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Mango gels: Characterization by small-deformation stress relaxation method

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ABSTRACT

Mango based alginate gels were formed by varying the concentrations of mango juice (50–92%), sodium alginate (3–5%), glucono-δ-lactone (1.5–2.5%) and calcium orthophosphate (1.5–2.5%) employing a second order central composite rotatable design. These gels were characterized by applying the method of small-deformation stress relaxation to obtain parameters such as initial stress, residual stress, extent of relaxation and relaxation time (λ). The initial and residual stresses showed high values when mango juice concentration was low (50.0–60.5%). The mango juice had a strong negative linear effect (significant at $p \leqslant 0.01$) followed by the positive effects of sodium alginate and glucono-δ-lactone (GDL) ($p \leqslant 0.01$). A method for the textural categorization of gels based on relaxation time (λ) was proposed. A λ value less than 1 s offered a highly soft gel while $\geqslant$100 s gave a good set hard gel; medium soft textured gels resulted for λ values between 1 and 10 s while good set textured gels were associated with 10 s < λ < 100 s. The application of principal component analysis (PCA) indicated that the concentrations of mango juice and alginate offered the dominating effects on the stress relaxation related indices.

1. Introduction

Food gels are complex in nature, and are rheologically characterized by employing small- and large- deformation test procedures. Under small applied strains within the linear viscoelastic region, the structure of gel remains unaltered (Zhang et al., 2005). The fundamental tests thus measure well defined parameters such as relaxation time and elastic modulus. It is, thus, desirable to employ fundamental test methods when the aim is to use rheological experiments to elucidate gel characteristics and structure. On the other hand, large-deformation tests partially simulate biting and chewing actions inside the mouth, and are of practical use for product development and consumer acceptance.

The test based on the measurement of small-deformation stress relaxation property has been frequently used to determine the viscoelastic behavior of foods mainly due to the ease of conducting the test. However, satisfactory interpretation of results is not completely resolved as food samples are hardly homogenous and isotropic, and linking the results with consumer preference is difficult. In addition, the linear viscoelastic range in food samples is generally very small. Regardless of all these limitations, researchers have tried to resolve the problems through fitting mathematical models to stress relaxation data and their interpretations. Low-strain stress relaxation studies have been conducted by Bhattacharya (2010); a model containing four parameters is reported to be suitable for stress relaxation characteristics. Tiwari and Bhattacharya (2011) have reported that the rheological changes in aerated agar and gellan gels at different solid concentrations can be better assessed by stress relaxation studies.

Several applications are possible for alginates and its derivatives in food and pharmaceutical industries. They have been used for the encapsulation of wide verities of bioactive materials, proteins, enzymes, antibodies, etc. (Coppi et al., 2002). This is due to its mild cross-linking condition required for the formation of gels. In addition, fruit juice/pulp based alginate gels are also possible to develop (Kaletunc et al., 1990) if the gelling ingredients are carefully chosen along with appropriate adjustment of the gel forming conditions. The fruit content, interaction with final soluble solids and added hydrocolloid on gel strength of strawberry and peach gels have been studied (Carbonell et al., 1991). The effect of gel forming variables on alginate gels includes the concentrations of gelling agent and calcium source, concentrations of sequestering agent, gel setting time, temperature, pH and most importantly the method of gel setting that effect of textural properties of the formed gels (Roopa and Bhattacharya, 2010). The characterization and modeling of time-independent and time-dependent flow behavior of sodium alginate dispersions have been reported (Roopa

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and Bhattacharya, 2009). Jaya and Durance (2008) have studied the stress relaxation behavior of dried alginate gels; these researchers have observed that the mechanism involved in stress relaxation is entanglement coupling of larger polymer chains in covalently cross-linked alginate gels. However, an understanding on the relaxation characteristics and sensory attributes is needed for developing fruit juice based gelled products. Scope thus exists to categorize the gels on the basis of textural attributes which affects the consumer acceptance of the product.

The objectives of the present study are (a) to determine the low-deformation stress relaxation behavior of mango gels by varying the concentrations of gel forming variables such as mango juice, sodium alginate, glucono-δ-lactone (GDL) and calcium orthophosphate and (b) classification of gels based on stress relaxation characteristics.

2. Materials and methods

2.1. Materials

Sodium alginate ($C_6H_7O_6Na$) and calcium orthophosphate were obtained from Loba Chemie, Mumbai, India. The sodium alginate used for the present study was of food grade type, and was composed of anhydro-β-D-mannuronic acid residues with 1,4 linkages. Glucono-δ-lactone (GDL) was obtained from Sigma Chemicals, St. Louis, USA. Mango juice (Tropicana, Pepsi Foods, Haryana, India) having 15 °Brix, pH of 4.5 and moisture content of 83.8 ± 1.2% was used to make mango gels. Petri dishes of 35 mm in diameter and 9 mm in height were used to set gels.

2.2. Methods

An experimental design (Table 1) consisting of 4 variables (5 levels each), second order central composite rotatable design (Myers, 1971) with 6 replications at the center points in coded levels of variables was employed. The independent variables were X_1 (concentration of mango juice), X_2 (concentration of sodium alginate), X_3 (concentration of glucono-δ-lactone) and X_4 (concentration of calcium orthophosphate). The selection of variables for gel formation and their ranges were based on preliminary laboratory trials on gel formation and setting (Mancini et al., 1999). The popular product mango juice has been selected to develop fruit juice based gels. Earlier study (Roopa and Bhattacharya, 2010) shows the effect of gel forming variables using pineapple juices. The range of concentration of sodium alginate was between 3 and 5 g/100 g considering the recommendation of Generally Recommended As Safe (GRAS) level in fruit and juices as a jellifying agent (Burdock, 1995). Calcium hydrogen orthophosphate and glucono-δ-lactone (GDL) were between 1.5 and 2.5 g/100 g because the mass of glucono-δ-lactone (GDL) is equal to that of the calcium source used (Mancini et al., 1999). Gels were prepared according to the experimental design (in coded and actual levels) as shown in Table 1. Mango gels were prepared by dissolving the required quantity of sodium alginate and calcium orthophosphate in mango juice for 30 min with continuous agitation of the sol (Roopa and Bhattacharya, 2010). Latter, glucono-δ-lactone (GDL) was added to the sol and mixed again. The mixed sol was immediately poured into petri dishes to set the gels.

The lubricated gel samples (about 35 mm in diameter and 9 mm in height) were compressed by using a flat stainless steel circular smooth plate having a diameter of 100 mm. A texture measuring instrument (Model # TAHD, Stable Microsystems, Surrey, UK) was employed while using appropriate load cells. The gels were compressed up to an applied engineering strain of 5% (within linear limit of viscoealstic range) using a cross head speed of 1 mm s^{-1}, and later allowed to relax for 600 s after the required extent of compression was achieved. Data were collected at the rate of 10 points per second. Parameters such as initial stress, residual stress, extent of relaxation and relaxation time were determined. The initial stress (IS) (σ_0) indicates the stress offered by the gel sample at the end of applied strain; it is the force per unit area of gel prior to initiating the stress relaxation test. The residual stress (RS) (σ_e) was obtained at the end of the relaxation test in a similar manner. The extent of relaxation (EOR) was calculated as $100^*(\sigma_0 - \sigma_e)/\sigma_0)$. Relaxation time (RT) ($\lambda$) was calculated from the plot of relaxation time (t) against $\sigma_0 t/[\sigma_0 - \sigma(t)]$ corresponding to the e^{-1} of initial stress (Yadav et al., 2006). Five samples were tested each time.

The overall non-oral sensory textural assessment of mango gels was performed by 10 trained panelists by pressing the gel samples with the index finger. The samples were categorized into highly soft gel, medium soft gel, good set textured gel and good set but hard gel based on the sensory observations (Table 4). These observations were latter correlated with the stress relaxation results. The reported results were obtained from three replications.

The methodology of principal component analysis (PCA) was employed (Jena and Bhattacharya, 2003) to inter-relate the four independent variables and four response functions. The analysis was performed by using the statistical software Statistica'99 (StatSoft, Tulsa, OH, USA) by loading to different axes and scores of the results were superimposed in a two-dimensional biplot (Lawless and Heymann, 1998) to obtain the PCA plots for interpretation of results.

Table 1
Design of experiments employed to set mango gels.

Expt. no	Variables (coded level)				Variables (actual level)			
	x_1	x_2	x_3	x_4	X_1	X_2	X_3	X_4
1	0	2	0	0	71.0	5.0	2.00	2.00
2	0	0	0	0	71.0	4.0	2.00	2.00
3	0	−2	0	0	71.0	3.0	2.00	2.00
4	0	0	0	0	71.0	4.0	2.00	2.00
5	0	0	0	−2	71.0	4.0	2.00	1.50
6	−1	−1	−1	1	60.5	3.5	1.75	2.25
7	0	0	0	0	71.0	4.0	2.00	2.00
8	−1	−1	1	−1	60.5	3.5	2.25	1.75
9	−1	1	1	−1	60.5	4.5	2.25	1.75
10	0	0	0	0	71.0	4.0	2.00	2.00
11	0	0	0	2	71.0	4.0	2.00	2.50
12	−1	−1	1	1	60.5	3.5	2.25	2.25
13	1	−1	1	1	81.5	3.5	2.25	2.25
14	1	1	−1	1	81.5	4.5	1.75	2.25
15	1	−1	−1	−1	81.5	3.5	1.75	1.75
16	−1	1	1	1	60.5	4.5	2.25	2.25
17	0	0	0	0	71.0	4.0	2.00	2.00
18	−1	1	−1	1	60.5	4.5	1.75	2.25
19	1	−1	1	−1	81.5	3.5	2.25	1.75
20	1	1	−1	−1	81.5	4.5	1.75	1.75
21	1	−1	−1	1	81.5	3.5	1.75	2.25
22	1	1	1	1	81.5	4.5	2.25	2.25
23	−1	−1	−1	−1	60.5	3.5	1.75	1.75
24	1	1	1	−1	81.5	4.5	2.25	1.75
25	0	0	2	0	71.0	4.0	2.50	2.00
26	0	0	0	0	71.0	4.0	2.00	2.00
27	−1	1	−1	−1	60.5	4.5	1.75	1.75
28	0	0	0	0	71.0	4.0	2.00	2.00
29	2	0	0	0	92.0	4.0	2.00	2.00
30	−2	0	0	0	50.0	4.0	2.00	2.00
31	0	0	−2	0	71.0	4.0	1.50	2.00

Independent variables:
X_1: Concentration of mango juice, X_2: Concentration of sodium alginate, X_3: Concentration of glucono-δ-lactone and X_4: Concentration of calcium orthophosphate.