectin polymer chain to decrease the viscosity. However, viscosities of the sunflower pectin in the tripolyphosphate buffer were low at all pH values.

The sunflower pectin viscosities with heat (100 °C for 2 min) and unheated treatment were higher than those of commercial high-methoxyl apple pectin at pH 3 (Miyamoto and Chang, 1992). The effect of pH is not entirely predictable, since it depends on the DM and any salts that may be present. Small amounts of sodium chloride increased greatly the viscosity of a 0.8%, 40 DM pectin solution when the pH was 2.9, but the viscosity decreased at pH 4.4. At still higher pH values (about 6), salt effects were minimized if phosphates were also added (Glicksman, 1969). It is believed that viscosity depends on the length and shape of the pectin chains (Bender, 1959). Sunflower pectin with a high viscosity at an acidic range had a high potential to be used to thicken some foods or increase the mouthfeel of some fruit juices and beverages.

**Effect of Pectin Concentration on Viscosity.** Pectin concentration had a significant effect on the viscosity of sunflower pectin (Tables 1–3). The viscosity of sunflower pectin increased with the increase of pectin concentration in all three buffer solutions and at all pH values. The extent of increase by concentration was the greatest at pH 3 for unheated sunflower pectin and at pH 2 for heated sunflower pectin. The unheated sunflower pectin had the highest viscosity at pH 3 and pectin concentration of 1%. The value of viscosity was >31 000 cP, which exceeded the maximum capacity of the viscometer.

**Effect of Heating on Viscosity.** Temperature is an important factor affecting the viscosity of pectin. The heat treatment decreased or increased the viscosity greatly at pH 2 and 3 (Tables 1–3), depending on the solubility of the pectin in the buffer solutions. The viscosity of sunflower pectin slightly decreased at pH 4, 5, and 6 after heating and decreased greatly at pH 3 in sodium citrate and SHMP buffers. The viscosity increased at pH 2 in all three buffers and at pH 3 in sodium tripolyphosphate buffer after heating. The increase in viscosity might be due to solubilization of pectin by heating. Generally, the sunflower pectin did not dissolve completely in all three buffer solutions at pH 2. After the pectin solution was heated, the viscosity

**Table 4. Effect of Different Pectin Concentration, Buffer Type, and Heating on Viscosity of Sunflower Pectin in 0.1 M Buffer Solutions at pH 3**

| pectin concn (% w/v) |                   | viscosity (cP)       |           |                  |
|----------------------|-------------------|----------------------|-----------|------------------|
|                      |                   | sodium citrate       | SHMP      | tripolyphosphate |
| 0.33                 | no heat           | *77.6d <sup>a</sup>  | *51.3e    | *1.03f           |
|                      | heat <sup>c</sup> | 5.78(e) <sup>b</sup> | 13.2(de)  | 66.6(d)          |
| 0.67                 | no heat           | *4380.3b             | *3867.7c  | *3.47f           |
|                      | heat              | 155.3(c)             | 590.3(b)  | 570.9(b)         |
| 1                    | no heat           | >31000a              | >31000a   | *80.0d           |
|                      | heat              | 598.0(b)             | 2477.5(a) | 190.3(c)         |

<sup>a</sup> Means followed by different letters differ at  $p < 0.05$ . <sup>b</sup> Means followed by different letters in parentheses differ at  $p < 0.05$ . <sup>c</sup> Heat treatment: 100 °C for 5 min. \*Significant difference between heat and no heat treatment at  $p < 0.05$ .

**Table 5. Effect of Buffer Type, Buffer Concentration, and Heating on Viscosity of Sunflower Pectin (at 0.67% Concentration) in Three Buffer Solutions at pH 3**

| buffer concn (M) |                   | viscosity (cP)          |          |                  |
|------------------|-------------------|-------------------------|----------|------------------|
|                  |                   | sodium citrate          | SHMP     | tripolyphosphate |
| 0.01             | no heat           | *935.9g <sup>a</sup>    | *1514.5f | *1584.4e         |
|                  | heat <sup>c</sup> | 18561.5(b) <sup>b</sup> | 128.2(h) | 148.0(hi)        |
| 0.05             | no heat           | *2135.8d                | *3.75h   | — <sup>d</sup>   |
|                  | heat              | 22910.5(a)              | 250.0(g) | 535.9(d)         |
| 0.1              | no heat           | *4380.3b                | *3867.7c | *3.47h           |
|                  | heat              | 155.3(hi)               | 590.3(d) | 571.0(d)         |
| 0.2              | no heat           | *8581.9a                | *1.85h   | *1.70h           |
|                  | heat              | 6446.1(c)               | 446.5(e) | 318.0(f)         |

<sup>a</sup> Means followed by different letters differ at  $p < 0.05$ . <sup>b</sup> Means followed by different letters in parentheses differ at  $p < 0.05$ . <sup>c</sup> Heat treatment: 100 °C for 5 min. \*Significant difference between heat and no heat treatment at  $p < 0.05$ . <sup>d</sup> —, means that viscosity cannot be measured because the pectin sample did not solubilize completely in the buffer.

of sunflower pectin would increase greatly at pH 2 because heat treatment increased the solubility of pectin and therefore increased the actual concentration in the solution. Miyamoto and Chang (1992) reported the viscosity of citrus high-methoxyl pectin was decreased by heating. However, the viscosities of sunflower pectin in 0.5 M sodium citrate buffer were not greatly affected by heating. In this study, buffer concentrations ranged from 0.01 to 0.2 M. Therefore, the flow behavior of sunflower pectin differed at a higher buffer concentration, such as 0.5 M.

**Effect of Buffer Type on Viscosity.** Table 4 compares the viscosities of sunflower pectin in three buffer solutions at pH 3, buffer concentration 0.1 M, and three pectin concentrations. In all pectin concentrations, the sunflower pectin had the higher viscosity in sodium citrate and SHMP buffers than in tripolyphosphate buffer. If heat treatment was considered, sunflower pectin in the sodium citrate buffer had the highest viscosity among the three pectin concentrations. The range of viscosity values was from 77.6 to >31 000 cP and from 51.3 to >31 000 cP in sodium citrate buffer and SHMP buffer without heat treatment, respectively. At all buffer concentrations, pH 3, and pectin concentration 0.67% (Table 5), sunflower pectin had the highest values of viscosity in sodium citrate among the three buffers if the heat treatment of pectin was involved.

**Effect of Buffer Concentration on Viscosity.** At 1% concentration, viscosities of pectin were greater than the capacity of the viscometer; therefore, the concentration of the pectin at 0.67% was chosen for buffer concentration comparisons. The effect of buffer concen-

**Table 6. Effect of pH Value, Buffer Concentration, and Heating on Viscosity of Sunflower Pectin (at 0.67% Concentration) in Sodium Citrate Buffer Solution**

| buffer concn (M) |                   | viscosity (cP) at    |            |           |         |         |
|------------------|-------------------|----------------------|------------|-----------|---------|---------|
|                  |                   | pH 2                 | pH 3       | pH 4      | pH 5    | pH 6    |
| 0.01             | no heat           | *1.23g <sup>a</sup>  | *935.9d    | *8.95g    | 9.89g   | 7.07g   |
|                  | heat <sup>c</sup> | 15.5(f) <sup>b</sup> | 18561.5(b) | 54.4(ef)  | 8.29(f) | 6.37(f) |
| 0.05             | no heat           | *1.23g               | *2135.8c   | *423.3f   | 8.42g   | 6.62g   |
|                  | heat              | 35.7(f)              | 22910.5(a) | 104.8(e)  | 7.45(f) | 5.91(f) |
| 0.1              | no heat           | *1.36g               | *4380.3b   | *15.6g    | 6.96g   | 7.45g   |
|                  | heat              | 8931.0(c)            | 155.3(e)   | 4.31(f)   | 5.91(f) | 5.21(f) |
| 0.2              | no heat           | *1.23g               | *8581.9a   | *574.8e   | 10.3g   | 3.86g   |
|                  | heat              | 31.1(f)              | 6446.1(d)  | 108.7(ef) | 8.81(f) | 6.24(f) |

<sup>a</sup> Means followed by different letters differ at  $p < 0.05$ . <sup>b</sup> Means followed by different letters in parentheses differ at  $p < 0.05$ . <sup>c</sup> Heat treatment: 100 °C for 5 min. \*Significant difference between heat and no heat treatment at  $p < 0.05$ .

**Table 7. Effect of pH Value, Buffer Concentration, and Heating on Viscosity of Sunflower Pectin (at 0.67% Concentration) in SHMP Buffer Solution**

| buffer concn (M) |                   | viscosity (cP) at  |                       |          |         |         |
|------------------|-------------------|--------------------|-----------------------|----------|---------|---------|
|                  |                   | pH 2               | pH 3                  | pH 4     | pH 5    | pH 6    |
| 0.01             | no heat           | 1.39d <sup>a</sup> | *1514.5b              | 11.57d   | 7.32d   | *8.81d  |
|                  | heat <sup>c</sup> | — <sup>d</sup>     | 128.2(e) <sup>b</sup> | 10.2(f)  | 6.62(f) | 2.77(f) |
| 0.05             | no heat           | 1.62d              | *3.75d                | *32.65c  | 8.42d   | *9.06d  |
|                  | heat              | —                  | 250.0(d)              | 12.85(f) | 7.71(f) | 4.82(f) |
| 0.1              | no heat           | *1.30d             | *3867.7a              | *9.85d   | *9.30d  | *8.60d  |
|                  | heat              | 776.5(a)           | 590.3(b)              | 7.40(f)  | 7.65(f) | 4.50(f) |
| 0.2              | no heat           | 2.06d              | *1.85d                | —        | *16.4cd | *10.6d  |
|                  | heat              | —                  | 446.5(c)              | 11.45(f) | 8.55(f) | 6.04(f) |

<sup>a</sup> Means followed by different letters differ at  $p < 0.05$ . <sup>b</sup> Means followed by different letters in parentheses differ at  $p < 0.05$ . <sup>c</sup> Heat treatment: 100 °C for 5 min. \*Significant difference between heat and no heat treatment at  $p < 0.05$ . <sup>d</sup> —, viscosity cannot be measured because the pectin sample did not solubilize completely in the buffer.

**Table 8. Effect of pH Value, Buffer Concentration, and Heating on Viscosity of Sunflower Pectin (at 0.67% Concentration) in Tripolyphosphate Buffer Solution**

| buffer concn (M) |                   | viscosity (cP) at     |          |           |            |           |
|------------------|-------------------|-----------------------|----------|-----------|------------|-----------|
|                  |                   | pH 2                  | pH 3     | pH 4      | pH 5       | pH 6      |
| 0.01             | no heat           | *1.44r <sup>a</sup>   | *1584.4a | *11.7b    | *8.10h     | *7.97i    |
|                  | heat <sup>c</sup> | 202.0(d) <sup>b</sup> | 148.0(e) | 8.35(fg)  | 6.94(ghi)  | 4.50(hi)  |
| 0.05             | no heat           | *1.54q                | —        | *6.43j    | 8.74f      | *8.61g    |
|                  | heat              | — <sup>d</sup>        | 535.9(b) | 10.7(f)   | 7.45(gh)   | 3.98(i)   |
| 0.1              | no heat           | *154q                 | *3.47k   | *9.58e    | *10.2d     | *10.8c    |
|                  | heat              | —                     | 570.1(a) | 6.75(ghi) | 5.78(ghi)  | 5.14(ghi) |
| 0.2              | no heat           | *2.83l                | *1.70p   | *2.70m    | *2.06n     | *1.85o    |
|                  | heat              | —                     | 318.0(c) | 6.94(ghi) | 85.14(ghi) | 4.37(hi)  |

<sup>a</sup> Means followed by different letters differ at  $p < 0.05$ . <sup>b</sup> Means followed by different letters in parentheses differ at  $p < 0.05$ . <sup>c</sup> Heat treatment: 100 °C for 5 min. \*Significant difference between heat and no heat treatment at  $p < 0.05$ . <sup>d</sup> —, viscosity cannot be measured because the pectin sample did not solubilize completely in the buffer.

tration on viscosity depended on pH, buffer type, and heating (Tables 6–8). In sodium citrate buffer solution, the viscosity of nonheated pectin at pH 3 increased with the increase of buffer concentration. The pectin had the highest viscosity without heat treatment at pH 3 and buffer concentration 0.2 M and with heat treatment at pH 3 and buffer concentration 0.05 M.

In SHMP buffer solution, the pectin had the highest viscosity without heat treatment at pH 3 and buffer concentration 0.1 M and with heat treatment at pH 2

**Table 9. Effect of Heat Treatment on Viscosity of Sunflower Pectin (at 0.67% Concentration) in Sodium Citrate Buffer Solution at Different pH Values**

| buffer concn (M) |                       | viscosity (cP) at |          |       |      |      |
|------------------|-----------------------|-------------------|----------|-------|------|------|
|                  |                       | pH 2              | pH 3     | pH 4  | pH 5 | pH 6 |
| 0.01             | heat (5) <sup>a</sup> | 15.5              | *18561.5 | 54.4  | 8.29 | 6.37 |
|                  | heat (2) <sup>b</sup> | — <sup>c</sup>    | 36.5     | 50.2  | 5.65 | 5.46 |
| 0.05             | heat (5)              | 35.7              | *22910.5 | 104.8 | 7.45 | 5.91 |
|                  | heat (2)              | —                 | 23.13    | 97.0  | 6.70 | 5.14 |
| 0.2              | heat (5)              | 31.1              | *6446.1  | 108.7 | 8.81 | 6.24 |
|                  | heat (2)              | —                 | 44.9     | 92.4  | 5.55 | 4.55 |

<sup>a</sup> Heat treatment: 100 °C for 5 min. <sup>b</sup> Heat treatment: 100 °C for 5 min, sheared and heated at 100 °C for 2 min again. <sup>c</sup> —, viscosity cannot be measured because the pectin sample did not solubilize completely in the buffer. \*Significant difference between heating 5 min and 2 min at  $p < 0.05$ .

**Table 10. Effect of Heat Treatment on Viscosity of Sunflower Pectin (at 0.67% Concentration) in SHMP Buffer Solution at Different pH Values**

| buffer concn (M) |                       | viscosity (cP) at |        |       |      |      |
|------------------|-----------------------|-------------------|--------|-------|------|------|
|                  |                       | pH 2              | pH 3   | pH 4  | pH 5 | pH 6 |
| 0.01             | heat (5) <sup>a</sup> | — <sup>c</sup>    | 128.2  | 10.22 | 6.62 | 2.77 |
|                  | heat (2) <sup>b</sup> | —                 | 116.7  | 8.65  | 5.00 | 2.76 |
| 0.05             | heat (5)              | —                 | *250.0 | 12.85 | 7.71 | 4.82 |
|                  | heat (2)              | —                 | 180.6  | 9.64  | 6.43 | 3.73 |
| 0.2              | heat (5)              | —                 | *446.5 | 11.45 | 8.55 | 6.04 |
|                  | heat (2)              | —                 | 330.1  | 9.39  | 7.01 | 4.76 |

<sup>a</sup> Heat treatment: 100 °C for 5 min. <sup>b</sup> Heat treatment: 100 °C for 5 min, sheared and heated at 100 °C for 2 min again. <sup>c</sup> —, viscosity cannot be measured because the pectin sample did not solubilize completely in the buffer. \*Significant difference between heating 5 min and 2 min at  $p < 0.05$ .

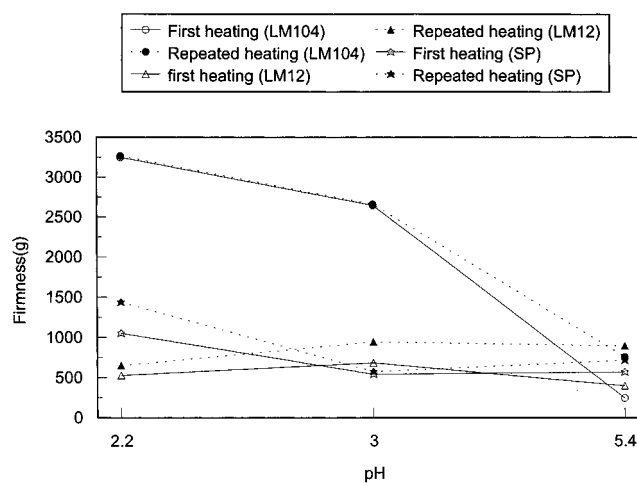
**Table 11. Effect of Heat Treatment on Viscosity of Sunflower Pectin (at 0.67% Concentration) in Tripolyphosphate Buffer Solution at Different pH Values**

| buffer concn (M) |                       | viscosity (cP) at |        |      |      |      |
|------------------|-----------------------|-------------------|--------|------|------|------|
|                  |                       | pH 2              | pH 3   | pH 4 | pH 5 | pH 6 |
| 0.01             | heat (5) <sup>a</sup> | *2.02             | 148.0  | 8.35 | 6.94 | 4.50 |
|                  | heat (2) <sup>b</sup> | 217.4             | 178.6  | 7.26 | 7.13 | 4.24 |
| 0.05             | heat (5)              | — <sup>c</sup>    | *535.9 | 10.7 | 7.45 | 3.98 |
|                  | heat (2)              | —                 | 64.5   | 8.10 | 5.53 | 2.89 |
| 0.2              | heat (5)              | —                 | *318.0 | 6.94 | 5.14 | 4.37 |
|                  | heat (2)              | —                 | 68.4   | 7.39 | 6.56 | 6.04 |

<sup>a</sup> Heat treatment: 100 °C for 5 min. <sup>b</sup> Heat treatment: 100 °C for 5 min, sheared, and heated at 100 °C for 2 min again. <sup>c</sup> —, viscosity cannot be measured because the pectin sample did not solubilize completely in the buffer. \*Significant difference between heating 5 min and 2 min at  $p < 0.05$ .

and buffer concentration 0.1 M. In sodium tripolyphosphate buffer solution, the pectin had the highest viscosity without heat treatment at pH 3 and buffer concentration 0.01 M and with heat treatment at pH 3 and buffer concentration 0.1 M. The viscosities of pectin at pH 4, 5, and 6 were lower in all three buffer solutions and at all buffer concentrations than that measured at pH 2 and 3.

**Effect of Shearing and Repeated Heating on Viscosity.** The effect of shearing and repeated heating on the viscosity of sunflower pectin depended upon the pH, buffer type, and buffer concentration used (Tables 9–11). In three buffers, at three buffer concentrations and pectin concentration 0.67%, the viscosity of sunflower pectin with heat treatment at 100 °C for 5 min was not significantly different from that of sunflower pectin with shearing and repeated heat treatment at

**Figure 1.**

100 °C for 2 min at pH from 4 to 6, and the viscosity significantly decreased at pH 3. Therefore, the viscosity of sunflower pectin was only partially heat reversible at pH 3.

Pectin molecules were believed to exist in rodlike aggregates, which could undergo disaggregation under various conditions (Fishman et al., 1986). The behavior of citrus pectin in solution was very complex and was affected by several factors such as pH, ionic strength, buffer concentration, molecular mass, degree of esterification, method of pectin purification, and chemical modification (Fishman et al., 1984). Further molecular characterizations are needed to explain why sunflower pectin viscosity varied in the ways observed in this study.

#### Heat Reversibility of Sunflower Pectin Jellies.

The importance of LM pectin to the food industry lies in part the unique ability of its solutions to yield thermoreversible gels in the presence of a dehydrating agent at a pH at or near 3 or in the presence of the calcium ion (Copenhagen Pectin A/S, 1992). The heat reversibility of LM pectin gels may be utilized in bakery jams and jellies for glazing. When pectin gels are heated and remelted to be poured on top of cakes, the LM pectin gels form a coherent and glossy glazing.

The firmness of the gels formed by SP, LM12, and LM104 without and with repeated heat treatment is shown in Figure 1. At pH 2.2 and 5.4, the firmness of the gel formed by SP significantly increased after the pectin gel was reheated at 100 °C for 2 min and had no significant change at pH 3. This indicated that SP gel had a good heat reversibility at the three pH conditions.

Compared with SP at three pH levels, LM12 gels increased significantly in firmness after the gels were reheated at 100 °C for 2 min and had heat reversibility in firmness. For LM104 gels, the firmness was significantly greater after the pectin gels were reheated than that without reheating at pH 5.4 and was not significantly changed at pH 2.2 and 3 due to the heat treatment. Therefore, LM104 gels also had heat reversibility in gel firmness at the three pH levels tested.

**Conclusions.** Sunflower pectin had the highest viscosity at pH 3 in the three buffer systems tested. The higher the concentration of sunflower pectin, the higher the viscosity of sunflower pectin. The effect of heating on the viscosity of sunflower pectin was more significant at pH 2 and 3 than at pH 4, 5, and 6, usually, the viscosity of sunflower pectin decreased if pectin was dissolved completely and increased if the pectin was partially dissolved after the pectin solution was heated

at 100 °C for 5 min. The effect of buffer concentration on the viscosity of sunflower pectin is different in the differently treated solutions. The viscosity of sunflower pectin was only partially heat reversible at pH 3. Sunflower head pectin has a high potential as a thickening agent for commercial food applications at an acidic range. The resulting gels were heat reversible and could be considered for use in some food applications that require reheating to produce low-calorie foods.

#### ABBREVIATIONS USED

DM, degree of methylation; LM, low-methoxyl; SHMP, sodium hexametaphosphate; SP, sunflower low-methoxyl pectin.

#### LITERATURE CITED

- Bender, W. A. In *Industrial Gums*; Whistler, R. L., Ed.; Academic Press: New York, 1959; pp 397–424.
- Chang, K. C.; Miyamoto, A. Gelling characteristics of pectin from sunflower head residues. *J. Food Sci.* **1992**, *57*, 1435–1438, 1443.
- Copenhagen Pectin A/S. *Handbook for Pectin General Description*; Copenhagen Pectin, Lille Skensved: Copenhagen, Denmark, 1992; pp 32–33.
- Fishman, M. L.; Pfeffer, P. E.; Barford, R. A.; Doner, L. W. Studies of pectin solution properties by high-performance size exclusion chromatography. *J. Agric. Food Chem.* **1984**, *32*, 372–378.
- Fishman, M. L.; Pepper, L.; Damert, W. C.; Phillips, J. G.; Barford, R. A. A critical reexamination of molecular weight and dimensions for citrus pectins. In *Chemistry and Function of Pectins*; Fishman, M. L., Jen, J. J., Eds.; American Chemical Society: Washington, DC, 1986; pp 22–37.
- Friedman, H. H.; Whitney, J. E.; Szczesniak, A. S. The texturometer—a new instrument for objective texture measurement. *J. Food Sci.* **1963**, *28*, 390–396.
- Glicksman, M. Pectins. In *Gum Technology in the Food Industry*; Academic Press: Tarrytown, NY, 1969; pp 159–192.
- Knowles, P. F. Morphology and anatomy. In *Sunflower Science and Technology*; Carter, J. F., Ed.; American Society of Agronomy: Madison, WI, 1978; pp 55–60.
- Miyamoto, A.; Chang, K. C. Extraction and physiochemical characterization of pectin from sunflower head residues. *J. Food Sci.* **1992**, *57*, 1439–1443.
- National Research Council. Pectin. In *Food Chemical Codex*, 2nd ed.; National Academy of Sciences: Washington, DC, 1972.
- Nelson, D. B.; Smit, C. J. B.; Wiles, R. R. In *Food Colloids*; Graham, H. D., Ed.; AVI Publishing: Westport, CT, 1977; pp 418–437.
- Sosulski, F. W.; Lin, M. J. Y.; Humbert, E. S. Gelation characteristics of acid-precipitated pectin from sunflower heads. *Can. Inst. Food Sci. Technol. J.* **1978**, *11* (3), 113–116.

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