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ORIGINAL ARTICLE

Quality of Jelly Candy Made of Acid-Thinned Starch Added with Different Non-Starch Polysaccharides

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Abstract

The quality of jelly candy made with acid-thinned starch added with different non-starch polysaccharides (pectin, carboxymethyl cellulose (CMC) and sodium alginate) at various addition levels were characterized. Pasting and flow properties of the jelly candy slurry were modified to different extents. Generally, pasting temperature, peak viscosity, and trough viscosity were increased and setback values were decreased with addition of non-starch polysaccharides. Frequency sweep test showed that G' values decreased in the order of Pectin > CMC > Alginate > control. Continuous flow analysis showed that a hysteresis loop was formed between upper and lower ramp curves. The largest hysteresis loop was demonstrated when pectin was added. This characteristic exhibited significant practical bearing, whereby when pectin was added to acid-thinned starch slurry, no tailing problem was seen during deposition, which normally forms due to a fast setting. Apart from this, pectin added jelly candy showed a smoother surface finish when compared to other samples. The non-starch polysaccharides studied were found to impart different textural characteristics such as softness, elasticity, and moistness to starch-based jelly candy.

Keywords*:* Acid-thinned starch, Pectin, Carboxymethyl cellulose, Sodium alginate.

1. Introduction

Acid-thinned starch-based jelly candy is receiving increasing attention of food technologists over gelatine-based candy due to the emerging and lucrative halal, kosher and vegetarian market. Starch-based jelly candy like many other high-solid food products, are regarded as being craft based. Therefore, literature reporting on all parts starting from the characteristics of starch during and after cooking in the presence of other ingredients, through rheological responses during pumping and deposition, and product quality during storage is relatively sparse. All these are of great importance for the confectionery industry since the lack of understanding of these properties will post obstacles to the industry when selecting the right ingredient and processing conditions to confer good product quality.

Non-starch polysaccharides (NSP) are commonly incorporated in starch-based gel systems to provide a wide range of properties such as texture, storage stability, processability, and control release (Yoshimura *et al.,* 1998; Shi and BeMiller, 2002; Lubbers and Guichard, 2003; Al-Marhoobi and Kasapis, 2005; Savary *et al.,* 2007). A voluminous literature exists on studying the effects of NSP on starch-based product properties. Among the NSPs tested are guar gum, pectin, alginate, kappa-carrageenan, xanthan, cellulose derivatives, just to name a few. In the work of Lee *et al.* (2002), sodium alginate, gum arabic, carboxymethyl cellulose, guar gum, locust bean gum and xanthan were reported to be able to reduce syneresis of sweet potato starch systems, while the opposite effect was induced by curdlan, gellan and kappa-carrageenan. However, in the same study, differential scanning calorimetry result showed that all NSPs retarded recrystallization of sweet potato starch after a series of freeze-thaw cycles. The reason for such contradictory effects might lie in the fact that the effects of these NSPs on starch systems may manifest in different ways, depending on the NSP structure, rheological properties, topological effective concentration, purity and the structural compatibility and the degree of molecular interactions between starch components and NSP molecules (Symons and Brennan, 2004; Funami *et al.,* 2005). Therefore, results across

literature may not be compared effectively due to the diversity of variables used.

In the present study, we focused on characterizing the effects of anionic NSP including high-methoxyl pectin (PEC), carboxymethyl cellulose (CMC) and alginate (ALG) on acid-thinned starch jelly candy slurry and final products. To the best of our knowledge, effects of anionic gums on acid-thinned starch have not been studied in detail, especially in a high-solid system mimicking the industrial preparation. The use of NSP in high-solid systems is significant, since a high solid content imparts high viscosity and make the food system difficult to handle during processing. With the addition of NSP, the characteristics and workability of a high-solid food can be enhanced. From the work of Al-Marhoobi and Kasapis (2005), it was suggested that the tendency for biopolymer to form progressively longer and, hence more thermally stable helices at high levels of co-solute should be increasingly counterbalanced by the inability to sustain those helices in the form of stable aggregates due to the scarcity of water molecules.

The current work was planned to study the effects of selected anionic NSP on acid-thinned starch pasting, retrogradation and rheological behaviour, and the influence of the selected anionic NSP on the textural properties and storage stability of starch-based jelly candy made therefrom.

2. Materials and Methods

2.1 Materials

Acid-thinned sago starch (Elastigel 1000 J) was obtained from National Starch and Chemical (M) Sdn. Bhd. (Selangor Darul Ehsan, Malaysia). Anionic NSPs including Carboxymethylcellulose Sodium Salt (Fluka bio Chemika, Switzerland), alginate acid salt from Brown algae (Fluka bio Chemika, U.K) and high-methoxyl pectin (Sim Company, Malaysia) were used in this study. Other ingredients such as sugar, glucose syrup (42 DE), tri-sodium citrate, corn starch, dextrose, citric acid were purchased as commercial products from SIM Company Sdn. Bhd. (Penang, Malaysia).

2.2 Pasting Properties

The pasting properties of acid-thinned starch added with or without added NSP (0.15, 0.3 and 0.6 g/kg, starch basis) were determined using a Rapid Visco Analyser (RVA, Model RVA Series 4, Newport Scientific Pvt. Ltd., Warriewood, Australia). Starch (5.0 g dry basis (db)) with or without added NSP, was weighed directly into RVA canister and distilled water was added to obtain a total sample weight of 25.0 g. The amount of sample added was corrected to 14% moisture content. The sample was held at 50 ^{o}C for 1 min, heated to 95 ^{o}C in 3.5 min, and then held at 95 ^{o}C for 2.5 min, before cooling to 50 ^{o}C in 3 min, and holding at 50 ^{o}C for 2 min. The agitation speed of the paddle was fixed at 960 rpm for the first 10 s to ensure uniformity of the dispersion, and was lowered to 160 rpm throughout the rest of the measurement. Parameters recorded including pasting temperature, peak viscosity, trough viscosity, breakdown, and setback. All tests were replicated three times.

2.3 Rheological Characteristics

Rheological evaluation was performed using a Rheometer (Rheometer, model AR 1000, TA Instruments Inc., New Castle, DE, United States). Starch (10.0 g db) with or without added NSP (0.3 g/kg, starch basis) was suspended in 100 ml distilled water in a 250 mL media bottle. Sample was gelatinized in water bath at 95 ^{o}C for 10 minutes. The samples were kept warm at 50 ^{o}C until used.

A frequency sweep ranging from 0.1 to 100 rad s^{-1} was performed at 50 ^{o}C and at 1% strain, which was pre-determined from the linear viscoelastic region of the sample. The geometry used was a 20 mm standard parallel plate and the gap size was fixed at 1000 μm. To avoid evaporation, paraffin oil was wiped over the sample edge. TA Rheologist Data Analysis software (Version 5.4.8) was used to obtain the experimental data and to determine storage modulus (G') and loss modulus (G").

Stepped flow curves were obtained by recording shear stress values when samples were subjected to a programmed shear rate increased from 0 to 1000 s^{-1} and decreased from 1000 to 0 s^{-1}, respectively. All measurements were carried out at 50 ^{o}C using a parallel plate geometry (40 mm diameter and 1 mm gap).

2.4 Making of Jelly Candy

Recipes for making starch-based jelly candy were adapted from those recommended by National Starch and Chemical Co. as shown in Table 1. Approximately 100 g of starch was cooked in 550 g of water until gelatinzation in a steam-jacketed kettle. Tri-sodium citrate solution was added during cooking. After two minutes of boiling, selected NSP was dry-blended with a portion of sugar and added to the candy slurry before adding in glucose syrup, dextrose and the remaining sugar. For a control, no NSP was added. The candy slurry was cooked with continuous stirring until a soluble solid content of 65 ºBrix was reached. The heat was then turned off before citric acid solution, coloring and flavoring agents were added. The batch was then cast into a corn starch mould which had been

Table 1 Formulation for starch-based jelly candies prepared with or without addition of anionic gums

Ingredients	Control (g)	Samples added with gums (g)
Glucose syrup (42 DE) (g)	360.0	360.0
Sugar (g)	300.0	300.0
Dextrose (g)	110.0	110.0
Acid-thinned starch (g)	100.0	100.0
Gums (g)	-	5.0
Water (for starch) (g)	550.0	550.0
Warm water (for citric acid) (ml)	20.0	20.0
Tri-sodium citrate (g)	4.0	4.0
Citric acid (g)	4.0	4.0
Flavouring agent (g)	1.0	1.0
Colouring agent (g)	0.5	0.5

pre-dried in a hot air oven at 65 ºC for 12 hours. Subsequently, the filled corn starch mould was tempered at 65 ºC for 12 hours. Finally, the jelly candies were removed from the starch mould.

Sample preparation was repeated twice for each formulation. Samples were stored in sealed polypropylene plastic bags at 25°C (0 day, 30 days and 180 days) until analysis.

2.5 Texture Profile Analysis (TPA)

Samples were subjected to Texture Profile Analysis (TPA) using a TA-XT Plus Texture Analyzer (Stable Micro Systems, Surrey, England) with a 30 kg load cell and a 25 mm diameter cylindrical probe. Measurements were conducted at a pre-test speed of 1.0 mm/s, a post-test speed of 10.0 mm/s, a test speed of 2.0 mm/s, and 20 g trigger force. Deformation level was fixed at 75 %.

Five repeated measurements were performed for each batch of sample and their average was taken. All the parameters were analyzed with Texture Exponent 32 software (Version 4.0.5.0). From the TPA curve, texture characteristics such as hardness, springiness, cohesiveness, chewiness, gumminess, adhesiveness, and resilience were determined.

2.6 Statistical Analysis

The statistical analysis of results obtained was conducted by the analysis of variance using statistical software SPSS 14.0 for windows (SPSS, Inc., Chicago, IL, USA). Duncan's test was used to determine significant difference (P<0.05) between samples mean values.

3. Results and Discussion

3.1 Pasting Properties

The pasting characteristics of acid-thinned starch suspension with or without addition of pectin (PEC), carboxymethyl cellulose (CMC) or alginate (ALG) are displayed in Fig 1. Each selected NSP was found to influence the pasting parameters of acid-thinned starch in the similar ways, but to different extent. In general, pasting temperature was increased significantly with progressive increase in NSP concentration, the most effective being given by alginate, followed by CMC and pectin. This observation is in line with earlier reports, which showing a decrease in enthalpy and shift in the gelatinisation peak to a higher temperature when NSPs were added to a starch system and this was attributed to incomplete starch gelatinisation as a result of limited water availability (Eliasson, 1980; Marchant and Blanshard, 1980; Khanna and Tester, 2006). According to Ferrero *et al*. (1996), when NSPs were added in a sufficiently high amount, it will compete with starch molecules for water during a gelatinisation process and result in a higher pasting temperature. On the other hand, our results contrasted to other reports which recorded a lower pasting temperature upon NSP addition. The plausible explanation given was varied, some attributed it to some kind of interaction between starch and the hydroxyl groups of the NSP (Christianson *et al.*, 1981), or an early detection of measurable viscosity increase due to media viscosity imparted by NSP, rather than granule swelling (Christianson *et al*., 1981).

As for peak viscosity, when NSP addition level was increased from 0 to 6%, peak viscosity recorded for alginate, CMC or pectin added samples was increased from 60 to 180 RVU, 60 to 120 RVU and 60 to 70 RVU, respectively. Peak viscosity reflects the maximum swelling capacity of the starch granules before being physically ruptured. Therefore, the result may suggest that the NSP added has promoted or enhanced the swelling capacity of the starch granules. Nevertheless, this could also suggest an increase viscous load during cooking of acid-thinned starch -

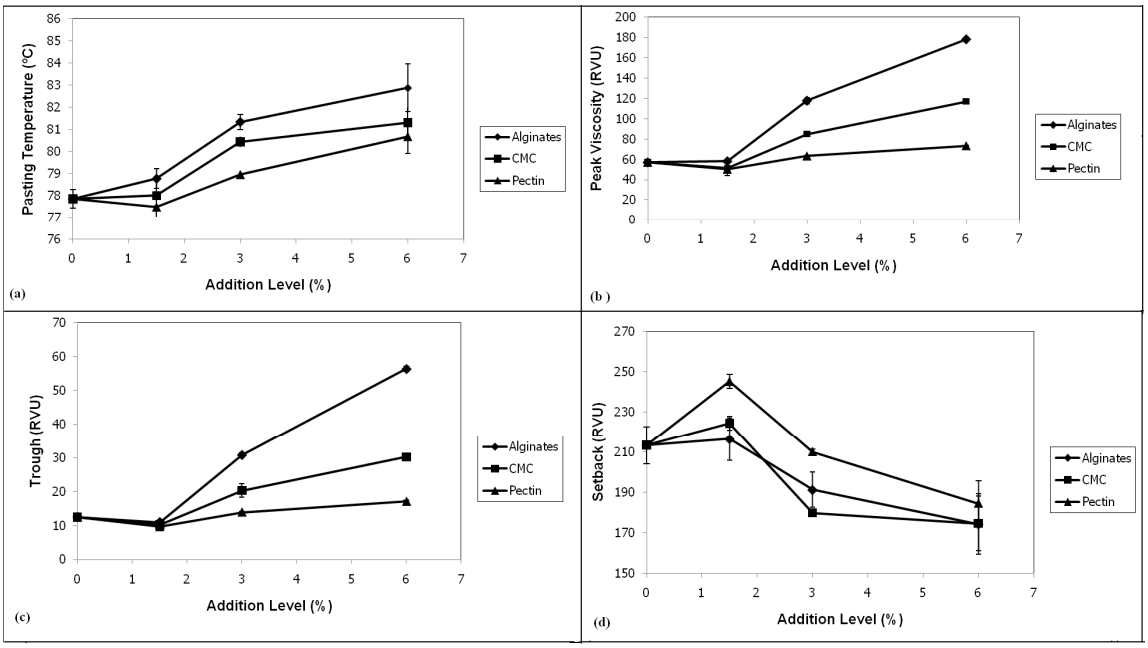

Fig 1: Pasting properties of acid-thinned starch suspension with or without addition of pectin, CMC or alginate.

slurry, especially when alginate and CMC were added. Similar trend has been reported in other research works dealing with the addition of alginate, xanthan, *iota- or kappa*-carrageenan, and galactomannan (Christianson *et al.*, 1981; Evans and Haisman, 1982; Alloncle *et al.*, 1983). This increase in peak viscosity upon addition of NSP was attributed to either the thickening effect caused by the NSP added, the interactions happened between NSP and swollen starch granules or leached molecules, or the cushioning effect imparted by NSP molecules present in the continuous phase of a starch suspension (Yoshimura *et al.*, 1998; Tester and Morrison, 1990). Similar trend as observed in peak viscosity was observed in trough value, whereby trough viscosity increased with increase in NSP addition level, with effectiveness decreasing in the order of alginate>CMC>pectin. This can be ascribed to the above-mentioned.

Interestingly, setback which was calculated as the difference between final viscosity and trough viscosity was found to be significantly lower in samples with 3% and 6% of added NSP when compared with control sample. This denotes that intermolecular associations of starch molecules had been inhibited. This delaying of retrogradation of amylose molecules could be attributable to a greater electrostatic repulsion caused by the more extended and highly negatively charged NSP molecules that subsequently decreased association between amylose molecules. Inevitably, the possibility of having insufficient or limited amylose leaching during the pasting in the presence of a high level of NSP could also account for this lower setback value observation. In opposition, pectin was found to be thermodynamically more compatible with starch components, whereby its presence at level of 1.5% increased the setback value significantly ($P<0.05$). These results are in satisfactory agreement with the previous publication where it was stated and experimentally observed that the suppressing effect of hydrocolloids depend on whether the hydrocolloid used is uncharged or charged in relation to phase separation between the two components (Annable *et al.*, 1994), as well as the hydrodynamic volume occupied by the hydrocolloid within the continuous phase (Zhou *et al.*, 2008).

3.2 Rheological Behaviour

Frequency sweep: The frequency sweeps response of acid-thinned starch slurry prepared with or without anionic gums are shown in Fig 2. G' is a measure of material elasticity and signifies its ability to store energy. According to Edwards *et al.* (1995), small strain dynamic tests could be used to study the network structure and it is more sensitive to subtle chances than large deformation compression tests. G' is proportional to the extent of the elastic component contributed by crosslinking, entanglement, and/or aggregation within a

system. As evident, when anionic gums were added to acid-thinned starch paste, an increase in storage modulus (G') was observed and G' was reduced in the order of 6% Pectin ≥ 3% Pectin > 1.5% Pectin ≥ 6% CMC ≥ 6% alginate > 3% alginate ≥ 1.5% alginate > 3% CMC > 1.5% CMC ≥ control. This pattern is indicative that, addition of anionic gums and its progressive increase in concentration favour polymer-polymer association in the system, and greater effect was shown by pectin followed by alginate and CMC.

Stepped flow analysis: Flow analysis revealed that all samples studied demonstrated a non-Newtonian shear thinning rheological behaviour. The consecutive forward and backward measurements during a shearing cycle, shows hysteresis loop (typical flow curve not shown). This thixotropy hysteresis loop is shown when a system is sheared under a constant rate, and the apparent viscosity decreases with time until a balance between structural breakdown and re-formation is reached (Shaw, 1999). Thus, thixotropy is commonly attributed to the breakdown/alignment of polymer chains or segments (Al-Malah *et al.*, 2000). The greater the loop area, the more "structured" a system is, or vice versa.

The shear stress and shear rate data were fitted to different models and best fitted curve was judged by standard error with the AR 1000 Rheometer. The best fitted model proposed was Herschel-Bulkley (H-B) model as follows:

$$\tau = \tau_o + k\dot{\gamma}^n$$

where τ, τ_o, k $\dot{\gamma}$ and n represent shear stress, yield stress, consistency index, shear rate and flow index., respectively (Barbosa-Canovas and Peleg, 1983; Paredes *et al.*, 1989; Gong *et al.*, 2010). The H-B rheological parameters of the ramp-up and ramp-down curves are shown in Table 2. In general, the shear stress – shear rate relation for the ramp-down curve was recorded at lower region as compared to the ramp-up curve. This suggests that these materials are showing thixotropic properties, with shear stress decreases with time. Interestingly, pectin added sample shows significantly higher thixotropy value as compared to the other samples (Fig 3), indicating that pectin can reinforce the network structure of the starch pastes. This can be interpreted as an increase in association between starch molecules and pectin molecules during high deformation shearing. As a result, when pectin added starch paste was deformed, a relatively longer time was required for it to return to its original consistency upon removal of shear stress.

As expected the static yield stress values were higher than the dynamic yield stress When the NSP concentration was increased from 1.5% to 6%, the static yield stress was found to range from 11.47 to 44.95, 23.35 to 26.37 and 11.70 to 17.42 Pa, for alginate, pectin and CMC added samples, respectively. The sample would not flow when being sheared at a shear stress lower than the yield stress and it is a useful parameter for judging the ease of handling of the sample (Wei *et al.*, 2001; Sun and Gunasekaran, 2009). The consistency index and the flow index does not have physical basis because they are purely a mathematical description. However, it is useful for identifying flow and engineering design (Duran and Costell, 1982). All samples show a flow index less than 1, indicative of a non-Newtonian and shear thinning flow behaviour. Consistency index was not compared among samples because these values are in different units.

3. 3 Textural Properties of Jelly Candy Prepared

Fig 4 shows the picture of some typical jelly candy samples prepared with acid-thinned starch pre-blended with or without 3% pectin, CMC or alginate. The starch mould used was printed with heart-shape pattern. From the samples obtained, it is obviously seen that the control sample is irregularly shaped with signs of tailing, and the shape was enhanced with addition of anionic gums. Among those gums added samples, pectin added ones was found to be the nicest and most regularly shaped. In addition, the surface of the pectin added jelly candy is much smoother when compared to the others. This can be examined by checking on the amount of starch thread marks shown on each samples surface.

The above observation substantiates the findings reported in the previous stepped flow analysis, whereby sample showing larger hysteresis loop is believed to show high time dependency, meaning to say it is not easy to recover to its original consistency upon deformation. Thus, it may be inferred that pectin added candy slurry may have virtually sufficiently long time to flow and take the shape of the starch mould. This inevitably will reduce the problem of having tailing which was formed owing to fast setting. This is why the appearance of pectin added candy is more appealing than the others. This observation has an important practical significance because it denotes that the deposited slurry should show optimal setting characteristics in order to assure good quality of the end-product.

Jelly candy generally possesses a long shelf life due to its low water activity, hence the effects of different anionic gums on the textural properties stability was studied over a period of 180 days storage at 25 ^oC.

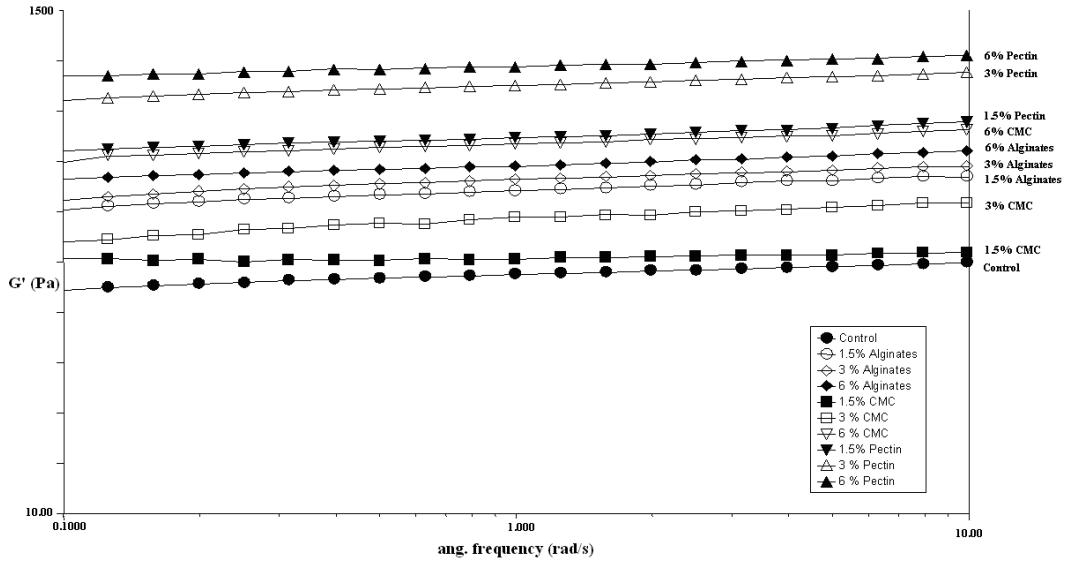

Fig 2: Variation of storage modulus (G') as a function of frequency for acid-thinned starch paste with or without addition of pectin, CMC, or alginate.

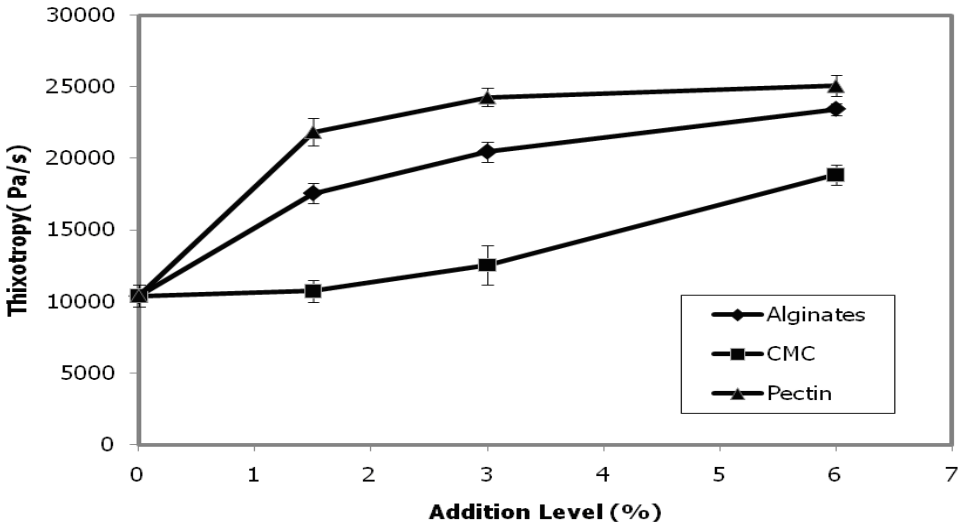

Fig 3: Variation of thixotropic properties for acid-thinned starch paste with or without addition of pectin, CMC, or alginates.

As shown in Fig 5, the hardness of the jelly candy (control) was not significantly changed 180 days of storage (Fig 5a). Similar result trend was observed in CMC added samples. As for alginate and pectin added samples, hardness was found to decrease with storage time. Decrease in hardness in sugar-starch-based foods upon storage is not unusual. This can be explained by considering water molecules being excluded from the amorphous phase when sugar molecules re-crystallize, and the so-called "free" water molecules then plasticize the starch network further and render it softer in texture.

According to Szcesniak (2002), springiness is defined as the rate at which a deformed material return to its original shape after the deforming force is removed. It was observed that all samples showed increased springiness upon addition of gums and storage (Fig 5b). A reverse trend was evident in gumminess which refers to the energy required to -

Table 2: Flow parameters of the samples determined by Herschel-Bulkley equation

Sample	Upward curve			Downward curve			Thixotropy (Pa/s)
	Static Yield stress (Pa)	Consistency index (Pa.s^n)	Flow index	Dynamic Yield stress (Pa)	Consistency index (Pa.s^n)	Flow index	
Control	9.56±2.39a	6.50±1.69a	0.44±0.04d	10.47±1.69a	2.46±0.24b	0.57±0.01d	10381 ± 755a
1.5% Alginates	11.47±5.51a	28.55±3.34d	0.33±0.02b	27.05±4.63d	12.46±2.08f	0.44±0.02a	17560 ± 680c
3% Alginates	21.40±1.48c	12.57±0.62b	0.43±0.01d	22.37±2.90bc	6.95±0.83d	0.51±0.02b	17935 ± 690c
6% Alginates	44.95±9.22d	13.91±4.01b	0.45±0.04d	24.04±0.66cd	12.72±0.28f	0.46±0.00a	17150 ± 137c
1.5% Pectin	23.35±5.31c	37.67±1.08e	0.26±0.00a	18.98±1.54b	4.70±0.48c	0.54±0.01c	21837 ± 953d
3% Pectin	24.85±6.50c	14.11±0.21b	0.43±0.00d	20.59±5.63b	8.38±1.98de	0.50±0.03b	24137 ± 635e
6% Pectin	26.37±0.34c	18.96±1.73c	0.40±0.01cd	24.52±1.19cd	6.36±0.36d	0.54±0.01c	25065 ± 516e
1.5% CMC	11.70±3.67a	14.30±4.03b	0.40±0.02cd	13.49±0.68a	5.14±0.07cd	0.53±0.02c	10730 ± 755a
3% CMC	13.32±2.78ab	14.11±0.24b	0.49±0.02e	18.35±0.66b	0.42±0.03a	0.65±0.01e	12540 ± 1394b
6% CMC	17.42±0.34b	14.92±1.93b	0.39±0.02c	19.13±0.66b	4.74±0.20c	0.54±0.01c	18856 ± 929a

Note: Mean ± standard deviation (n=3). Means within the same column with the same superscript letter are not significantly different (P>0.05) according to Duncan's multiple range test.

* $\tau = \tau_o + k \dot{\gamma}^n$; τ, shear stress; τ_o, yield stress; k, consistency index; $\dot{\gamma}$, shear rate and n, flow index.

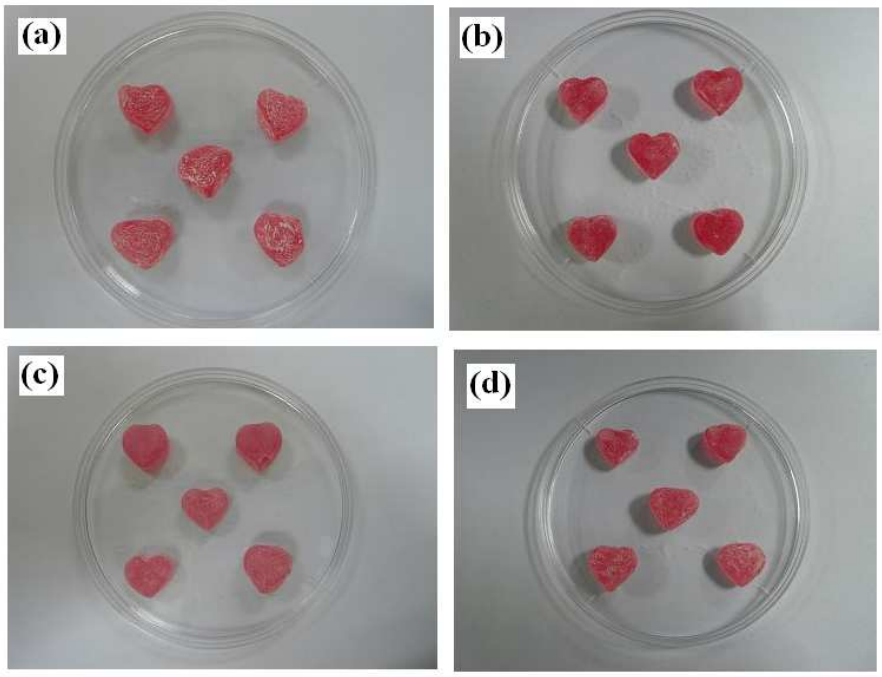

Fig 4: Samples of jelly candy made of acid-thinned starch; (a) Control (b) pectin added (c) CMC added and (d) alginate added.

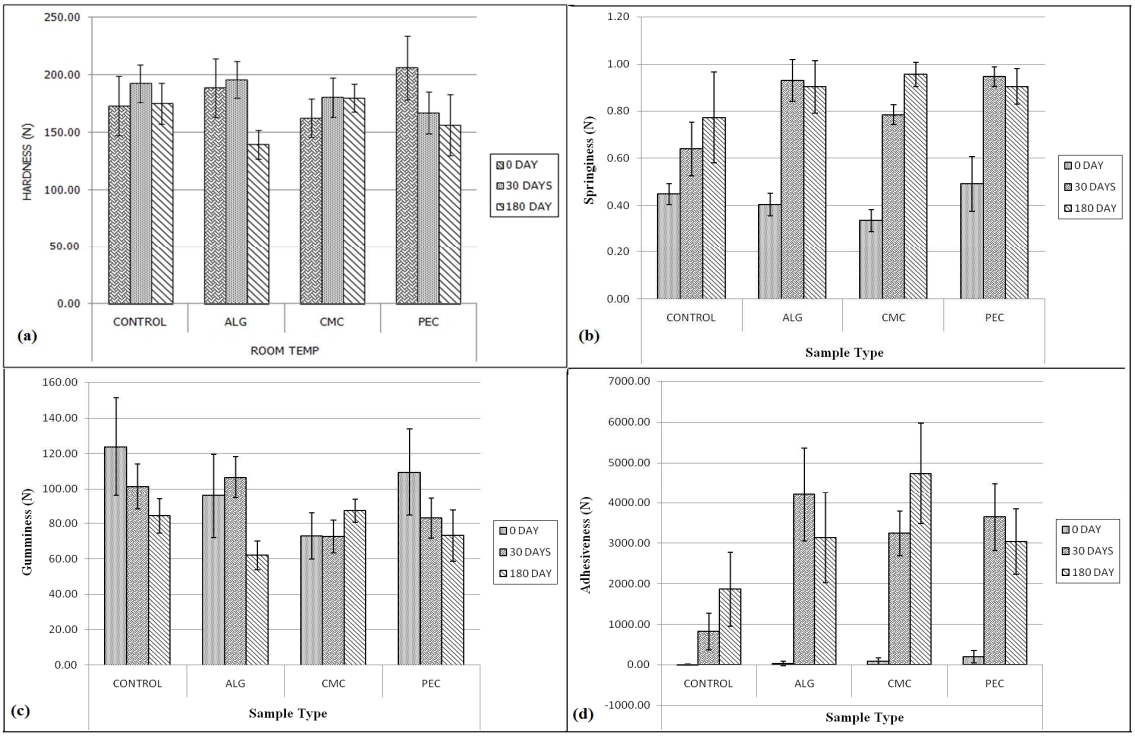

Fig 5: Texture stability of acid-thinned starch jelly candy prepared with or without addition of pectin, CMC or alginate.

disintegrate a semi-solid food to a state ready for swallowing (Szcesniak, 2002). On the other hand, samples adhesiveness was found to increase with addition of gums and storage. The inclusion of gums in starch based jelly candy seemed to better retard retrogradation and give rise to softer (lower hardness and gumminess), more elastic (higher springiness) and more moist (higher adhesiveness) textural properties.

4. Conclusions

It may be worthwhile to note that the understanding of the structure-property-processing relationship of a complex food system is necessary and it could pave for a path for developing a better consumer-acceptable product quality.

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