Journal of Food Science and Technology (Iran)

Homepage:www.fsct.modares.ir

Scientific Research

Investigating the physicochemical properties of pregelatinized starch produced from acorn starch (Zagors) by using single drum drying

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The purpose of this research is to investigate the effect of the changes in the starch slurry with the concentration of 30, 50 and 70% of dry substance of pregelatinized acorn starch using a single drum dryer at a drum temperature of 120°C and a speed of 20 rpm which is called PGS30, PGS50 and PGS70 in order on physical and chemical properties including viscosity, hydration, freezing and melting stability, degree of gelatinization, morphology and FTIR .The rheological properties were evaluated using a rapid viscosity analyzer as a function of temperature. Pregelatinized starches showed viscosity at 25°C in cold water, but native acorn starch did not show viscosity at room temperature. Native acorn starch gradually started to absorb water as the temperature increased. So that the highest viscosity (peak) was created by native acorn starch, PGS30, PGS50 and PGS70 respectively. Native acorn starch granules have a smooth surface and non -homogeneous shape (mostly oval and spherical) and also have surface cavities and wrinkles. The morphology of pregelatinized starches changed significantly so that the granules of PGS50 and PGS70 samples are continuous and porous with a honeycomb-like structure. And there was no significant change in freezingthaw stability compared to native acorn starch $(p<0.05)$. The spectroscopic evaluation of native acorn starch was consistent with PGS samples up to the spectral range of 13343 cm. But in the PGS70 and PGS50 samples, several spectral intervals occurred between 3747 and 3945, which indicates the complete gelatinization of these two samples. Pregelatinized starches also have more swelling power, water absorption and solubility than native acorn starch $(p \ge 0.05)$.

ABSTRACT ARTICLE INFO

Article History:

Received 2022/ 10/ 15 Accepted 2023/ 0 1/ 01

Keywords:

Acorn, Native acorn starch, Pregelatinized starch, Single drum dryer, Degree of gelatinization .

DOI : 10.22034/FSCT.19.133.115 DOR : 20.1001.1.20088787.1401.19.133.10.5

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1. Introduction

The forests of the western and northwestern regions of Iran are the main habitat of oak trees.*QuercusPersica*), the country that includes more than 49% of the country's forests. Oak trees are the dominant tree species in the forests of Kohgiluyeh and Boyer Ahmad, Kermanshah and Lorestan provinces [1]. Oak fruit contains 5% protein, 7% fat, 70 -80% hydrocarbon [2, 3]. The fruit of oak trees is one of the sources of starch that very little research has been done on its structural and functional characteristics [4]. Starch is the most important source of stored energy in plants, which is abundantly found in cereal grains (such as wheat, corn, and rice) and in tuberous plants (such as potatoes). From the nutritional point of view, starch provides 70 -80% of the daily energy required by humans, and it is especially important in terms of providing the required calories in third world countries. Starch also has many uses and applications in the food, pharmaceutical, chemical, and paper, paint, and fabric industries [5 -7]. However, limitations such as low cutting and thermal resistance, thermal decomposition and high tendency to retrograde have limited its applications in the modern food industry [8].

Starch modification, which involves changing the physicochemical properties of starch to improve its functional properties, can be used to adapt starch to specific applications. Starch modification is generally achieved through chemical, physical, enzymatic and genetic methods or a combination [9]. Modification of starch can lead to changes in its properties and thus facilitate their use in various industries [10]. Among these methods, physical methods have attracted more attention because these methods are free of chemicals and compared to others. The methods are easier [9]. Pregelatinized starch is produced in three ways by roller, spray and extrude dryers [11 -13]. Depending on the method, conditions and source of starch, starch The pre -gelatine produced will have different characteristics [12, 14]. The extrusion process, which is widely used in the food industry today, is used in the production of breakfast cereals, snacks, ready -to -eat foods, diet foods, and animal feed [15, 16].

Pre -gelatinized starch is a physically modified starch that has the ability to absorb water and swell in cold water to increase viscosity and also achieve desired properties of dough and thickness [17]. Among the various available methods for the production of pregelatinized starch, the roller dryer is the most economical and the simplest available method compared to other methods [18]. Compared with the roller dryer and extrusion technology, the spray dryer is a more suitable method for the production of pregelatinized starch, in which the pregelatinized starch can be quickly converted into powder materials [19]. Previous studies have shown that the starch dried by the dryer Spraying shows the ability to flow and swell [20]. Pregelatinized starch can be divided into two groups: completely gelatinized starch and relatively gelatinized starch. Completely gelatinized starch is used in pharmaceutical formulations and as main components, bulking agents or thickening agents of many food and non -food products. Relatively gelatinized starches show a combination of the inherent characteristics of both natural and fully gelatinized starch [21, 22]. In many cases, the use of pregelatinized starch instead of natural starch allows for a simple production process and be shortened[19]. In addition, spray dryers gelatinize starch grains uniformly and produce them with minimal cutting and damage [23]. The spray drying process can disrupt the starch structure and produce an amorphous starch and severely affect the granule disintegration [24]. The objectives of this research were to use oak fruit starch to produce pregelatinized starch using a single roller dryer and to investigate the physicochemical properties of the resulting pregelatinized starch.

2- Materials and methods

acorn fruit*QuercusPersica*), was purchased from Kohgiluyeh and Boyer Ahmad provinces, and its starch was extracted by the acid method [25]. To produce pregelatinized starch, a single roller dryer model 1530 of Dongtal company, made in China, was used.

2-1- Production of pregelatinized starch by single roller dryer method First, starch slurry with concentrations of 30, 50, and 70 percent, dry matter was prepared and named as PGS30, PGS50, and PGS70 respectively. In order to pregelatinize starch, a single roller dryer was used with a temperature of 120 degrees Celsius and a speed of 20 rpm. For this purpose, starch slurry was poured on the surface of the roller. So that after gelling, a thin layer of gelled starch was formed on the main drum, which was scraped from the drum while rotating with a blade and packed with 120 micron mesh after passing through the mill, then the relevant tests were performed [12].

2-2- **Tests** performed on **gelatinized starch**

2-2-1- Viscosity

Viscosity measurement with rapid viscosity analyzer 1 It was investigated as a function of temperature. To measure viscosity, 3.5 grams of each sample was kept at 25°C for 2 minutes. The temperature increased from 14°C to 95°C, which was kept at 95°C for 3 minutes, and finally the samples were cooled to 25°C for 5 minutes. Various parameters including peak viscosity², storage viscosity³, final viscosity⁴, drop viscosity⁵ And the viscosity returned⁶ was measured [26].

2 - 2 -2 Measurement of water absorption index (WAI) *<u>x* solubility index</u> **(WSI) ⁸and swelling power (SP) 9**

2.5 grams of pregelatinized starch (W0) and 30 grams of distilled water were poured into the test tube, then placed in a hot water bath at a temperature of 90 degrees Celsius for 10 minutes and the sample was cooled to room temperature. Then the test tube was The intensity of 4000 relative centrifugal force for 10 minutes (Centrifuge model Hermle, Z306 and made in Germany). The supernatant liquid was poured into a container whose weight was previously determined and placed in the oven at 110°C for one day and night. After the end of the time, the container is weighed again and the difference

between the empty container and the full container after the oven is called (Wds), and the sediment remaining in the test tube is reported as the weight and number obtained (Wss)[27].

 $WAI = (g / g) = Wss / W0Equation (1)$

WSI = (g / 100 g) = Wds / W0 *100*equation (2)*

 $SP = (g / g) = Wss / (W0 - Wds)$ *Equation (3)*

2 - 2 - 3 - freeze -thaw stability:

Starch suspension (6% weight-volume) was heated in a hot water bath at 95 degrees Celsius for 15 minutes. Then it was cooled to 50°C in a water and ice bath and kept at this temperature for 15 minutes. The starch paste was poured into a test tube with a lid, then it was kept at -18°C and 4°C for 24 hours. Then, it was centrifuged with a device (Hermle model centrifuge, Z306, made in Germany) at a speed of 8000 centrifugal force for 10 minutes and the percentage of water was measured [28].

2-2-4-degree of gelatinization (DG)¹⁰

50 grams of starch was mixed with 50 ml of 0.05 M potassium hydroxide and the above suspension was centrifuged with 4000 relative centrifugal force for 10 minutes (Hermle Z306 centrifuge, made in Germany). The amount of 1 ml of the supernatant was mixed with 1 ml of 0.05 M hydrochloric acid and brought to 10 ml with distilled water, then the amount of 0.1 ml of iodide reagent (1 g of iodine and 4 g of potassium iodide with distilled water) (the volume reached 100 ml) was added to the above mixture. After mixing, the absorbance at 600 nm was reported with a device (spectrophotometer model Shimadzu, UV -2600 made in Japan) [29]. Where A1 is the absorbance of the test group at 600 nm and A2 is the absorbance of the control group. *Equation (4)*

Degree of Gelatinization $(DG) = A1 / A2$ **2 - 2 -5 Electron Microscope (SEM)¹¹**

The surface of pregelatinized starch powder was examined and observed using (scanning electron microscope model JSM -6380LV, made in Tokyo, Japan). The sample was fixed with conductive

¹. Rapid Visco Analyzer

². Peak Viscosity

³. Trough Viscosity

⁴. Final Viscosity

⁵ . Breakdown

⁶ . Setback

⁷ . Water absorption index

⁸. water solubility index

⁹. swelling power

¹⁰. Degree of Gelatinization

¹¹. Scanning electron micrograph

silver glue and then coated with a layer of gold. and 1000 magnification should be used [28].

2-3-6 structural examination of starch by spectrometry(FTIR)**¹²**

The infrared spectra of natural starch and oak pregelatinized starches were reported with a device (RXIPerkin/Elmer model, made in USA).

2-3-Statistical design

Experiments were performed in triplicate and their mean \pm standard deviation was reported. Analysis of variance (ANOVA) was used to prove the presence or absence of statistically significant differences between the means. Means were compared using Tukey's test and at 95% confidence level (p≤0.05). Statistical analyzes with Minitab¹³ Version 18 and charts were done with Excel program¹⁴ 2018 version were drawn.

3. Results and Discussion

3-1-Viscosity

According to the results obtained in Table 1, natural oak starch does not show viscosity at 25°C, but pre -gelatinized starches have viscosity in cold water using a single roller dryer. PGS starches (pergelatinized starch) can have viscosity in cold water because some of them are gelatinized. However, natural oak starch cannot absorb water at low temperature. As the temperature increases, it gradually starts absorbing water, so that the highest viscosity (peak) was created by natural oak starch PGS30, PGS50 and PGS70. Bemiller et al. (2011) reported that the peak viscosity is considered to be the balance point between the degree of swelling and disruption of starch granules. The higher the gelatinization of pregelatinized starches, the less the remaining starch granules

and the degree of swelling, which results in It leads to a decrease in viscosity. As a result of shear stress, starch molecules are destroyed and reduce the viscosity [31]. Also, Bemiller et al. (2011) reported that small starch molecules absorb less water and swell less than larger molecules, resulting in a lower peak viscosity [31]. Drop viscosity is calculated from the difference between peak viscosity and holding viscosity. which is sometimes used to describe the stability of starch gel [31]. The drop viscosity of natural starch was reported to be 1515 centipoise, while the drop viscosity of pregelatinized sample PGS30=906, PGS50=1051 and PGS70=709 decreased significantly $(p\geq 0.05)$. Since the drop viscosity reflects the stability of the dough, the lower the amount of dry matter of the starch slurry to produce pre gelatinized starch, the stability of the starch dough will be higher. The changes in storage viscosity are due to the fact that the size of the degraded molecules is smaller and has a larger contact surface, which results in a decrease in viscosity when kept at a temperature of 95 degrees Celsius [31]. During the cooling process, the organization between starch molecules, especially amylose, traps water between starch chains, resulting in gel formation and increased viscosity, which is defined as final viscosity. The difference between the storage viscosity and the final viscosity is defined as the return viscosity. As shown in Table 1, the return viscosity of the produced pregel starches decreased significantly $(p\geq 0.05)$. Satarapayu et al. (2007) have investigated the degraded molecules and the porous structure leading to the ability to retain water, which prevents the rearrangement of amylose and thus delays retrogression [32].

Mean \pm standard deviation (n=3); Different capital superscripts within the same column indicate significant difference (P<0.05).

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¹². Fourier transform infrared

¹³. Minitab

¹⁴. Excel

NAS: Native Acorn Starch; PGS30: pregelatinized starch milk suspension (30 % w/v); PGS50: pregelatinized starch milk suspension (50 % w/v); PGS70: pregelatinized starch milk suspension (70 % w/v).

3 - 2 - Swelling power, water absorption index and solubility index in water

Another feature of PGS starch is its swelling power, solubility index and water absorption. The results of which are shown in Figure 1. The swelling power is between 26.11 and 36.66% g/g. Compared to natural oak starch, it has a high swelling power. Swelling power is an important parameter of starch hydration properties, which is significantly affected by the interaction between amorphous and crystalline starch chains. During gelatinization, the crystalline structure of starch is disrupted due to the breaking of inter and intramolecular hydrogen bonds, which leads to a change in the ability of starch to bind water. Dry pregelatinized starch has significantly higher swelling power and solubility in cold water than natural starch. This may be because the pregelatinization process leads to the weakening of intramolecular hydrogen bonds and the reduction of interactions between amylose and amylopectin molecules and between amylopectin chains. The weakening of bonds is associated with the formation of structures with less stability and the increase in the leakage of amylose molecules, which leads to the formation of structures with more stability. The water absorption and solubility indices were significantly compared to natural starch, and this can be attributed to the destruction of starch grains, the reduction of crystallinity and the destruction of starch molecules during pre gelatinization, and the porous structure. PGS starch can absorb more water compared to natural starch, and the more small molecules in starch, the more water absorption increases. Also, Sanchi et al. (2001) reported the dissolution rate and swelling power in wheat, corn and rice starch. gelatinized has increased [33]. Tokaman and colleagues (2007) have investigated that during the spray drying process, heat can induce partial gelatinization on the surface of starch granules, which leads to the formation of solid bridges. These solid bridges help starch particles adhere to form granular particles. which can absorb more water and increase the swelling power [34]. The solubility index is an index that shows the change

of starch and molecular decomposition, which is used to measure the amount of soluble components released by starch after the extrusion process [35]. The solubility index of extruded starch was significantly higher than natural starch [29, 36, 37].

3 - 3 -Freeze -thaw stability

Changes in the stability of natural oak starch and pregelatinized starches in refrigeration and freezing conditions are shown in Table 2. The freezing and refrigeration stability of starch has been evaluated by measuring the amount of liquid separated from the gel after centrifugation. Based on the obtained results, no significant difference is observed ($p<0.05$). So that the hydration of the samples shows that the starch starts to swell and gelatinize when the distance between the amylose chains is broken and the pre -gelatin bond increases. Therefore, this may be due to an increase in the amount of bonding forces in the amorphous regions of the granule.

According to the studies conducted by Pachecoa et al. (2014), starches with high amylose such as potato (31 -20.1%), corn (32.5 -22.4%), taro (28 - 9.7%) 29.) and cassava (23.6 –18.6%), which have high amylose, waterlogging occurs due to

the large volume of water output during the retrogradation process [38]. White et al. (1989) have studied the freeze -thaw process. These authors showed that waxy corn and regular corn starch samples recovered 58% and 59% of initial gelatinized enthalpy, respectively [39].

Table 2 Syneresis at refrigeration and freezing condition (%), Degree of gelatinization (%) ofNSA, PGS30, PGS50 and PGS70

Mean \pm standard deviation (n=3); Different capital superscripts within the same column indicate significant difference (P<0.05).

NAS: Native Acorn Starch; PGS30: pregelatinized starch milk suspension (30% w/v); PGS50: pregelatinized starch milk suspension (50% w/v)7; PGS70: pregelatinized starch milk suspension (70% w/v).

Woodruff et al. (1938) investigated the microstructural change produced by freezing starch paste and were among the first to conclude that freezing causes retrogradation. Degradation often increases when starch gel (starch dough) is exposed to freezing and thawing treatments [40].

3-4 degrees of gelatinization

The degree of gelatinization in natural starch and pregelatinized starch samples is presented in Table 2. The degree of gelatinization in natural starch increased by 8.35% and pre -gelatinized starches PGS30=64.16, PGS50=83.86 and PGS70=97.66, so that the PGS70 sample shows that the gelatinization process is almost done and the effect had a significant effect on the degree of gelatinization of pre -gelatinized starch. When the percentage of dry matter of starch slurry to produce pre -gelatinized starch is lower, the starch grains absorb water better and swell faster, and as a result, it turns into gel more easily, which leads to an increase in the degree Gelatin becomes raw material with water content.

Gunner et al. (1984) reported that the degree of gelatinization of corn starch decreased with the increase in humidity [41]. According to the research of Rus et al. (2015), hydrogen bonds between amylose and amylopectin molecules make it harder for water to penetrate starch granules [42]. The research of Ituriaya and colleagues (2004) showed that as the gelatinization temperature increases, starch granules will swell more during cooking [43]. The degree of gelatinization of starch in bread is

usually reported as 96%. Studies showed that the degree of gelatinization of starch in the center of bread is higher than its crust [44]. According to the research of Salehifar et al. (2009) reported that the minimum amount of moisture for starch gelatinization is 32% and less than that, gelatinization does not take place, and with the increase of humidity, the amount of gelatinization increases so that if the ratio of water to starch is 2:1 The starting temperature of gelatinization is considered to be 57 $\mathrm{^{\circ}C}$ [45].

3-5-morphology

The morphology of pregelatinized starch and raw starch samples is shown in Figure 2. The shape and size of oak starch granules can be related to oak species, environmental conditions, oak fruit growth stages and starch isolation methods [46 - 49]. The raw oak starch granules have a smooth surface and non -homogeneous shape (mainly oval and spherical) and also have surface cavities and wrinkles. The structure of pregelatinized starch granules changed significantly so that the PGS30 sample was irregularly shaped and with several The cavities and samples of PGS50 and PGS70 are similar to each other due to less dry matter and more water content after drying the granules in a continuous and porous structure with a honeycomb -like structure. The absence of healthy starch grains in PGS samples indicates It gives complete gelatinization of all three samples during heating and drying on a single roller dryer. Similar findings have been reported on other PGS cereal starches [13, 50].

Fig 2 Scanning electron micrographs of native starch acorn, PGS30, PGS50 AND PGS70. NAS: Native Acorn Starch; PGS30: pregelatinized starch milk suspension (30 % w/v); PGS50: pregelatinized starch milk suspension (50 % w/v); PGS70: pregelatinized starch milk suspension (70 % w/v).

According to the research of Alameel et al. (2005), the temperature of the spray dryer also affects the morphology of starch particles. When spray drying is carried out at a lower temperature, there is more shrinkage and the average particle size is often lower than that obtained at a higher temperature [51]. Some studies have shown that the high temperature of roller dryers leads to the decomposition of starch, the breaking of intermolecular hydrogen bonds, and complete or partial gelatinization [52, 53]. Nakorno et al. (2009) reported that the degree of damage of pregelatinized rice starch granules by a double roller dryer decreases with the increase of amylose [54]. and at high temperatures, water is absorbed into the granules. Then the grains expand and break down, which leads to the complete gelatinization of starch and the formation of uniform gel materials, and after extrusion, due to pressure reduction and water evaporation, it expands in the form of a porous honeycomb structure [55].

3 - 6 - Structural investigation of starch by spectrometry (FTIR)

The comparison of vibrational frequency of chemical bond spectra of natural starch and oak PGS starches is shown in Figure 3. The profiles are similar, but the magnitude of the sample curves was different and numerous. The difference in these peaks is related to changes in the amount of amylose and amylopectin and other chemical compounds [30]. The spectra of the samples showed that the peaks confirm the polysaccharide nature of starch. Mostly the peaks are approximately cm^{-1} 760, 928, 1010, 1156, 1360, 1645, 2921, 3424, and 3335, which are consistent with the findings, but in PGS70 and PGS50 samples, several spectral intervals occurred in the range of 3747 to 3945, which indicates the complete gelatinization of these two samples. These peaks are related to various bonds such as C-OH, C-C, C-H, O-H, etc.

Fig 3 Scanning electron micrographs of native starch acorn, PGS30, PGS50 AND PGS70. NAS: Native Acorn Starch; PGS30: pregelatinized starch milk suspension (30 % w/v); PGS50: pregelatinized starch milk suspension (50 % w/v); PGS70: pregelatinized starch milk suspension (70 % w/v).

4 - General conclusion

Starch is the most abundant organic compound in nature, however, the protected structure of natural starches limits its properties and applications. Therefore, it is often necessary to modify them to obtain specific properties. As a green and safe physical modification method, gelatinization can improve the cold water solubility and swelling power and water absorption of starch and expand the application range of starch. The pregelatinized starch produced by single -roll dryer shows serious structural degradation, which increases cold water solubility and cold dough viscosity, but shows a relatively low hot dough viscosity, which indicates the improvement and stability of the gel and Retrogression properties compared to natural starch In summary, pregelatinized starch can significantly change the structure and functional properties of starch. which has a significant impact on the quality of the dough and the final product. Due to the superiority of pregelatinized starch compared to natural starch, today this starch can be used in

baby food, cold and instant food, cold sauces and cold desserts.

5- Resources

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پژوهشی مقاله علمی_

بررسی خواص فیزیکوشیمیایی نشاسته پری ژالتینه تولید شده از نشاسته بلوط)زاگرس(با استفاده از خشک

کن تک غلطکی

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