Iran Agricultural Research (2017) **36**(**1**) 1-6

Effects of Heat-Moisture Treatment on Physicochemical Properties of Wheat Starch

M. Majzoobi[*1], F. Roushan[1], M. Kadivar[2], A. Farahnaky[1], N. Seifzadeh[1]

[1]Department of Food Science and Technology, College of Agriculture, Shiraz University, Shiraz, I. R. Iran
[2]Department of Food Science and Technology, Isfahan University of Technology, Isfahan, I. R. Iran.

* Corresponding Author: majzoobi@shirazu.ac.ir

ARTICLE INFO

***Article history*:**

Received **24 May 2014**
Accepted **6 March 2016**
Available online **14 November 2016**

***Keywords*:**

Heat-moisture treatment
Physicochemical properties
Treatment time
Wheat starch

ABSTRACT- Modified starch has wider applications than the native starch in different food products. Therefore, a variety of methods have been developed in order to produce modified starches including chemical, physical and enzymatic methods. Amongst them, physical modifications have been known as chemical-free and hence are more acceptable. Heat-moisture treatment is a physical method for starch modification. The properties of the heat-moisture treated starch depend upon the source of starch and treatment conditions including time and temperature. The main objective of this study was to determine the functional properties of heat-moisture treated wheat starch under different conditions. Therefore, wheat starch was heat-moisture treated at 105 °C for 14 and 16 h and the physicochemical properties of the starch were studied. The results showed that the heat-moisture treatment caused the formation of some cracks and spots on the surface of the starch granules, while preserving the whole integrity of the granules. The water solubility of the samples increased while increasing the treatment time had negative effects on water solubility. The x-ray diffraction pattern of the samples remained unaffected but the degree of crystallinity increased significantly. The gelatinization temperature increased while the enthalpy of gelatinization decreased as a result of modification. Gel texture became harder, less adhesive and elastic after modification. Increasing the treatment time dramaticallyenhanced all these properties.

INTRODUCTION

Wheat starch is one of the most common types of starches worldwide with numerous applications in food and non food products. However, like other native starches, it has some limitations such as low water solubility, rapid retro gradation and poor film properties. Therefore, different modification methods have been applied to increase starch functional properties (Hermansson and Svegmark, 1996; Zavareze and Guerra Dias, 2011). Amongst the different modification methods such as chemical, physical and enzymatic methods, physical modification has been known as chemical free and safe to consume and hence has attracted much attention (Rutenberg and Solarek, 1984). One of the methods used for physical modification of starch is heat-moisture treatment. Heat-moisture treatment involves treatment of starch granules at moisture levels lower than 35% (w/w) held for 15 min to 16 h and at a temperature range that is below gelatinization temperature but higher than the glass transition temperature (84-120 °C). The mentioned conditions can change the physicochemical properties of the starch mainly because of the increase in the starch molecular interactions (Hoover, 2001).

Several studies have focused on the functional properties of heat-moisture treated starches of different sources including potato, cassava (Gunaratne and Hoover, 2002), pea, lentil (Chung et al., 2009), rice (Hormadok and Noomhorn, 2007), sorghum (Olayinka et al., 2008), corn (Chung et al., 2009) and wheat starches (Hoover and Vasanthan, 1994). In the latter study conducted on wheat starch, the effects of different moisture contents at a constant temperature were studied. Sun et al. (2014 a, b, 2015) reported that the heat-moisture treatment can strengthen the interactions between wheat starch and xylitol, maltitol and erythritol. Chen et al. (2015) indicated that the heat-moisture treatment can reduce the *in vitro* digestibility of wheat starch. Other studies spotlighted the effects of treatment time and moisture content on the functional properties of corn (Francoet al. 1995; Chung et al., 2009), rice (Hormadok and Noomhorn, 2007) and potato (Vermeylen et al., 2006) starches. The results of these studies indicated that the treatment conditions including time, moisture content and source of starch have great influences on the physicochemical properties of the heat-moisture treated starch. Therefore, it is of great importance to determine the effect of different treatment conditions on the functional properties of the starches. Based on the literature, there is not enough information to show the effects of different conditions

on the heat-moisture treated wheat starch. The main aim of this study was to determine the effects of heat-moisture treatment duration time on the functional characteristics of wheat starch.

MATERIALS AND METHODS

Wheat starch was purchased from Fars-Glucosin starch producing factory, Marvdasht, Fars, Iran. The wheat starch contained 9.04 ± 0.42% moisture; 0.24 ± 0.01% protein; 0.37 ± 0.10% ash and 0.69 ± 0.01% lipid as determined by the Approved Methods of the AACC (AACC, 2000) and 26.35 ± 0.21% amylase content as determined after de-fatting of starch with 80% methanol using the iodine method as described by Williams et al., (1970).

Heat-Moisture Treatment

Wheat starch with initial moisture content of 9.5% was weighed into glass jars and then moisture content increased to 25% by water spraying and totally mixing using a spatula. The closed jars were left at ambient temperature overnight for moisture equilibration, then placed in an oven set at 105 °C for 14 and 16 h, individually. After that, the jars were cooled down to room temperature, opened and the heat-moisture treated samples were dried in an oven to a moisture content of about 10%. The dried samples were sieved to avoid clumping and to obtain particle size of 75-120 µm, then packed in polyethylene bags and kept at room temperature for further experiments explained in the following sections. The heat-moisture treated samples obtained after 14 and 16 h of treatment were named HMT1 and HMT2, respectively.

Starch Granules Morphology

A tiny amount of each sample was evenly stuck on a scanning electron microscopy (SEM) stub, coated with a thin layer of gold. Then the starch granules were observed at 20 kV by means of SEM (Model 5526, Cambridge, UK) (Majzoobi et al., 2012).

X-ray Diffraction

All of the starch samples were first placed in a relative humidity box containing super saturated NaCl for 5 days at room temperature to obtain similar relative humidity of 75%. X-ray diffraction pattern of the samples were determined with an X-ray diffractometer (Model D8 Advance, Germany) (Majzoobi et al., 2012). The degree of starch crystallinity was calculated by dividing the area under the peaks to the total curve area both determined by using the instrument software (EVA, Version 9.0).

Thermal Properties

Thermal properties of the samples were determined using a differential scanning calorimetery (DSC) (Perkin-Elmer, Beaconsfield, UK). Starch (3 mg) was weighed in an aluminium pan and 6 mg of water was added. The pan was sealed hermetically and equilibrated overnight at room temperature. Then, it was placed in the DSC instrument and scanned at a heating rate of 10 C/min. An empty pan was used as the reference. The starch thermal transitions defined as To (onset), Tp (peak), Tc (conclusion) and enthalpy of gelatinization H J/g were obtained from the DSC curves.

Textural Properties

Starch gels were prepared by heating a starch suspension (10% starch solid) at 95 °C for 30 min and then cooling the sample to 50 °C. The hot paste was poured into a cylindrical plastic container with diameter and height of 1×1cm and stored for 24 h at 4°C before measurement. The textural properties of the gels were determined using a Stable Micro Systems TAXT-2i Texture Analyzer. Samples were compressed at a pre-test speed of 5.0 mm/s, test speed of 2.0 mm/s, post test speed of 5.0 mm/s, time interval of 10 s and strain deformation of 25% using a cylindrical plunger with diameter of 10 mm. The texture profile parameters derived from the force-distance curve were the maximum force achieved through the first bite of TPA (hardness), elasticity (springiness) as a ratio of the time recorded between the start of the second area and the second probe reversal to the time recorded between the start of the first area and the first probe reversal. Cohesiveness as the ratio between the positive area under the second peak and the area under the first peak and stickiness as the negative work between the two cycles were also calculated (Steffe, 1996).

Statistical Analysis

The experiments were performed in a completely randomized design. Each experiment was performed in triplicates. Analysis of variance (ANOVA) was performed and the results were separated using the Multiple Ranges Duncan's test ($P<0.05$) by making use of statistical software of Statistical Package for Social Sciences 16 (SPSS) (SPSS, Inc., New Jersey, USA).

RESULTS AND DISCUSSION

Effect of Heat Moisture Treatment of Starch Solubility

The results (Fig. 1) showed that the heat-moisture treatment significantly increased the solubility of the samples compared to the native starch. However, increasing the treatment time significantly reduced the solubility of the samples. Similar findings were reported for the heat-moisture treated wheat and potato starches (Kulp and Lorenz, 1981). The heat-moisture treatment can increase the interaction between starch molecules particularly in the crystalline region, resulting in lower solubility of amylopectin. Consequently, the separation and leaching of the amylose molecules during boiling of the samples will be easier resulting in higher solubility of the heat-moisture treated starches. Increasing the treatment time can enhance the junction zones of the

starch molecules and the crystalline regions which may prevent the exit of the amylose resulting in a reduction in water solubility.

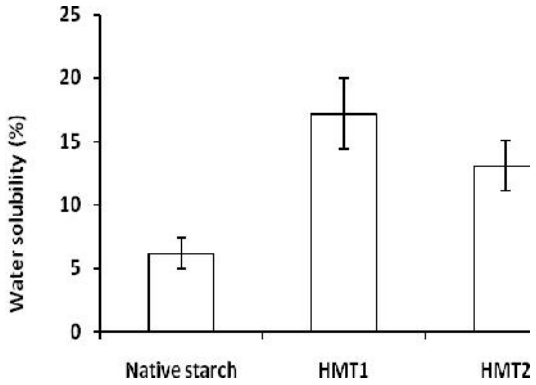

Fig. 1. Effect of heat-moisture treatment on water solubility of wheat starch. HMT1: heat-moisture treated wheat starch for 14 h; HMT2: heat moisture treated starch for 16 h. Different letters on the bars indicate statistical difference (P<0.05).

Effects of Heat-Moisture Treatment On The Granules Morphology

Granule morphology, size and surface properties have a great role in many food and non-food utilizations of starch. The electron micrographs of the samples are given in Fig. 2. The two types of the granules common for wheat starch, i.e. the large granules (A-type) and small granules (B-type) can clearly be observed for the native wheat starch (A). Heat-moisture treatment and its length had no significant effect on the integrity, size or shape of the samples. Similar observations were reported for maize (Hoover and Manuel, 1996), wheat (Hoover and Vasanthan, 1994), finger millet (Adebowale et al., 2005) and rice (Khunae et al., 2007) starches. However, the exterior of the granules was slightly affected particularly by increasing the treatment time. It seems that the surface of the granules was shrunk and some cracks appeared as the result of heat-moisture treatment. Such effect was more pronounced for the samples prepared at longer treatment times (B and C). Formation of some cracks on the surface and some holes inside the granules of the heat-moisture treated maize and potato starches have also been reported (Kawabata et al., 1994).

Effects of Heat-Moisture Treatment on The X-Ray Diffraction And Degree of Crystallinity of Starch

The crystallinity of the granules is mainly related to the double helixes formed by the branches of amylopectin and is normally between 15 and 45% (Hoover, 2001). Determination on the X-ray pattern of the starch samples (Fig. 3) showed that the heat-moisture treatment had no significant (P<0.05) effect on the X-ray diffraction pattern of the samples. The X-ray pattern of all starches was of the A-type representative of cereal starches, with spacing at 3.8, 4.8, 5.2 and 5.8 A°. The only difference between the samples was related to their degree of crystallinity that increased with increasing the treatment time. The degree of crystallinity of native starch was 35.76% that increased significantly to 36.86% and 37.56% after 14 h and 16 h of treatment, respectively. Similar to the finding of this study, an increase in the intensity of the X-ray diffraction of corn starch and sweet potato starches has been reported in previous studies (Hoover and Manuel, 1996; Vieira and Sarmento, 2008). The increase in the degree of crystallinity of the samples is due to the displacement of the double helical chains within the starch crystals, resulting in a crystalline matrix that is more orderly than in native starch. In contrast to this study, a shift from B to A pattern after heat-moisture treatment of potato and true yam starches has been found which is related to the different origin of the starches. It is also found that the degree of crystallinity decreased after the treatment (Gunaratne and Hoover, 2002; Vermeylen et al., 2006). For corn starch, a reduction in the degree of crystallinity was reported after heat-moisture treatment (Vieira and Sarmento, 2008). The reduction in the degree of crystallinity of these starches after heat-moisture treatment is related to the increase in semi-crystalline lamella of the granules.

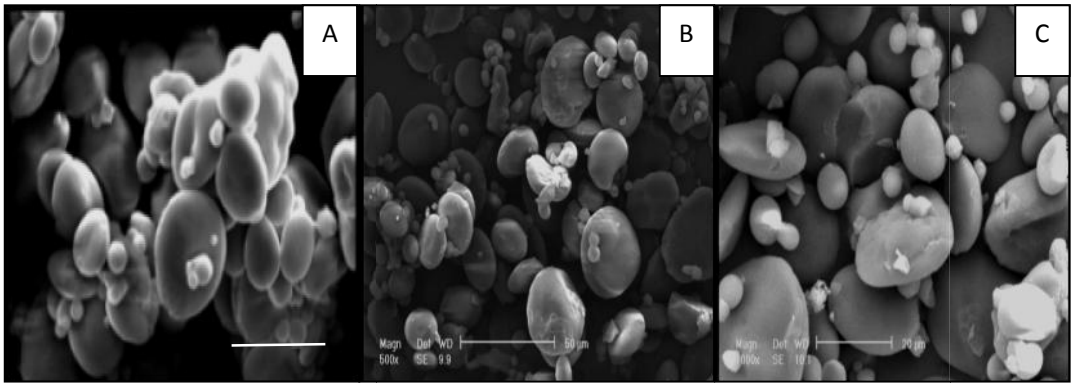

Fig. 2. Scanning electron micrographs of the native wheat starch granules (A), heat-moisture treated starch for 14 h (HMT1) (B) and heat-moisture treated starch for 16 h (HMT2) (C). Bars on the micrographs are 20 µm.

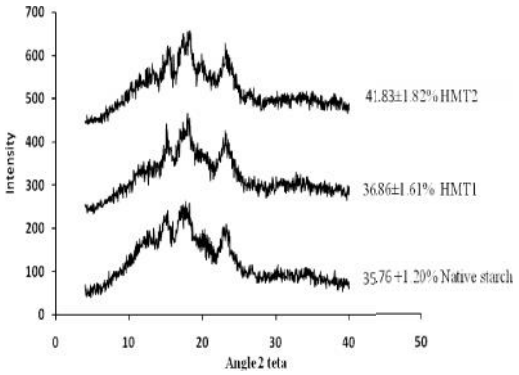

Fig. 3. The X-ray diffraction pattern of the native and heat moisture treated wheat starches. Values on the figure are the degree of crystallinity±standard deviation.

Effect of Heat-Moisture Treatment on Thermal Properties of Wheat Starch

The onset, peak and conclusion temperatures of the wheat starch increased significantly as the heat-moisture treatment time increased (Table 1). Similar results have been noted for potato, cassava, true yam (Gunaratne and Hoover, 2002), corn (Maache-Rezzoug et al., 2008), pea and lentil (Chung et al., 2009) starches. The increase in the gelatinization temperature may be related to the structural changes within the starch granules which involve amylose-amylose and amylose-lipid interactions. These interactions can reduce the mobility of the amorphous region. As a result, a higher temperature is required for the swelling and disruption of the crystalline regions to take place leading to an increased gelatinization temperature (Hoover and Vasanthan, 1994).

The results (Table 1) showed that the ΔH of the samples decreased after the heat-moisture treatment and with increasing the treatment time. A reduction of the ΔH occurring after the heat-moisture treatment has been reported in potato, cassava (Gunaratne and Hoover, 2002), jackbean (Lawal and Adebowale, 2005), corn, pea and lentil starches (Chung et al., 2009). For corn starch that was heat-moisture treated at 100 °C, no significant change in the ΔH was found (Hoover and Manuel, 1996). It has been reported that the decrease in the ΔHis a consequence of the distribution of double helices represented in the crystalline and non-crystalline regions of the granules (Gunaratne and Hoover, 2002). Furthermore, it has been indicated that the reduction on the ΔH after the treatment may be related to the partial gelatinization of amylose and amylopectin molecules that are less stable during heating (Hormadok and Noomhorn, 2007).

Effect of Heat-Moisture Treatment on Textural Properties of Wheat Starch

The textural properties of gel are determining factors affecting the quality of the final products. Molecular structure of starch, the volume and deformation of the granules and the interaction between the continuous and dispersed phases have a great role in the textural properties of the starch gels. Heat-moisture treatment had some effects on the textural properties of starch gels (Table 2).. The gel hardness increased as a result of heat-moisture treatment and treatment time significantly enhanced this parameter. A similar result was reported for heat-moisture treated rice starch by Hormadok and Noomhorn (2007). Heat-moisture treatment increases the molecular interactions which allow the formation of more junction zones in the continuous phase of the gel, resulting in a harder gel (Liu et al., 2000). The heat-moisture treatment and its time had no significant effect on the cohesiveness of the samples. Gel stickiness and elasticity reduced when the starch was modified but increasing the treatment time had no significant effect on these parameters.

Table 1. Thermal properties of the native and heat-moisture treated starches obtained after 14 h (HMT1) and 16 h (HMT2) of treatment

Sample	T_O (°C)	T_P (°C)	T_C (°C)	ΔH (J/g)
Native starch	60.18 ±0.63^c	65.32± 0.53^c	70.74± 0.93^c	11.21 ±0.19^a
HMT1	63.36 ±0.19^b	68.52± 0.01^b	76.71± 0.26^b	9.11 ±0.38^b
HMT2	64.13 ±0.42^a	69.52 ±0.27^a	78.99 ±0.50^a	8.14 ±0.53^c

Values are the average of triplicates ± standard deviation. Different letters in each column show the significant statistical difference between the averages (P<0.05).

Table 2. Textural properties of gels obtained from native and heat-moisture treated starches for 14 h (HMT1) and 16 h (HMT2).

Starch	Hardness (N/m^2)	Cohesiveness	Stickiness (N/m^2)	Elasticity
Native starch	14394.90± 0.00^c	0.65 ±0.04^a	1273.88± 127.38^a	1.07± 0.02^a
HMT1	26114.64± 509.55^b	0.71± 0.00^a	-1273.88 ± 127.38^b	0.97 ±0.01^b
HMT2	38853.50± 891.71^a	0.71 ±0.05^a	-891.71 ±524.77^b	0.98± 0.00^b

Values are the average of triplicates ± standard deviation. Different letters in each column show the significant statistical difference between the averages (P<0.05).

CONCLUSIONS

Heat-moisture treatment of the wheat starch resulted in the new functional properties. The time of this process appeared to be critical since it both affects the process length and the physicochemical characteristics of the modified starch. Therefore, selection of the appropriate processing time is of great importance. Increasing the processing time resulted in a series of changes from starch granules to starch molecules inside the granules. It can enhance the crystalline structure of the starch granules which can further influence other functional properties of the starch including water solubility, gel texture and thermal properties. Further studies are required to study the application of such starch in different food products and also to determine if the observed changes can have positive or negative effects on the quality of foods.

REFERENCES

AACC. 2000. Approved Methods of the AACC (10th ed). American Association of Cereal Chemists, St. Paul, MN.

Adebowale, K.O., Afolabi, T.A., & OluOwolabi, B.I. (2005). Hydrothermal treatments of finger millet *Eleusine coracana* starch. *Food Hydrocolloids*, 19, 974-983.

Chen, X., He, X., Fu, X., & Huang, Q. (2015). In vitro digestion and physicochemical properties of wheat starch/flour modified by heat-moisture treatment. *Journal of Cereal Science*, 63, 109-115.

Chung, H., Liu, Q., & Hoover, R. (2009). Impact of annealing and heat-moisture treatment on rapidly digestible, slowly digestible and resistant starch levels in native and gelatinized corn, pea and lentil starches. *Carbohydrate Polymers*,75, 436-447.

Franco, C.M., Ciacco, C.F., & Tavares, D.Q. (1995). Effect of heat-moisture traement on the enzymatic susceptibility of corn starch garnules. *Starch/Stärke*,47, 223-228.

Gunaratne, A., & Hoover, R. (2002). Effect of heat-moisture treatment on the structure and physichcoemical properties of tuber and root starches. *Carbohydrate Polymers***,** 49, 425-437.

Hermansson, A.M., & Svegmark, K. (1996). Developemnt in the understanding of starch functionality. *Trends in Food Science and Technology*,**7**, 345-353.

Hoover, R., & Vasanthan, T. (1994). Effect of heat-moisture treatment on the strcuture and physiccheomical properties of cereal, tuber and legume starches. *Carbohydrate Research*,252, 33-53.

Hoover, R., & Manuel, H. (1996). Effect of heat-moisture treatment on the structure and physicochemical properties of normal maize, waxy maize, dull waxy maize and amylomaize V starches. *Journal of Cereals Science*, 23, 153-162.

Hoover, R. (2001). Composition, molecular structure and physicochemical properties of tuber and root starches: A review. *Carbohydrate Polymers*, 45, 253-267.

Hormadok, R., & Noomhorn, A. (2007). Hydrothermal treatment of rice starch for imporvment of rice noodle quality. *LWT-Food Science and Technology*, 40, 1723-1731.

Kawabata, A., Takase, N., Miyoshi, E., Sawayama, W., Kimura, T., & Kudo, K. (1994). Microscopic observation and X-ray diffractometer of heat/moisture-treated starch granules. *Starch/Stärke*, 46, 463-469.

Khunae, P., Tran, T., & Sirivongpaisal, P. (2007). Effect of heat-moisture treatment on structural and thermal properties of rice starches differing in amylose content. *Starch/Stärke*, 59, 593-599.

Kulp, K., & Lorenz, K. (1981). Heat-moisture treatment of starches. I. Physichichemical properties. *Cereal Chemistry*, 58, 46-48.

Lawal, O.S., & Adebowale, K.O. (2005). An assessment of changes in thermal and physichoemmical para,eters of jack bean *Canavalia ensiformis* starch following hydrothermal modifications. *European Food Research Technology*, 221, 631-638.

Liu, H., Corke, H., & Ramsden, L. (2000). The efefct of autoclaving on the acetylation of ae, wx and normal maize starches. *Starch/Stärke*, 52: 353-360.

MaacheRezzoug, Z., Zarguili, I., Loisel, C., Queveau, D., & Buleon, A. (2008). Structural modifications and thermal transitions of standard maize starch after DIC hydrotermal treatment. *Carbohydrate Polymers*, 74, 802-812.

Majzoobi, M., Saberi, B., Farahnaky, A., & Tongdang, T. (2012). Physicochemical properties of cross-linked-annealed wheat starch. *Iranian Polymer Journal*, 218, 513-22.

Olayinka, O.O., Adebowale, K.O., & OluOwolabi, B.I. (2008). Effect of heat-moisture tretament on physichochemical properties of white sourghum starch. *Food Hydrocolloids*, 22, 225-230.

Rutenberg, M.W., & Solarek, D. (1984). Starch derivatives: production and uses. In: Whistler, R.L., BeMiller, J.N. & Paschall, E.F. (eds). Starch: Chemistry and Technology. Academic Press, London.

Steffe, J.F. (1996). *Rheological methods in food process engineering*. New York: Freeman Press.

Vermeylen, R., Goderis, B., & Delcour, J.A. (2006). An X-ray study of hydrothemally treated potato starch. *Carbohydrate Polymers*, 64, 364-375.

Vieira, F.C., & Sarmento, S.B.S. (2008). Heat-moisture tretament and enzymatic digestibity of Peruvian carrot, sweet potato and ginger starches. *Starch/Stärke*, 60**,** 223-232.

Williams, P.C., Kuzina, F.D., & Hlynka, I. (1970). A rapid colorimetric procedure for estimating the amylose content of starches and flours. *Cereal Chemistry* 47, 411–20.

Zavareze, E., & Guerra Dias, A.R. (2011). Impact of heat-moisture treatment and annealing in starches: A review. *Carbohydrate Polymers* 83, 317-328.

.

تحقیقات کشاورزی ایران (۱۳۹۶) ۳۶(۱)۱-۶

تاثیر فرایند حرارتی-رطوبتی بر ویژگی‌های فیزیکو شیمیایی نشاسته گندم

مهسا مجذوبی[۱*]، فریده روشن[۲]، مهدی‌کدیور[۳]، عسگر فرحناکی[۱]، نگین‌سیف‌زاده[۲]

[۱]گروه علوم و صنایع غذایی، دانشکده کشاورزی، دانشگاه شیراز، شیراز، ج .ا. ایران
[۲]گروه علوم و صنایع غذایی، دانشکده کشاورزی، دانشگاه صنعتی اصفهان، اصفهان. ج. ا. ایران.

*نویسنده مسئول

اطلاعات مقاله

تاریخچه مقاله:
تاریخ دریافت: ۱۳۹۳/۳/۳
تاریخ پذیرش: ۱۳۹۴/۱۲/۱۶
تاریخ دسترسی:۱۳۹۵/۸/۲۴

واژه‌های کلیدی:
فرایند حرارتی-رطوبتی
ویژگی های فیزیک وشیمیایی
زمان فرایند
نشاسته گندم

چکیده- نشاسته اصلاح شده کاربردهای بیشتری نسبت به نشاسته‌های طبیعی در محصولات غذایی مختلف دارد. بنابراین روش‌های متنوعی برای تولید نشاسته اصلاح شده وجود دارند که شامل روش‌های شیمیایی، فیزیکی و آنزیمی می باشد. در بین آنها اصلاحات فیزیکی به عنوان روش‌های فاقد مواد شیمیایی شناخته شده اند و قابل قبول‌تر می‌باشند. فرایند حرارتی-رطوبتی یک روش فیزیکی برای اصلاح نشاسته به شمار می‌رود. ویژگی‌های نشاسته فرایند شده به روش حرارتی-رطوبتی به منشاء نشاسته و شرایط فرایند شامل زمان و دما بستگی دارد. هدف اصلی از انجام این تحقیق ویژگی‌های نشاسته گندم حرارتی-رطوبتی شده در شرایط مختلف فرایند بود. لذا نشاسته گندم در دمای ۱۰۵درجه سانتیگراد در مدت ۱۴ و ۱۶ ساعت فرایند شد و ویژگی‌های فیزیکی شیمیایی آن بررسی گردید. نتایج نشان داد که فرایند حرارتی-رطوبتی باعث ایجاد شکاف‌ها و لکه‌هایی بر سطح گرانول‌ها شد در حالی که ساختار اصلی گرانول ها حفظ شد. حلالیت در آب نشاسته افزایش یافت ولی افزایش زمان فرایند اثر منفی بر حلالیت در آب نمونه ها داشت. الگوی پراش اشعه ایکس نمونه‌ها بدون تغییر باقی ماند اما درصد کریستاله بودن به طور معنی‌داری افزایش داشت. در اثر اصلاح نشاسته دمای ژلاتینه شدن افزایش ولی آنتالپی ژلاتینه شدن کاهش یافت. بافت ژل سفت‌تر، پیوستگی و برگشت‌پذیری آن بعد از اصلاح نشاسته کمتر شد. افزایش زمان فرایند کلیه این ویژگی‌ها را تشدید کرد.