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Studying the Interaction of Xanthan Gum and Pectin with Some Functional Carbohydrates on the Rheological Attributes of a Low-Fat Spread

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GRAPHICAL ABSTRACT

Effects of xanthan gum (XG) (0.1 wt%) and pectin (PE) (0.5 wt%) alone and in combination with different concentrations (0.2 and 0.4 wt%) of locust bean gum (LBG), modified starch (MS), and Na-alginate (ALG) on some of the rheological characteristics of low-fat spreads, including flow behavior curves, rheological modeling, apparent viscosity, rheological modules (storage modulus ($G'$) and loss modulus ($G''$)), and delta degree ($G''/G'$) were studied. Results showed the power-law model was better than the Herschel– Bulkley model to describe the flow curve of dispersions. The $k$-value in the power-law model increased with increase in biopolymers concentration in solution. All samples exhibited shear-thinning flow behavior with a low yield stress. Dynamic oscillatory shear test showed that the spreads had a viscoelastic solid behavior with a gel-like structure. The $G'$ value was increased by increasing frequency from 0.03 to 15 Hz, while the $G''$ and $G''/G'$ values decreased. Also, MS in combination with XG and PE led to increase the $G'$ values of spreads in comparison with ALG and LBG. Moreover, microstructural and stability observations revealed that the spreads prepared with 0.1% XG-0.2% LBG significantly had the highest oiling out.

Keywords Carbohydrate biopolymers, dynamic rheology, locust bean gum, power-law model, structural characteristics, water-in-oil spread

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Color versions of one or more of the figures in the article can be found online at www.tandfonline.com/ldis.
1. INTRODUCTION

Consumers are exercising more, eating healthy foods, and decreasing the foods consumption with high fat content. To respond to this consumers’ demand, the food industry has offered reduced-fat types of standard food products. World consumption of some fatty foods as butter has decreased during recent years, while spreads are more often chosen by the consumers. In order to prepare the different formulations of low-fat spreads, it is necessary to apply a combination of nonfat ingredients with different functional roles to replace the quality characteristics lost when fat is removed. Manufacturing of these low-fat spreads with a considerably larger aqueous phase seriously requires the use of biopolymers like polysaccharides as thickeners or gelling agents. The ability of carbohydrate biopolymers to cross-link and at high enough contents to form a tangled, interconnected molecular network in water is extensively known.

Fat reduction and the replacement of fat with an appropriate fat substitute into a spread structure affect the rheological attributes. Thus, the determination of rheological properties of these products can help many researchers for designing and modeling purposes, food product development, quality control, sensory evaluation, consumer acceptability, and long-term stability purposes.

Alexa et al. by studying the rheological, microstructural, and textural characteristics of water-in-oil (W/O) spreads containing κ-carrageenan found that concentration of this biopolymer had a critical role in their physical stability. They also showed that κ-carrageenan had a key role in controlling the rheology of the aqueous phase of W/O spreads during spread formation, storage, and subsequent reheating. Another study conducted by showed that the incorporation of both types of maltodextrin gel as fat replacers in reduced-fat spreads led to a substantial increase in values of thixotropy degree, apparent viscosity, elastic moduli, and firmness. Mousazadeh et al. also characterized stability and rheology of pistachio oil-spreads stabilized by xanthan gum (XG). Results of these researchers revealed that all the rheological attributes and also physical stability were increased by adding XG to the spreads.

To the best of our knowledge, there is a lack of information available on the interaction effect of various carbohydrate biopolymers on the rheological characteristics of low-fat spreads. Thus, the aim of the present research was to evaluate the rheological performance of some carbohydrate biopolymers such as alginate (ALG), modified starch (MS), locust bean gum (LBG), XG, and pectin (PE) in order to improve the structural and physical stability of low-fat spreads.

2. MATERIALS AND METHODS

2.1. Materials

Normal butter (82% fat content) was provided by Pegah Co. (Tehran, Iran). Distilled monoglycerides (DMG) 0291 and polyglycerol polryricinoleate (PGPR) 4175 were supplied by Emulsion-Holland B.V. (Zierikzee, Netherlands). XG and LBG were obtained from Sigma-Aldrich Chemical Co. (St. Louis, MO, United States). Powdered starch (ST), sodium-ALG, Citrus PE, lactic acid, and natamycin (pimaricin) as an antifungal agent were purchased from Merck Chemical Co. (Darmstadt, Germany).

2.2. Sample Preparation

The low-fat spreads were produced by preparing two phases of oil and water. The fat-soluble ingredients including surfactants of DMG 0291 (0.6% w/w) and PGPR 4175 (0.2% w/w) were added to melted butter (40% w/w). To prepare the water phase, natamycin (250 ppm), PE (0.5% w/w), XG (0.5% w/w), MS (0, 0.2, and 0.4% w/w), LBG (0, 0.2, and 0.4% w/w), and ALG (0, 0.2 and 0.4% w/w) for the various formulations (Table 1) were dispersed in deionized water (≈70°C) acidified by lactic acid (pH = 4.5), using a blender (IKA-WERK, RW 20 DZM, Staufen, Germany). While the oil phase was mixing, the prepared water phase was slowly added to the oil phase to form an initial coarse emulsion. The obtained mixture was transferred for 2 hours to a laboratory-scale conch in order to achieve the suitable viscosity and satisfactory texture and flavor for spreads. Interactions between disperse and continuous phases at the end of the conching stage were promoted, and different formulations were then hot-filled into special containers, cooled and kept at 4°C for further analyses. Fourteen spreads with different formulations were prepared to investigate the rheological characteristics (Table 1).

2.3. Dynamic Rheological Measurements

Rheological characteristics including steady shear viscosity, frequency, and amplitude sweep oscillatory shear tests were carried out at 24.0 ± 0.1°C using a dynamic rheometer (MCR 301, Anton Paar Germany GmbH, Ostfildern-Scharnhausen, Germany). Viscosity of low-fat spread was measured at 0.1–1001/s. Couette geometry with a cup (33.93 mm diameter) and a bob system (32.05 mm diameter, 33.29 mm length) was used. Amplitude sweep tests were performed at strain 0.01–10% with a fixed frequency of 1 Hz to determine the linear viscoelastic range, where the dynamic parameters (storage modulus (G′) and loss modulus (G″)) are independent of the magnitude of applied strain. A constant strain of 0.012% was selected for the frequency sweep test according to the obtained results. Frequency sweep test was also performed at frequency 0.03–15 Hz with a constant strain (0.012%) to investigate the dynamic rheological properties such as G′, G″, and tan δ (G″/G′). Frequency (ω) dependence of G′ and G″ was modeled with the power-law parameters a, b, x, and y described by Equations (1) and (2):

\[ G' = a\omega^x \]  
[1]
Power-law model parameters for 14 low-fat spreads produced and comparison of the quality of the fit of this model to Herschel–Bulkley model.

### Table 1

<table>
<thead>
<tr>
<th>Formulation No.</th>
<th>Composition</th>
<th>Power law $k$ (Pa s)$^n$</th>
<th>$n$</th>
<th>$R^2$</th>
<th>Herschel–Bulkley $R^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>PE 0.5%</td>
<td>5834.4 ± 708.2$^b$</td>
<td>0.656 ± 0.012$^c$</td>
<td>0.9188 ± 0.0021</td>
<td>0.9184 ± 0.0150</td>
</tr>
<tr>
<td>2</td>
<td>XG 0.1%</td>
<td>3925.9 ± 12.2$^e$</td>
<td>0.625 ± 0.041$^d$</td>
<td>0.9265 ± 0.0051</td>
<td>0.9258 ± 0.0511</td>
</tr>
<tr>
<td>3</td>
<td>PE 0.5% + MS 0.2%</td>
<td>5620.0 ± 128.6$^b$</td>
<td>0.666 ± 0.023$^c$</td>
<td>0.9258 ± 0.0092</td>
<td>0.9235 ± 0.0116</td>
</tr>
<tr>
<td>4</td>
<td>PE 0.5% + MS 0.4%</td>
<td>6712.1 ± 25.3$^a$</td>
<td>0.684 ± 0.011$^bc$</td>
<td>0.9550 ± 0.0214</td>
<td>0.9486 ± 0.0451</td>
</tr>
<tr>
<td>5</td>
<td>XG 0.1% + MS 0.2%</td>
<td>4487.5 ± 89.1$^d$</td>
<td>0.634 ± 0.005$^d$</td>
<td>0.9445 ± 0.0511</td>
<td>0.9436 ± 0.0712</td>
</tr>
<tr>
<td>6</td>
<td>XG 0.1% + MS 0.4%</td>
<td>5678.3 ± 121.3$^b$</td>
<td>0.658 ± 0.000$^e$</td>
<td>0.9544 ± 0.0027</td>
<td>0.9215 ± 0.0223</td>
</tr>
<tr>
<td>7</td>
<td>PE 0.5% + LBG 0.2%</td>
<td>2584.1 ± 18.7$^i$</td>
<td>0.574 ± 0.004$^f$</td>
<td>0.9001 ± 0.0021</td>
<td>0.8792 ± 0.0091</td>
</tr>
<tr>
<td>8</td>
<td>PE 0.5% + LBG 0.4%</td>
<td>4133.8 ± 45.2$^e$</td>
<td>0.712 ± 0.015$^b$</td>
<td>0.9724 ± 0.0041</td>
<td>0.9570 ± 0.0140</td>
</tr>
<tr>
<td>9</td>
<td>XG 0.1% + LBG 0.2%</td>
<td>2624.8 ± 66.9$^i$</td>
<td>0.703 ± 0.021$^b$</td>
<td>0.9481 ± 0.0151</td>
<td>0.9263 ± 0.0090</td>
</tr>
<tr>
<td>10</td>
<td>XG 0.1% + LBG 0.4%</td>
<td>3630.6 ± 21.6$^f$</td>
<td>0.772 ± 0.037$^a$</td>
<td>0.9882 ± 0.0410</td>
<td>0.9786 ± 0.0412</td>
</tr>
<tr>
<td>11</td>
<td>PE 0.5% + ALG 0.2%</td>
<td>2854.1 ± 74.2$^h$</td>
<td>0.624 ± 0.005$^d$</td>
<td>0.9840 ± 0.0061</td>
<td>0.9542 ± 0.0261</td>
</tr>
<tr>
<td>12</td>
<td>PE 0.5% + ALG 0.4%</td>
<td>4867.3 ± 21.1$^e$</td>
<td>0.683 ± 0.030$^bc$</td>
<td>0.9733 ± 0.0320</td>
<td>0.9632 ± 0.0310</td>
</tr>
<tr>
<td>13</td>
<td>XG 0.1% + ALG 0.2%</td>
<td>3062.7 ± 44.4$^g$</td>
<td>0.523 ± 0.009$^f$</td>
<td>0.9151 ± 0.0520</td>
<td>0.8841 ± 0.0031</td>
</tr>
<tr>
<td>14</td>
<td>XG 0.1% + ALG 0.4%</td>
<td>4968.2 ± 101.3$^c$</td>
<td>0.648 ± 0.007$^c$</td>
<td>0.9346 ± 0.0091</td>
<td>0.9342 ± 0.0046</td>
</tr>
</tbody>
</table>

$^a$Values with the same letter within a column ($k$ and $n$) are not significantly different at $p < 0.05$.

$b$, Consistency coefficient; $n$, flow behavior index; $R^2$, determination coefficient.

\[ G' = b\gamma^n \]

Power-law (Equation 3) and Herschel–Bulkley (Equation (4)) models were applied to obtain the better flow curves for the produced spreads\[^8\]:

\[ \sigma = k(\gamma^n) \]

\[ \sigma = \sigma_0 + k(\gamma^n) \]

where $\sigma$, $\sigma_0$, $\gamma$, $k$, and $n$ are shear stress, yield stress, shear rate, consistency coefficient, and flow behavior index, respectively.

Each experiment was triplicated using freshly prepared samples, and the values reported are means of these repetitions.

### 2.4. Stability Evaluation

Stability or oiling out (OO) of the different samples was measured as the ratio of oil volume over total volume for each sample upon standing.\[^8\] Briefly, 50.0 g of each sample was carefully poured into glass cylinders (100 ml) and then stored at 21 ± 1°C for 3 months. The OO of each sample was expressed as a percentage of the total weight of the butter in the tube (Equation (5)):

\[ OO = \frac{M_{oil}}{M_{total}} \times 100 \]

### 2.5. Light Microscopic Observations

The microstructures of the low-fat spreads were observed using microscopy as previously described by Gharibzahedi et al.\[^9\] Images were made immediately after spread preparation at temperature room. A drop of molten sample was diluted with 1 g/1 SDS solution and placed on a microscope slide preheated at 50°C, covered with a cover slip. Microstructure was visualized by phase-contrast light microscopy (Leica Galen III, Wetzlar, Germany) at 40× magnifications and recorded by means of a with a macro zoom lens (Mavica FD88, Sony, Tokyo, Japan) mounted on the microscope.

### 2.6. Data Analysis

The obtained data were subjected to analysis of variance using SPSS 13 software (SPSS Inc., Chicago, IL, USA). The F-test was used to determine significant effects of applied carbohydrates type and their contents. The means were compared using the Duncan’s multiple ranges test at a significant level of $P < 0.05$. Drawing the graphs and fitting models was performed by Microsoft Office Excel 2007.

### 3. RESULTS AND DISCUSSION

#### 3.1. Flow Curves and Rheological Models

Study of flow behavior of the prepared low-fat spreads showed that they had a non-Newtonian shear-thinning
behavior. Thus, a nonlinear increase in the amount of shear stress by the increasing shear rate was found. This fact is clearly illustrated in Table 1, as all the samples had a flow behavior index ($n$-value) in the range 0.523–0.772. This pseudoplastic nature demonstrated the reduction of apparent viscosity by enhancing shear rate because of the disruption of formed aggregates in a three-dimensional network. Mun et al.[10] revealed that an increase in external shear forces on this emulsion system leads to progressive deformation and disruption of the aggregated particles, which in turn decreases emulsion resistance to flow and reduces its apparent viscosity over time. In the current work, it was observed that the spreads had a low yield stress in their flow curves. Other researchers pointed out that this force is needed to break down the intermolecular interactions among the dispersed particles in order to initiate flow.[8,11]

According to Equations (3) and (4), Herschel–Bulkley and power-law models were used to find the better flow curves for the spreads formulated with different carbohydrate biopolymers. Table 1 shows power-law model with a high determination of coefficient ($R^2$, 0.9001–0.9882) was the best equation to fit the shear-rate/shear-stress data. The spreads containing 0.5% PE and 0.4% MS had the highest $k$-value (Table 1). A high consistency value was also obtained for low-fat spreads formulated with 0.5% PE and 0.1% XG–0.4% MS (Table 1). Higher ability of PE than XG is probably due to the higher amounts applied of this carbohydrate in low-fat spreads as increasing the PE molar mass promotes the formation of strong gels in these systems through hydrogen bindings.[12] Increasing the consistency of spreads using MS also can be attributed to the swelling and rupturing of its granular structure as it is suspended in water and heated on agitation. The formation of a consistent paste could improve controlling the quality of starch-based food products like spreads.[13] Moreover, the consistency can be considerably enhanced at higher concentration because of formation of a more closely packed structure via the rigidity of the granule. Thakur et al.[14] reported the formation of junction zones in high-methoxyl PE gels occurs in the “smooth regions” in the molecule and is assisted through hydrophobic interactions between the methoxyl groups and hydrogen bonds between un-dissociated carboxyl and secondary alcohol groups at acidic pH and high sugar content. High consistency of the spreads prepared with XG can be directly related to its complex molecules structure with high molecular weight (~3000 kDa) that forms the complicated aggregates by polymer entanglement and hydrogen bonds.[15] There are also a large number of free carboxyl groups in XG structure that have a high potential to absorb water and increase the consistency magnitude.[16] Thus, a strong interaction was probably happened between the some swollen starch granules and XG in the continuous phase and increased the viscosity of spreads produced with 0.1% XG–0.4% MS.[17] The results obtained in this study showed that combination of LBG at low concentration with both polysaccharides of XG and PE led to a minimum consistency ($p < 0.05$) among all the samples (Table 1). The nature of these polysaccharides and decrease of the intermolecular interactions of continuous phase trapped in the branched structure can explain the low apparent viscosity of aggregated emulsion systems.[18] Low apparent viscosity of the spreads consisting of XG with 0.2% LBG could be because of the XG conformational changes.[19] Casas and Garcia-Ochoa[20] stated that at temperatures higher than 50°C, XG conformation evolves disordered ones, although these molecules also interact with LBG, these interactions are weaker, and thus low viscosity is observed. A high OO value (36.2% w/w) was also found for this formulation. Therefore, this fact can be attributed to a decrease in viscosity amount and an increase in droplets size of this emulsion (Figure 1). Figure 1 clearly reveals that the droplets of

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**FIG. 1.** Light microscopy images of the spreads containing 0.1% XG–0.2% LBG (a) and 0.5% PE and 0.4% MS (b). The magnification bar shown applies to the micrographs.
spreads formulated with 0.1% XG–0.2% LBG were significantly greater than those prepared with 0.5% PE and 0.4% MS (the highest apparent viscosity). It seems that intra or intermolecular hydrogen bonding in the aqueous phase of spreads composed of 0.1% PE and 0.2% LBG is insufficiently formed as by the increasing concentration LBG from 0.2 to 0.4%, the consistency was significantly increased.

3.2. Viscoelastic Characteristics

Frequency sweep tests of low-fat spreads containing only XG and PE are shown in Figure 2. As shown in Figure 2a, \( G' \) for the both samples was higher than \( G'' \) over the whole frequency range. At all frequencies from 0.03 to 15 Hz, the intermolecular bonds did not have enough time to break during the period of oscillation, and for this reason, the network behaved as a gel, showing properties more typical of a solid than a liquid (\( G' > G'' \)). Other researchers also reported that existence of a strong internal strength in an emulsion can be revealed by the rapidly rising values of storage modulus in compared to the loss modulus values.\(^{[21,22]}\) PE showed a significant effect (\( p < 0.05 \)) on the \( G' \) and \( G'' \) magnitude (Figure 2a). This fact is possibly due to the formation of strong interactions among PE molecules. At first, no network has yet been constructed, and each PE molecule behaves very much like a single-dispersed molecule. As the PE dispersion is sheared, its molecules relax rapidly because of their high molecular mobility. Therefore, no extensive stress buildup, particularly at low oscillation frequencies, is observed. PE molecules by increasing frequency begin to associate by reason of formation of a three-dimensional network through hydrogen bonds and hydrophobic interactions.\(^{[23]}\)

Tan \( \delta \) or phase angle (\( G''/G' \)) is one of the indices representing viscoelasticity characteristics. Phase angle for an ideal viscous solution is infinite; while for an ideal elastic solution, it is zero. When tan \( \delta \) is greater than 1, the solution is more viscous than elastic, and the opposite conclusion is correct. Thus, the gelling point can be defined as the point of tan \( \delta = 1.\)\(^{[24]}\) Figure 2b showed that this factor was lower

![Figure 2](image-url)  
**FIG. 2.** Frequency sweep tests of spreads containing 0.5% PE ((a) ■, \( G' \); □, \( G'' \) (b) ○, tan \( \delta \)) and 0.1% XG ((a) ●, \( G' \); ○, \( G'' \) (b) ●, tan \( \delta \)).

![Figure 3](image-url)  
**FIG. 3.** Frequency development of the storage modulus of different formulations of low-fat spreads ((a) XG-LBG and PE-LBG; (b) XG-ALG and PE-ALG; and (c) XG-MS and PE-MS).
than 1, suggesting that solid-like elastic behavior is more dominant in low-fat spreads than liquid-like viscous behavior. Figure 3 shows frequency dependences of $G'$ of the spreads as a function of used polysaccharides. Based on the power-law parameters in Equation (1), an exponent $x$ near zero was found almost in all samples that represents a characteristic behavior of a fully cured gel.$^{[25]}$ However, an increase was considered in the $G'$ by increasing frequency (Figure 3). In contrast, the $G'$ of low-fat spreads with various compositions decreased by increasing frequency (Figure 4). This fact was confirmed with higher values of factor $a$ than $b$ (Equations (1) and (2)) in all samples showing that $G'$ predominates in the major part of the spectra, and solid-like behavior was observed. Use of MS in the combination with both basic polysaccharides (XG and PE) led to an increase the $G'$ of spreads in comparison with ALG and LBG. As previously described by Valdez et al.$^{[26]}$ enhancement in elastic modulus of the spreads containing MS as a result of frequency development could be associated to the closer packing of micro-gelled colloidal particles or water-swollen starch granules leading to greater friction forces between the droplets subjected to shear. High $G'$ of the samples containing ALG (especially

![FIG. 4](image1.png)  
**FIG. 4.** Frequency development of the loss modulus of different formulations of low-fat spreads ((a) XG-LBG and PE-LBG; (b) XG-ALG and PE-ALG; and (c) XG-MS and PE-MS).

![FIG. 5](image2.png)  
**FIG. 5.** Frequency development of the delta degree $\delta$ of different formulations of low-fat spreads ((a) XG-LBG and PE-LBG; (b) XG-ALG and PE-ALG; and (c) XG-MS and PE-MS).
concentration of 0.4%) and XG (Figure 3b) could be due to the formation of intermolecular bonding like hydrogen bonding through an interaction of hydroxyl- and carboxyl-groups of such polymers, resulting in an increasing of viscosity of the composite dispersion.[27] Pongjanyakul and Puttipipatkhachorn[28] also suggested that these predicted molecular interactions led to viscosity synergism of XG with ALG. The phase angle of the different spreads decreased by increasing frequency from 0.03 to 15 Hz and accompanied the domination of the elastic over the viscous behavior (Figure 5). However, the spreads formulated with XG and LBG had lower elasticity than the other samples at low frequencies. This behavior can be due to the increase in synergistic system viscosity by the interaction between both polymers. However, the viscosity amounts could be decreased by promoting time due to the XG conformational changes and decrease of the bond strength.[19,20]

4. CONCLUSIONS
In the current investigation, the dynamic rheological characteristics of low-fat spreads formulated with different polysaccharides such as XG, PE, LBG, MS, and ALG were investigated. Also, microstructural and stability of these systems were partially studied to interoperate their rheological properties. The characterization of rheological characteristics of the samples showed that power-law model was a better model than Herschel–Bulkley model by fitting the experimental data and calculating the consistency and power-law index. The viscosities of all samples were thus decreased by increasing shear rate because of the deformation and disruption of clusters or aggregates of droplets and their ordering within the flow field. However, the viscosity amounts were increased by increasing concentration of ALG, MS, and LBG from 0.2% to 0.4%. Results also showed that the storage modules of the all spreads decreased with frequency, while the loss modules increased. Delta degree decreased with increasing frequency from 0.03 to 15 Hz revealed that solid-like elastic behavior dominates over liquid-like viscous behavior. In general, the lowest OO level was for low-fat spreads prepared with 0.5% PE and 0.4% LBG. The formation of the suitable viscous systems at higher LBG contents may have benefits to improve the texture and mouthfeel of reduced fat products like spreads. Therefore, a desirable influence on the flavor profile of these products can obtain by the trapping fat droplets LBG microparticles at high concentrations of this gum. Future studies will concentrate on the specific chemical and structural modifications, which could present insights into new functional characteristics of food hydrocolloids.

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