



RESEARCH PAPER

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Effect of xanthan and carboxymethyl cellulose gums on some physical and sensory characteristics of baslvqShamsborhany Roonak^{1*}, Ghiassi Tarzi Babak², Seyyedineh Ardabili Mehdi²¹*Food Science and Technology, Science and Research Branch, Islamic Azad University, Tehran, Iran*²*College Of Food Science & Technology, Science and Research Branch, Islamic Azad University, Tehran, Iran***Key words:** Wheat starch, DSC, xanthan, carboxymethyl cellulose, retrogradation.

Article published on February 01, 2015

Abstract

Starch and gums are hydrocolloids which frequently used in food systems to provide proper texture, moisture and shelf life of the food. Starch-gum interaction in food systems can change the starch gelatinization and physical properties. The present study aimed to investigate the influence of the addition of xanthan gum and carboxymethyl cellulose at the concentrations of the (0.1, 0.2 and 0.3% (w / w) on the physical properties (moisture content, retrogradation, breaking force and rupture strength) and sensory properties of wheat starch and after storage of 1,30 and 60 days were analysed. The texture profile analysis(TPA) and Differential Scanning Calorimetry (DSC) of the starch gels were also evaluated. All hydrocolloids were also able to reduce the loss of moisture content during baslvq storage, reducing the dehydration rate of starch gel. xanthan gum and carboxymethyl cellulose at the concentration(respectively 0.2%, 0.1%(w/w) in the mixed gel appeared to result in a lower change in textural properties during storage. Differential scanning calorimetry (DSC) was used to characterize the behaviour of wheat starch with and without additives. Results showed that xanthan gum and carboxymethyl cellulose gum retarded the retrogradation of wheat starch at investigated temperature (6 °C refrigeration). Carboxymethyl cellulose gum retarded retrogradation of wheat starch more than xanthan gum. Additions of hydrocolloids caused a decrease in gelatinization enthalpy of wheat starch. Sensory evaluation showed that samples containing 0.3 and 0.2% xanthan gum had the highest quality.

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Introduction

Baslvq is a traditional product of Iranian confectionary and ingredients include starch, water and sugar which also known as starch gel. Baslvq is one of the traditional sweets product consumed in Iran. The baslvq is usually 2 to 3 cm long and 3 to 4 cm wide with a thickness of about 2.5cm. It is produced by mixing all its ingredients to proper consistency and cooling for three hours. Starch is one of the most abundant polysaccharides in nature competing only with cellulose. Its application depends on its physical and chemical properties, which vary according to the botanical source. Starch is widely used in food systems as thickening, gelling, and stabilizing agent (Eliasson And Gudmundsson, 2006), and its functionality influences or controls properties such as texture, volume, consistency, moisture retention, and shelf life of the food. Starch granules are composed of two polysaccharides, linear amylose, and branched amylopectin molecules held together by hydrogen bonds, either directly or via hydrate bridges (Song *et al.*, 2006). The most important process that happens during starch heating in water is called gelatinization. During heating, starch crystalline structure is broken and the granule absorbs water and irreversibly swells. Swollen granules, fragments from granules, and leached amylose constitute a complex system in which the granules are dispersed in a continuous phase of amylose-water (Hermansson and Svegmarm, 1996).

Retrogradation is basically a process of crystallization of gelatinized starch molecules that takes place due to a strong tendency of hydrogen bond formation between adjacent molecules (Hoover, 2001). Retrogradation rate is affected by the ratio of amylose and amylopectin, molecular size, temperature, pH, lipids, hydrocolloids, sugar and botanical sources (Aee *et al.*, 1998). Differential scanning calorimetry (DSC) is a thermal analysis technique that has been widely used to characterize the phase transition including crystallization, melting transition and glass transition (Koo *et al.*, 2005; Babic *et al.*, 2006; Aćkar *et al.*, 2010). Analysis of

DSC data can provide additional information about starch, such as its structure and composition, its interaction with other components, the effects of water, and related properties.

Even though information about starch-hydrocolloid systems exists, there are still unknown aspects because of the complexity of such systems. It is generally accepted that each hydrocolloid affects pasting properties of gel starch in a different way (Bahnansey and Breene, 1994; Rojas *et al.*, 1999). This can be attributed to many factors, mainly the molecular structure of hydrocolloids and/or ionic charges of both starches and hydrocolloids (Sudhakar *et al.*, 1995; Shi and BeMiller, 2002).

The use of hydrocolloids in starch-based products, due to their functional properties (mainly stabilizing), has been a good alternative to modify starches (enhance stability, modify texture, reduce costs, control moisture and retard retrogradation) (Weber *et al.*, 2009; Achayuthakan and Supphantharika, 2008; Liu; Eskin and Cui, 2006; Song *et al.*, 2006; Chaisawang and Supphantharika, 2005).

xanthan gum (polysaccharide produced by *Xanthomonas campestris*) and carboxymethyl cellulose are the most frequently used hydrocolloids in food products (Mali *et al.*, 2003).

A synergic effect between hydrocolloid and starch has been observed by many researchers (Biliaderis *et al.*, 1997; Chaisawang and Supphantharika, 2006; Song *et al.*, 2006; Kim; Yoo, 2006), but the factors that influence this interaction are still not totally clear.

According to Biliaderis *et al.* (1997) and Chaisawang and Supphantharika (2006), the gelatinization characteristics of starch are directly influenced by 1) the morphological structure of gum present in the continuous phase (gel matrix) where the starch granules are involved, 2) the swelling power between the granules, and 3) the electrostatic interactions between starch granules and gum molecules.

The objective of this study was to investigate the effect of xanthan gum (XG) and carboxymethylcellulose (CMC) on the thermal, and physicochemical properties of wheat starch gel.

Materials and Methods

Materials

Wheat starch consisted of 25% amylose and 75% amylopectin (w/w) (International..., 1987). Moisture content was measured after 24 h of drying at 105 °C and was 14% on a dry matter basis. Specific density measurements using the picnometric method gave a value of 1.29 g cm⁻³ and ash was 13%. Xanthan gum and carboxymethyl cellulose were obtained from commercial sources of food gums.

Preparation of the starch-hydrocolloids mixtures

The starch-hydrocolloids mixtures evaluated in this study were prepared dispersions with 10.0% (w/w) of total solids. Ten experiments were carried out; one included wheat starch without hydrocolloids and the other ones included mixtures of starch with hydrocolloids (Table1) .

Table 1. The type of gums and concentrations.

Gums	Concentrations
Xanthan	0.1, 0.2, 0.3 % (w/w)
Carboxymethyl Cellulose	0.1, 0.2, 0.3 % (w/w)

The starch-XG mixture was prepared by dispersing the xanthan gum in water under agitation and heat treatment at 80 °C for complete dissolution of the gum. After that, it was cooled at 25 °C, and the starch was added, also under agitation.

The starch-CMC mixtures was prepared by dispersing the CMC gum in water under agitation. After complete dissolution of hydrocolloid, the starch was added under agitation.

Moisture content

Baslvqs were cut in to small pieces (5g). Moisture content was determined by measuring weight loss products, upon drying in an oven at 105 until constant weight (AACC 44-15.02).

Thermal properties

Gelatinization and retrogradation properties of starch and starch-hydrocolloids mixtures were determined using a differential scanning calorimeter (DSC Pyris 1, Perkin Elmer, Norwalk, USA). Starch-water and starch-hydrocolloid mixture samples (0.3 g), were weighed in aluminum pans and sealed. The weighed samples were scanned at a rate of 10 °C/minutes over a temperature range of 25-110 °C. An empty pan was used as a reference, and the analysis was performed in one. The changes in enthalpy (ΔH in J/g of dry starch), onset temperature (T_o), peak temperature (T_p) and conclusion temperature (T_c) for gelatinization and retrogradation were obtained from the exotherm DSC curves.(Suphantharika, & Chaisawang, 2005; Pongsawatmanit *et al*, 2007)

Texture profile analysis (TPA)

The gel texture was determined using a TA-XT2i Texture Analyzer (Stable Micro Systems Ltd, Surrey, UK) equipped with a Texture Expert for Windows Version 1 equipment software and a 5 kg load was used for force calibration. These specimens were subjected to deformation levels of 50% of the original height for single compression cycle at room temperature (25 °C). Hemispherical probe (P/0.5HS) punched into the gel with a constant crosshead velocity of 1 mm/s. The textural parameters, i.e., breaking force and rupture strength, were measured according to the definitions of Pons and Fiszman (1996). The breaking force is defined as the Gravity observed during the compression cycle, whereas the rupture strength is the first significant peak where the force falls off during the probe penetrates in to the product.

Sensory Evaluations

The baslvqs quality and sensory attributes were evaluated after 1, 30, 60 days of production. Sensory evaluation was accomplished by the 7-point hedonic scale determination by 8 trained panelists. The trained panelists evaluated: color intensity, surface adhesion, stiffness, chewing ability, flavor and aroma.

Statistical analysis

All samples were analyzed in triplicate and all data were reported as mean values and standard deviations. Statistical analysis of the results was conducted by the variance analysis, and differences among samples were determined by the Duncan's multiple range test at the 5% level using Statistica 7.0 (Statsoft, 2007).

Results and discussion

Moisture content

The effect of hydrocolloid addition is summarized in (Fig. 1 and Table 2). The addition of hydrocolloids to starch gel increased its water absorption significantly ($P < 0.05$). Water absorption was increased by addition of hydrocolloid, highest absorption was observed when adding xanthan 0.1

and 0.2%, results that agree with Friend, Waniska, and Rooney (1993) and Rosell *et al.* (2001a,b); that effect has been attributed to the hydroxyl groups in the hydrocolloid structure which allow more water interactions through hydrogen bonding.

During storage the most evident changes are related to moisture content loss and gel hardening. In Fig. 1, it can be observed the effect of several hydrocolloids on the moisture retention of starch gel. starch gels containing hydrocolloid showed lower loss of moisture content, therefore higher water retention in the starch gel compared to the control. No additional retention was observed by increasing the amount of hydrocolloid added to the starch gel recipe. (Bárceñas and Rosell, 2006; Ghodke, 2009; Guard *et al.*, 2004; Friend *et al.*, 1993).

Table 2. Analysis of variance of total moisture after 1,30 and 60 days.

Source variation	d.f	Mean Square		
		Total moisture% after 1 day	Total moisture% after 30 days	Total moisture% after 60 days
Control versus the rest	1	147.007**	58.073**	39.858**
gum	1	69.620**	1.227	2.276
concentration	2	18.462**	9.252*	9.602**
Gum*concentration	2	14.420**	8.557*	14.927**
Error	14	2.150	2.082	1.101
CV (%)		5.278	6.133	4.885

*, **: Significant at 1% and 5% probability level, respectively.

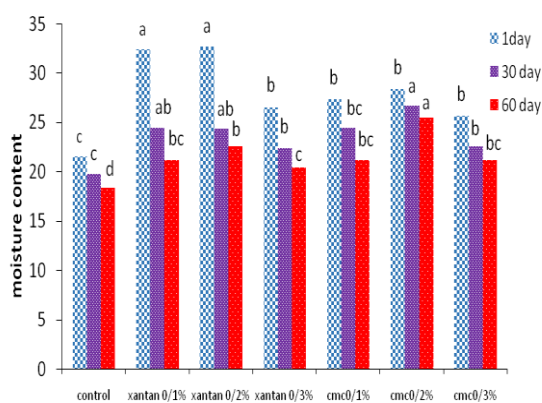


Fig. 1. Influence of different hydrocolloids in the moisture content of samples 1, 30 and 60 day after production. Hydrocolloid name is followed by the dosage of the hydrocolloid expressed in percentage. CMC: Carboxymethyl cellulose; Different superscripts indicate significant differences at $P < 0.05$.

Thermal properties

Differential scanning calorimetric (DSC) is a powerful thermal analysis technique that is used to examine transitions such as crystallization and melting by measuring heat absorption or heat loss from a sample as a function of temperature or time. In this paper, DSC was used to determine the effect of xanthan and CMC gums on the gelatinization of starch after 1, 30 and 60 days of storage at 6 °c refrigeration. Onset (T_o), peak (T_p), conclusion (T_c) gelatinization temperatures, and enthalpy (ΔH) determined by the DSC for the starch gel with and without gum addition are shown in Table 3 and 4. Rojas *et al.* (1999) and Cameron *et al.* (1993) found that hydrocolloids had little or no effect on the peak gelatinization temperature of wheat and waxy maize starches. Jouppila and Roos (1997) proposed that melting

temperatures (onset, peak and conclusion temperature) of crystallized starch increases with increasing storage temperature. Similar results for wheat starches were obtained in this study. The T_o and T_c values were decrease by the addition of gum, the lowest value was observed for the starch-xanthan gum mixture with content 0.1 and 0.3 percent after 1and 30 days of storage at 6 °c refrigeration. On the other hand, Chaisawang and

Suphantharika (2006) reported that the addition of xanthan and guar gums at the concentration of 0.35% in relation to starch weight increased the onset temperature of native cassava starch suspension (6.0 % w/v). In the present study, the higher increases observed for onset and complete temperatures were of 0.2 and 0.3%, when carboxymethyl cellulose gum was added to starch.

Table 3. Exothermal gelatinization characteristic of wheat starch (WS) suspensions with and without added xanthan gum (XG) and Carboxymethyl cellulose gum (CMCG). After 1 day at 6°C.

sample	T_o [°C]	T_p [°C]	T_c [°C]	ΔH_g [J/g]
WS	110.01	121.37	127.21	150.63
WS+XGo.1%	92.92	113.57	125.10	282.48
WS+XGo.2%	106.46	121.02	150.11	121.65
WS+XGo.3%	102.17	114.39	125.01	181.36
WS+CMCGo.1%	105.71	117.15	125.09	212.07
WS+CMCGo.2%	108.56	122.31	127.02	91.64
WS+CMCGo.3%	116.08	124.06	126.04	51.98

Gelatinization parameters: T_o , onset temperature; T_p , peak temperature; T_c , conclusion temperature; ΔH_g , gelatinization enthalpy.

Table 4. Exothermal gelatinization characteristic of wheat starch (WS) suspensions with and without added xanthan gum (XG) and Carboxymethyl cellulose gum (CMCG). after 30 and 60 days at 6 °C.

<i>After 30 days at 6°C</i>				
sample	T_o [°C]	T_p [°C]	T_c [°C]	ΔH_g [J/g]
WS	108.67	125.37	135.25	222.68
WS+XGo.1%	100.19	117.00	126.20	157.04
WS+XGo.2%	107.14	119.22	126.09	108.14
WS+XGo.3%	101.94	114.00	124.88	125.54
WS+CMCGo.1%	104.03	117.89	126.01	122.72
WS+CMCGo.2%	128.83	132.92	135.09	17.57
WS+CMCGo.3%	111.78	127.92	130.25	117.22

Gelatinization parameters: T_o , onset temperature; T_p , peak temperature; T_c , conclusion temperature; ΔH_g , gelatinization enthalpy.

Continued Table 4.

<i>After 60 days at 6°C</i>				
sample	T_o [°C]	T_p [°C]	T_c [°C]	ΔH_g [J/g]
WS	111.08	128.44	145.11	261.35
WS+XGo.1%	101.63	114.86	140.21	213.95
WS+XGo.2%	107.23	117.80	126.02	140.41
WS+XGo.3%	101.91	115.33	126.03	204.14
WS+CMCGo.1%	109.16	117.04	127.03	91.85
WS+CMCGo.2%	108.38	120.66	127.05	96.22
WS+CMCGo.3%	113.95	124.14	125.01	99.41

DSC analysis has often been applied to measure starch recrystallization (retrogradation) (Funami *et al.*, 2005; Koo *et al.*, 2005; Babić *et al.*, 2009). It is well known that the enthalpy of melting of recrystallized starch increases throughout storage time. Retrogradation exotherms of starch gel with and without addition of hydrocolloids were observed after 1, 30 and 60 days of storage at 6 °c refrigeration.

A decrease in gelatinization enthalpy (ΔH) was observed, however, when gums were added as compared to the control, which can be attributed to a reduction in water availability causing partial gelatinization of crystalline regions in starch granules and effect of starch-hydrocolloids interactions (Rojas *et al.*, 1999; Funami *et al.*, 2005; Chaisawang and Supphantharika, 2006). Sample with addition of CMC gum (0.2 %) had the lowest retrogradation enthalpy (ΔH) after 30 days at 6 °C (17.57 J/g; control sample 222.68 J/g). Generally, hydrocolloids at a lower concentration (0.1 %) had a greater effect on wheat starch gel retrogradation decrease.

wheat starch samples without addition of XG and CMCG showed greater retrogradation than the samples with hydrocolloids added after 1, 30 and 60 days of storage at 6 °c refrigeration. Addition of XG and CMCG increased or decreased the melting temperatures of recrystallized wheat starch depending on type and concentration of hydrocolloids, storage time and temperature of storage.

Texture profile analysis (TPA)

Textural changes occurred after 1, 30 and 60 days of storage at 6 °c refrigeration of starch gel with and without xanthan and carboxymethyl cellulose gums are presented in Fig. 2 and Table 5 for gel breaking force. The textural parameters, i.e., breaking force and rupture strength values were significantly ($P < 0.05$) decrease by the addition of gums as compared to the control. In general, the results of breaking force showed the same tendency as those of the rupture strength. Considering the starch gel without hydrocolloids showed the most pronounced increase in both breaking force and rupture strength, which led to gel hardening and consequently unacceptable texture after 60 days of storage at 6 °c refrigeration. Therefore, the textural properties of the starch gel might be dominated by those of the pure xanthan. This hypothesis was confirmed by the work of Giannouli and Morris (2003), who found that much stronger gel networks of pure xanthan were formed, when xanthan solutions were frozen and thawed. xanthan gum and carboxymethyl cellulose at the concentration(respectively 0.2%, 0.1% (w/w) in the mixed gel appeared to result in a lower change in textural properties during storage. Biliaderis, Arvanitoyannis, Izydroczyk, and Prokopowich (1997) proposed that the effect of addition of hydrocolloids results from two opposite phenomenon. First, an increase in the rigidity as a consequence of the decrease in the swelling of the starch granules and amylose, and, secondly, a weakening effect on the starch structure due to the inhibition of the amylose chain associates, although the weight of each effect will be dependent on the specific hydrocolloids.

Table 5. Analysis of variance of Breaking force after 1,30 and 60 days.

Source variation	d.f	Mean Square		
		Breaking force(N) after 1 day	Breaking force(N) after 30 days	Breaking force(N) after 60 days
Control versus the rest	1	0.936**	0.056	0.147
gum	1	6.251**	6.748**	2.408**
concentration	2	2.460**	3.351**	2.111**
Gum*concentration	2	1.616**	1.981**	1.274**
Error	14	0.0812	0.0412	0.0451
CV (%)		16.800	8.261	6.048

*, **: Significant at 1% and 5% probability level, respectively.

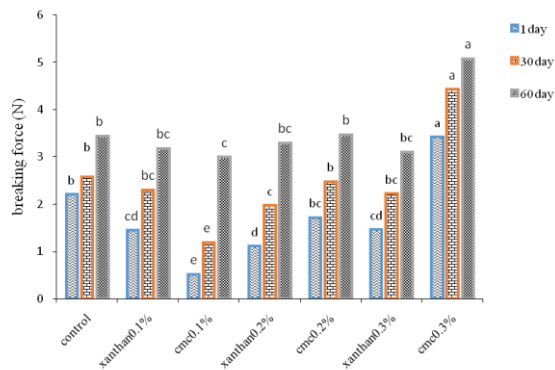


Fig. 2. Effect of hydrocolloids on starch gel breaking force (gravity: N=mg). CMC, Carboxymethyl cellulose; Different superscripts indicate significant differences at $P < 0.05$.

CMC and xanthan showed the extreme effects, xanthan reduced the gel breaking force and rupture strength, given softer gel than the control. Conversely, the addition of CMC increased the breaking force of the starch gel. In the case of hydrocolloids, mainly xanthan, the softening effect should be attributed to their water retention capacity, and a possible inhibition of the amylopectin retrogradation, since xanthan preferential binds to starch (Collar, 2001), and in consequence avoid starch–gluten interactions.

Sensory Characteristics of Baslvq

The effect of various hydrocolloids on sensory properties of baslvq is shown in Table 6 and 7. Each attribute was scored from 1 (lowest) to 7 (highest). According to Table 6, all gum-added baslvq received scores higher than 3 in all evaluated parameters. Regarding the color intensity and Surface adhesion, baslvq containing CMC received the smallest score,

being even the worst at the low concentration (0.1%) while 0.3% xanthan scored the highest ($P < 0.05$). With the exception of 0.3 % CMC, other hydrocolloides improved significantly baslvq, as compared to the control baslvq ($P < 0.05$). Baslvq chew-ability received significantly ($P < 0.05$) better scores when hydrocolloids were added. With the exception of CMC at the highest concentration (0.3%), no differences were observed in taste and in the case of aroma, only a significant difference was obtained with CMC at 0.3%, concluding that hydrocolloids do not affect those sensory properties of baslvq. Regarding the Stiffness, 0.3% CMC got the highest scores, while baslvq with 0.3%_ xanthan received the smallest score. Xanthan improved overall acceptability of baslvq ($P < 0.05$). Regarding the overall acceptability, baslvq containing CMC had the lowest score (among hydrocolloid-added baslvqs), being the worst at the highest concentration (0.3%CMC). Among different sensory attributes, the chew ability and Surface adhesion of the resulting baslvq samples showed higher scores than the control baslvq. The improving effect of the xanthan on the sensory quality of baslvq could be due to their influence on the gel texture that yields softer gels. The reasons for the adverse effects of 0.3% CMC on some sensory attributes are not completely understood, although some hypotheses have been proposed. For example, it seems that through a strong interaction with the starch. Strengthening effect of 0.3% CMC observed in the Texture profile analysis may explain the low scores in baslvq chewability, and texture. Sensory analysis allows concluding that addition of xanthan improve sensory properties of baslvq, giving higher scores for overall acceptability.

Table 6. Analysis of variance of sensory evaluation.

Source variation	d.f	Mean Square					
		Color intensity	Surface adhesion	Stiffness	Chew-ability	Taste an aroma	Overall acceptability
Repetition	7	0.31	0.16*	0.041	0.30	0.016	0.83*
Treatments	6	26.2**	33**	33.58**	31.4**	9.79**	17.5**
Error	42	0.168	0.18	0.100	0.179	0.098	0.82
CV (%)		9.6	10.6	7.9	10.9	8.9	16.1

*, **: Significant at 1% and 5% probability level, respectively

Table 7. Effect of different hydrocolloids on the baslvq sensory evaluation

<i>Treatments</i>	<i>Levels(%)</i>	<i>Color intensity</i>	<i>Surface adhesion</i>	<i>Stiffness</i>	<i>Chew-ability</i>	<i>Taste an aroma</i>	<i>Overall acceptability</i>
Control	-	2.125 ^f	1.375 ^g	5.875 ^b	1.000 ^g	3.875 ^a	3.750 ^d
Xanthan	0.1	5.250 ^c	5.000 ^c	3.000 ^e	5.000 ^c	4.125 ^a	5.250 ^{ab}
	0.2	6.000 ^b	6.000 ^b	1.875 ^f	5.625 ^b	3.875 ^a	5.375 ^a
	0.3	6.750 ^a	7.000 ^a	1.375 ^g	6.500 ^a	3.875 ^a	4.625 ^{bc}
CMC	0.1	2.250 ^f	2.250 ^f	5.000 ^c	2.875 ^e	4.125 ^a	3.750 ^d
	0.2	3.375 ^e	2.750 ^e	4.000 ^d	4.000 ^d	3.875 ^a	4.125 ^{cd}
	0.3	4.000 ^d	4.000 ^d	6.875 ^a	2.125 ^f	1.125 ^b	1.000 ^e

^a Values are the mean of three replications. Different letters in each column indicate significant differences (P< 0.05). CMC: carboxymethyl cellulose.

Conclusions

The present study showed that addition of some hydrocolloids improved the physical properties of baslvq. Baslvq water absorption, which has a great effect on gel quality and its shelf-life, was increased by all hydrocolloids tested and xanthan had the highest effect. The addition of xanthan gum and carboxymethyl cellulose gum at the concentrations of the (0.2 ,0.1(w / w) starches caused a decrease in gelatinization enthalpy values. Hydrocolloids slowed retrogradation of starch gels at investigated temperature (6°C refrigeration). xanthan gum suppressed retrogradation of wheat starch more than carboxymethyl cellulose gum, on the other hand, xanthan gum had a greater effect on reduction of the recrystallization of wheat starch. CMC and xanthan showed the extreme effects, xanthan reduced the gel breaking force and rupture strength, given softer gel than the control. Conversely, the addition of CMC increased the breaking force of the starch gel. Sensory analysis revealed that when hydrocolloids were added to the wheat starch formulation, some sensory indexes of the gel, such as chew-ability, were enhanced more pronouncedly than the other indexes. Addition xanthan and CMC in to formulation resulted in an increase in overall acceptability of baslvq, and the highest improvement was brought about by xanthan. Overall, the best treatment in terms of

starch gel quality was obtained by the addition of 0.2% xanthan to the starch.

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