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Functional Properties of Some Non-conventional Treated Starches

Monica R. Nemţanu, Mirela Braşoveanu *National Institute for Laser, Plasma and Radiation Physics Romania*

1. Introduction

Starch is one of the most widespread and used biopolymers in different applications such as in the paper, textile, food, pharmaceutical and cosmetics industries (Richardson & Gorton, 2003). Even starch is useful in various industrial fields, its use as a native form has been restricted by some limitations (high viscosity, low solubility in cold water, paste instability, etc.) in specific applications due to its structure. Taking into account this aspect, starch is frequently submitted to the chemical, physical and enzymatic treatments or their combination in order to modify it and to obtain some functional properties suitable in specific industrial applications. The most used way to obtain modified starch is by chemical methods which are most of the time complex, expensive and time consuming.

Modern, non-conventional methods of starch modification are generally physical techniques (e.g., ionizing and non-ionizing radiation treatments, plasma treatment) which are fast, low cost and environmentally friendly because they do not use pollutant agents, do not allow the penetration of some toxic substances in the treated products and do not generate undesirable residual products, do not require catalysts and laborious preparation of sample. There are reported several studies related to starch treatment with ionizing radiation, especially with gamma radiation (MacArthur & D'Appolonia, 1984; Sokhey et al., 1993; Kang et al., 1999; Wu et al., 2002; Ezekiel et al., 2007). Recently, information regarding starch treatment with electron beam (e-beam) in different irradiation dose range, either at low or relative high doses (0 – 25 kGy) (De Kerf et al., 2001; Pimpa et al., 2007) or at very high doses (> 50 kGy) (Kamal et al., 2007; Shishonok et al., 2007) were reported. The studies concerning the effects of ionizing radiation were performed on starches extracted from various vegetal sources such as corn (Kang et al., 1999; De Kerf et al., 2001; Adeil Pietranera & Narvaiz, 2001), wheat (MacArthur & D'Appolonia, 1984; Koksel et al., 1996), potato (Ezekiel et al., 2007; Shishonok et al., 2007), barley endosperms (Faust & Massey Jr., 1966), rice (Wu et al., 2002; Bao et al., 2005) or sago (Pimpa et al., 2007). The reported data showed that ionizing radiation treatment generates free radicals on starch molecules that can alter their size and structure (Raffi et al., 1980; Ciesla et al., 1991; Grant & D' Appolonia, 1991; Sabularse et al., 1991; Sokhey et. al. 1993). Therefore, the hydrolyze of the chemical bonds occurs by cleavage of starch macromolecule into lower fragments of dextrin, which can be either electrically charged or uncharged as free radicals (Kang et al., 1999). This type of treatment may change the functional and physicochemical properties of starch leading to the increase of solubility (Bao & Corke, 2002), the reduction of swelling power (MacArthur & D'Appolonia, 1984) as

well as the reduction of relative viscosity of starch paste (Adeil Pietranera & Narvaiz, 2001) as a function of the irradiation dose. The use of radioisotopic sources (⁶⁰Co or ¹³⁷Cs) handling sources with continuous emission of radiation involves an additional risk of radiological accident compared to the usage of charged particle accelerators which send out radiation only during the technological process and, nowadays, are preferred for applications because they do not provide any risk of radioactive contamination. This is the reason explaining the late interest to study the influence of accelerated electrons on starch characteristics.

Also, investigations on starch modification by treatment with microwave or plasma were reported lately. Microwave treatment on starch were carried out especially on samples with high water content, over 20%, and less on samples with low $(1 - 5%)$ or limited $(7 - 15%)$ moisture content. The results showed that microwaves may determine the rearrangement of intermolecular structure indicating that the starch granule structure is disintegrated and, consequently, they lead to slight reduction of the water absorption ability, of solubility and swelling power as well as changes of the gelatinization temperature, syneresis or paste viscosity (Lewandowicz et al., 1997; Lewandowicz et al., 2000; Gonzalez & Perez, 2002; Gonzalez Parada & Perez Sira, 2003; Szepes et al., 2005; Luo et al., 2006; Lares & Perez, 2006) strongly correlated with experimental treatment parameters. Starch exposed to electrical discharges (plasma) may suffer either crosslinking phenomenon (Zou et al., 2004, Nemtanu & Minea, 2006) or depolymerization to dextrins with different molecular weights (Lii et al., 2002a; Lii et al., 2002b) depending on the experimental conditions (atmosphere, pressure, time of exposure, etc.).

The goal of our work is to present the experimental results concerning the comparison of physicochemical characteristics of starches from different botanical sources subjected to the accelerated electron beam treatment.

2. Experimental

Native starches from different botanical sources as cereals (corn, wheat and rice) and tubers (potato) were used in the experiments. The packed starch samples were irradiated with accelerated electron beam using a linear accelerator with medium energy of 6 MeV (INFLPR, Bucharest-Magurele, Romania), at ambient temperature and normal pressure. The irradiation doses between 10 and 50 kGy were checked using cellulose triacetate film.

pH was measured for 1% aqueous solutions of starch at 25°C with a Denver pHconductivity meter (Denver Instruments, USA).

Starch was dispersed in water at temperature of 85°C by stirring for 30 min and the mixture was centrifuged (2000 x *g*, 15 min), and then the supernatant was collected. The supernatant was evaporated at 110°C and weighed. The solubility was the ratio in weight of the dried supernatant to the initial weight of the starch. The sediment was weighed and the swelling power (SP) was calculated according Wattanachant et al. (2002):

$$
SP = \frac{w_s}{w_i (100 - s\%)} \cdot 100\tag{1}
$$

where: w_s – weight of sediment [g],

 w_i – weight of initial sample [g], s% - solubility of sample [%].

The paste clarity was determined according to method described by Craig et al. (1973) slightly modified. The clarity was measured on starch aqueous pastes of 1% concentration and expressed as a transmittance $(T%)$ at 620 nm. The measurements were carried out against water blank in a Cary 100 Bio spectrophotometer (Varian, Inc., USA) and were repeated after 24, 48 and 72 hours for the same samples stored at room temperature (25 °C) to evaluate the stability of the clarity pastes.

The colorimetric characteristics of 1% starch aqueous pastes were measured with Cary Bio 100 spectrophotometer (Varian, Inc., USA) using transmittance in spectrum visible region (360 – 830 nm) with standard illuminant D65, and expressed as color attributes of CIELAB and CIELCH systems. The data have been analyzed using the Color Application of the Cary Win UV v. 3.10 software.

Gel consistency determination was carried out according Tang method cited by Wu et al. (2002). Gel consistency was measured for starch pastes of 5% in 0.2N KOH solution at 25°C as the length of the gel in a test tube held horizontally for 1 hour.

The rheological measurements were performed on 5% aqueous starch suspensions using a rotational HAAKE VT® 550 viscometer (ThermoHaake, Germany) with NV coaxial cylinder. The shear stress, τ and apparent viscosity, η_a of the samples were measured at different temperatures for different shear rates, $\dot{\gamma}$. The obtained data were analyzed with RheoWin v. 3.5. software.

The calorimetric investigation was realized on mixture of starch with 70-80% water using a dynamic scanning calorimeter (PerkinElmer, Inc., USA). The measurements were carried out at temperature within 20 – 90°C in nitrogen atmosphere. The samples were heated in sealed aluminum pans at a heating rate of 5 K·min-1 .

The samples for GPC analysis were prepared according to the method described by Han & Lim (2004) slightly modified. Thus, starch (10 mg) was wetted with ethanol (20 μl), and then dispersed in 1M NaOH (500 μl). The mixture is then magnetically stirred (1000 rpm) for 10 minutes at 35°C. The starch solution obtained was diluted to 50mM NaOH with water and gently stirred again (150 rpm) for 30 minutes at room temperature, followed by centrifugation (3500 x *g*, 10 min). GPC analysis was performed using a Breeze system (Waters, USA) which consists in 1525 binary pump, autosampler 717+, 2414 differential refractive index detector, in-line degasser AF and temperature modul system. The calibration was carried out with pullulan standards for two Ultrahydragel columns (Waters, USA). The mobile phase used for GPC was 50mM NaOH and the flow rate was 0.5 ml/min. The obtained data were analyzed with Breeze v. 3.30 SPA software.

The experimental data statistic interpretation was performed using factor analysis of principal components with StatistiXL v.1.8 (2007) software in order to correlate the functional properties of studied starches.

3. Results and discussion

Free radicals are produced when polymers are exposed to ionizing radiation treatment (Woods & Pikaev, 1994). These active species are responsible for initiating chemical reactions leading to modifications like (1) formation of network in polymer chains called crosslinking, (2) molecular weight reduction by chain scission and (3) polymerizing a monomer and grafted onto the base polymer chain called grafting (Sarma, 2005). The effects of radiation depend on radiation type and irradiation dose as well as on chemical structure of polymer, thus the

evaluation of physicochemical properties of starches treated with electron beam represents an important stage in order to elaborate such modification method.

3.1 pH evaluation

pH is an important property in starch industrial applications being used generally to indicate the acidic or alkaline properties of liquid media. A variation of pH value can significantly modify the evolution of other characteristics of starch after electron beam treatment.

Aqueous solutions of native studied starches had relative neutral pH that decreased by electron beam treatment with irradiation dose (Table 1) so that for a doses of 50 kGy, the solutions had an acidic character (pH = 4.7 – 4.8) for corn (Nemtanu et al., 2006a, Nemţanu, et al., 2006b), wheat and potato starches. In the case of rice starch, it was observed just a slight change of pH value as a consequence of e-beam treatment (Table 1).

Table 1. pH values of the studied starch aqueous solutions

The decrease of solution pH value corresponds to the increase of its hydrogen ion concentration. The descending change of solution pH after e-beam treatment of starch – similar results on gamma-irradiated dry bean starch (Duarte & Rupnow, 1994) or e-beam treated sago starch (Pimpa et al., 2007) being previously reported by other research groups – indicates the formation of chemical groups with acidic character such as carboxyl, carbonyl or peroxide groups. This behavior is sustained also due to the fact that e-beam treatment was carried out in presence of oxygen determining thus the appearance of free radicals, compounds with carbonyl bonds (aldehydes/ketones), organic peroxides or other polysaccharide degradation products (Ershov, 1998) that can lead to the increase of starch acidity.

3.2 Solubility and swelling power

Solubility and swelling power are properties which provide the evidence of the magnitude of interaction among starch chains within the amorphous and crystalline domains, the extent of this interaction being influenced by amylose/amylopectin ratio, and by the characteristics of amylose and amylopectin in terms of molecular weight/distribution, degree and length of branching, and conformation (Hoover, 2001; Ratnayake et al., 2002).

The changes that appear in starch granule in an aqueous system depend on temperature and water availability from system. When starch molecules are heated in excess of water, the crystalline structure is disrupted and the water molecules become linked by hydrogen bonding to the exposed hydroxyl groups of amylose and amylopectin, which causes an increase in granule swelling and solubility (Singh et al., 2003).

The fact that the cereal starches (corn and wheat) exhibited generally lower solubility can be attributed to the more compact structure and different cristallinity than tuber starch (potato) according Collision (1968). The e-beam treatment led to the solubility value increase for all studied starches (Table 2). It was remarked the significant value of the sample solubility reached after treatment as the irradiation dose increase. This notable increase of solubility demonstrates clearly that the starch molecules suffer important changes as consequence of a degradation phenomenon induced by irradiation.

Table 2. Solubility values [%] for studied starches

The improvement of starch solubility in water is an important request in the application where starch is involved, especially nowadays when modern technologies impose higher and higher quality standards and very simple and fast technological operations.

Swelling power is a measure of hydration ability of starch granule and it is determined by weighing the swelled starch and retained water. Such property is very important for certain starch application like those from food industry where the quality of starch-based products is strongly related to the capacity of starch granule to retain water and swell. A direct consequence of starch swelling is the increase of solubility, paste clarity and viscosity – properties of interest, especially in food industry.

The swelling power of native starch increases with the temperature increasing as a result of crystalline structure relaxation so that both amylose and amylopectin can form easily hydrogen bonds with water molecules.

Table 3. Swelling power values [%] for studied starches

The e-beam treatment of starch caused the reduction of swelling power as the increase of the irradiation dose (Table 3). This behavior could be attributed to the fact that starch granules become sensitive being weaker and easier to break after e-beam treatment. In addition, a consequence of starch degradation induced by irradiation can be also the inhibition of granule ability to trap water and provoke the swelling explaining thus the swelling power reduction by irradiation.

3.3 Visual characteristics

Material or product quality is strongly related to its visual aspect (form, color, and transparency/opacity) especially as this visual aspect, by conferred information, influences consumer's perception and so the acceptance or rejection of that material or product.

3.3.1 Clarity and its stability

Clarity is one of the most important attributes in the starch paste properties, particularly in food industry where it influences directly brightness and opacity of products, an important factor to determine the clarity of starch paste being the physical arrangement of molecules which contribute to swelling ability of granules (Craig et al., 1973).

Light transmittance offers information regarding the starch paste behavior when light goes through it, thus the higher transmittance value the paste is more transparent.

Native corn starch paste had a low clarity ($T_{620 \text{ nm}}$ < 10%) (Nemțanu et al., 2006b, Nemțanu et al., 2007) which increased spectacularly ($T_{620 \text{ nm}} \approx 85\%$) after 24 hour storage (Fig. 1a). The sample clarity changed insignificantly during further storage time.

After electron beam treatment, the clarity of corn starch paste, immediately after preparation (Nemţanu et al., 2006b), increased as the increase of the irradiation dose getting to a value of approximately 30%(Nemţanu et al., 2007) (Fig. 1a). However, for doses of 40 and 50 kGy, the clarity was slightly lower than the samples treated with doses up to 30 kGy. Such behavior appeared due to the modifications induced by electron beam treatment in starch structure, if we take into account de study reported by Craig et al. (1973). During storage time, the samples treated with 10 and 20 kGy showed similar evolution like native starch, meaning an increase of the clarity, but not so spectacularly as control sample, after 24 hours, value kept even after 72 hours of storage. Therefore, the clarity of native and 10 – 20 kGy treated starch pastes showed instability in the first 24 hours followed by its stabilization unlike the samples treated with doses of 30 – 50 kGy which stabilized before 24 hour storage time.

The value of native wheat starch clarity was higher than of corn starch being approximately 19% (Fig. 1b), but it showed similar evolution during storage time. However, it was remarked the increase of clarity only up to $~60\%$ after 24 hour storage time, namely lower than for corn starch value. The clarity of pastes prepared from wheat starch treated with electron beam at different doses varied insignificantly in comparison to native wheat starch immediately after preparation. The paste clarity of e-beam treated wheat starch increased during storage time in a similar way as paste of native wheat starch. Taking into account these observations, it can be considered the paste clarity of wheat starch treated with electron beam in range of 10 – 50 kGy was stable after 24 hour storage period.

The native rice starch had also an opaque paste ($T_{620 \text{ nm}} \approx 5\%$) (Nemtanu, 2005) and its clarity increased ($T_{620 \text{ nm}}$ ≈ 50%) after storage of 24 hours (Fig. 1c) without significant change even after 72 hours.

The e-beam treatment led to an increase of the rice starch paste clarity, immediately after preparation, as the irradiation dose increase (Nemtanu et al., 2005), so that the clarity was ~60% for irradiation dose of 50 kGy (Fig. 1c). The samples treated with doses in the range of 10 – 30 kGy showed similarly evolution of clarity increase as control sample. As a result of these findings, it can be certainly considered the rice starch samples treated with doses of 40 – 50 kGy had stable clarity of their pastes during storage of 72 hours while the clarity of samples treated with 10 – 30 kGy has been stabilized after 24 hours from their preparation.

Native potato starch paste showed very good clarity with $T_{620 \text{ nm}} \approx 90\%$ which decreased nevertheless as the storage time (Fig. 1d). The clarity of potato starch paste was improved as

a result of applying e-beam treatment on starch. The clarity decreased slowly after 24 hours and significantly after 72 hours for all samples. However, the clarity stabilization occurred after 24 hours for samples treated with doses of 40 – 50 kGy. Therefore, the potato starch samples treated with electron beam showed very clear pastes, the samples treated with doses of 40 – 50 kGy presenting even an obvious stability after 24 hour storage period.

Fig. 1. Clarity of starch pastes: (a) corn, (b) wheat, (c) rice and (d) potato, before and after ebeam irradiation

As it was mentioned for solubility and swelling power, different values of clarity and its stability of cereal starches in comparison to tuber starch can be due to the physical arrangement of molecules within granules, which obstructs their ability to swell and disperse in water. In addition, the starch paste clarity during storage can be influenced by the interaction of several factors such as granule swelling, granule remnants, leached amylose and amylopectin, amylose and amylopectin chain length, intra- or inter-bonding, lipid and cross-linking (Jacobson et al., 1997).

The decrease of the transmittance value, meaning the increase of the starch paste turbidity indicates the re-association of initial broken bonds in orderly structure (retrogradation) and it could be noticed by liquid phase separation and sediment.

The paste behavior of e-beam treated starches studied using clarity indicates that this treatment can lead to the improvement of this property as a consequence of starch molecule degradation and formation of lower molecular fragments. According to Song et al. (2006), the increase in the transmittance value of starch pastes might be attributed to the introduction of carboxyl group, which retained the water molecules to form hydrogen bonds in the starch granules, thereby, increasing the clarity of starch pastes. Taking into account that, the formation of carboxyl groups after e-beam treatment of starch was suggested by the increased acidity of the irradiated starch pastes, we can consider this also as a factor which contributes to the increase of the clarity. Moreover, the evolution in time of irradiated starch paste clarity showed that the retrogradation phenomenon (formation of an orderly structure) was delayed by treatment of starch with accelerated electron beam.

3.3.2 Colorimetric parameters

Color is a fundamental property or, in other words, one of the vital components of visual quality of any material or product playing major role for the quality evaluation and consumer's decisions. Moreover, color variations confirm the changes that appear in structure or composition of a material or product and may be used as a quality index. Color perception is three dimensional, meaning that three terms are needed to describe a

color (Marcus, 1999): hue, lightness, and chroma (Fig. 2).

The International Commission on Illumination (CIE - Commission Internationale de l'Eclairage in French) (Ohta & Robertson, 2005), the organization responsible for international recommendations for photometry and colorimetry, recommended standard colorimetric systems including illuminants, colorimetric observer, light sources and the methodology used to derive values for describing color. The CIE Color Systems utilize three coordinates to locate a color within colorimetric space. Nowadays, the most used colorimetric spaces are CIE 1976 or CIELAB ($L^*a^*b^*$) and CIELCH ($L^*C^*h^{\circ}$). When color is expressed in CIELAB (Fig. 3), *L** defines lightness, *a** denotes the red/green value and *b** the yellow/blue value being presented in Cartesian coordinates of color. Thus, *L** has values from 0 (black) to 100 (white); $a^* > 0 \rightarrow$ red color and $a^* < 0 \rightarrow$ green color; $b^* > 0 \rightarrow$ yellow color and b^* <0 \rightarrow blue color.

Fig. 3. CIELAB colorimetric system

CIELCH (D65/CIE 1964 100) is a polar representation of the CIELAB rectangular coordinate system and describes color in terms of lightness *L**, chroma (saturation) *C** and hue *h°*.

Investigated spectrocolorimetric parameters in our study showed different colorimetric behavior of cereal starches than tuber starch after application of electron beam treatment. In addition, the evolution of these parameters indicated clearly that changes occurred globally in the chemical structure of studied starches.

Therefore, it was noticed that yellow-blue and red-green coordinates modified after e-beam treatment of starches from perspective of L*a*b* color coordinates. The pastes of cereal starches exhibited the color movement in the *+a** direction depicted a shift toward red while *+b** movement indicated the shift toward yellow as the increase of the absorbed dose. But, the pastes of potato starch showed the color movement in the *-a** direction toward green and *-b** toward blue with irradiation dose increase.

Transposing the color in L*C*h° coordinates, it was observed that all native starch pastes showed very low values of chroma *C** (4 – 10) indicating the tendency of color to grey. The values of chroma *C**and hue *h°*for cereal starch pastes increased and decreased, respectively, exhibiting red-yellowish color after irradiation with electron beam (Fig. 4a-c). The pastes of the irradiated potato starch showed an opposite effect to that presented by cereal starches (Fig. 4d), meaning that the chroma decreased and the hue intensified indicating the movement of color to green.

Regarding the lightness, it intensified for all starches when the irradiation dose increased. It was remarked that the high lightness value ($L^* \approx 90\%$) of potato starch paste which tended to maximum by irradiation.

All these aspects related to the chromatic attributes of the investigated starch pastes confirmed the e-beam treatment influence on the starch macromolecular structure. The color change toward red-yellowish can be due to caramelization reaction of compounds with low molecular weight resulting from starch macromolecule degradation.

3.4 Gel consistency

The origin of rigid and weak gel properties is specific to molecular association of polymer chains (Dea, 1989). Cereal starches in their native form showed medium consistency (Nemţanu et al., 2006a; Nemţanu et al., 2006b; Yu & Wang, 2007) while the native form of potato starch was characterized by high consistency.

After treatment with accelerated electron beam, the gel consistency value increased with the increase of the irradiation dose indicating the reduction of this property (Table 4).

Fig. 4. CIEL*C*h parameters of starch pastes: (a) corn, (b) wheat, (c) rice and (d) potato, for ■ control sample and doses of • 10 kGy, \triangle 20 kGy, \blacktriangledown 30 kGy, \blacktriangle 40 kGy, and * 50 kGy

Irradiation dose	Starch source			
[kGy]	Corn	Wheat	Rice	Potato
	85 ± 2	88 ± 7	70 ± 7	45 ± 3
10	102 ± 9	108 ± 12	107 ± 10	83 ± 6
20	104 ± 10	112 ± 11	112 ± 9	87 ± 8
30	109 ± 4	116 ± 11	113 ± 9	89 ± 9
40	120 ± 7	128 ± 9	122 ± 8	95 ± 2
50	127 ± 10	132 ± 8	116 ± 12	101 ± 8

Table 4. Gel consistency values in mm, for studied starches

There are studies in literature reporting similar effect for gamma irradiated rice starch (Wu et al., 2002; Yu & Wang, 2007), and besides this, in their study, Yu & Wang (2007) found out that the increase of gel consistency is due to the reduction of the apparent amylose content.

3.5 Rheological properties

Rheological properties of a material are a consequence of the molecular interactions occurring within its molecular structure. During gelatinization, the starch granules swell to several times its initial size and, consequently, their constituents are leached out from inside granules, mainly amylose, leading to a three dimensional network (Steeneken, 1989; Tester & Morrison, 1990). These changes are responsible of rheological characteristics showed by starch suspensions during heating and shear rate. The rheological behavior of starch is led by several factors such as amylose content, granule size distribution, granule form or granule-granule interaction (Okechukwu & Rao, 1995; Kaur et al., 2002; Morikawa & Nishinari, 2002; Singh et al., 2003; Singh & Kaur, 2004). Joslyn (1970) claimed the viscosity is strongly related to molecular chain length and, on the other hand, to space arrangement of molecule determined by intermolecular bonds as well as hydrogen or van de Waals bonds.

The control samples of the studied starches showed non-Newtonian behavior for which the ratio of the shear stress and shear rate is not constant, the shear stress dependence of shear rate indicating the pseudoplastic character of the starch suspensions. Electron beam treatment of starches affected this kind of behavior and it was noticed the tendency toward Newtonian behavior as the irradiation dose increase.

Taking into consideration the experimental results which characterize gel consistency and behavior flow of starches, it was expected the potato starch to show high viscosity in comparison to cereal starches. The determination of the starch apparent viscosity at 250 C and 200 s -1 confirmed our expectations. Thus, native potato starch exhibited the highest apparent viscosity in the investigated starches while the cereal starches can be classified in the following order: rice starch > corn starch > wheat starch.

The apparent viscosity (25⁰ C, $\dot{\gamma}$ =200 s⁻¹) of the starch suspensions presented descending evolution with dose increasing (Fig. 5) for all investigated starches. Thus, the viscosity of corn starch suspension decreased significantly from 294.24±27.95 mPa·s to 16.35±0.82 mPa·s after irradiation with 30 kGy (Fig. 5a). This significant decrease of viscosity can be found again for the other starches. For instance, the wheat starch suspension with initial viscosity value of 136.94±11.92 mPa·s reached to the viscosity of 19.07±1.02 mPa·s (Fig. 5b) after 20 kGy irradiation, and the rice starch suspension with higher initial value of viscosity (321.53±20.15 mPa·s) got to de 16.67±1.53 mPa·s after irradiation with 40 kGy (Fig. 5c). The potato starch viscosity having a very high value of 692.79±34.64 mPa·s, by irradiation with 30 kGy, decreased dramatically to 16.82±1.42 mPa·s (Fig. 5d), value comparable with that of corn starch irradiated with 30 kGy, so indicating high radiosensitivity of potato starch.

Temperature is one of the main interesting variables in technological processes which can affect rheological parameters. Studies of molecular thermodynamics showed that temperature has a direct effect on molecular motion, thus as the temperature increases, molecular motion takes place at a faster rate and consequently viscosity also depends on temperature (Figura & Teixeira, 2007). Viscosity decreases with increasing temperature, and fluidity – the inverse quantity of viscosity – increases with increasing temperature because the degree of molecular motion is different at different temperatures.

The rheological analysis of the investigated starch pastes, at different temperatures, showed that the apparent viscosity ($\dot{\gamma}$ =200 s⁻¹) decreased with increasing temperature, as we expected, for all studied suspensions, both control and e-beam irradiated samples. The obtained results indicated that the dependence of rheological characteristics on temperature is insignificantly influenced by e-beam treatment.

Fig. 5. Variation of apparent viscosity of starch suspensions: (a) corn, (b) wheat, (c) rice and (d) potato, with irradiation dose

3.6 Gelatinization

Gelatinization is one of the most important properties of starch when it is heated in excess of water. Gelatinization phenomenon of starch is practically a phase transition associated with transformation of granule crystalline phase in amorphous one, which causes irreversible changes of different functional properties such as granule swelling power, solubility, lose of optical birefringence. Gelatinization occurs initially in the amorphous regions, as opposed to the crystalline regions, of the granule, because hydrogen bonding is weakened in these areas (Singh et al., 2003). Thus, the differences which can appear in transition temperatures for different kinds of starches may be attributed to the differences in the cristallinity degree. High transition temperatures have been reported to result from a high degree of cristallinity, which provides structural stability and makes the granule more resistant towards gelatinization (Barichello et al., 1990). Both gelatinization and swelling are properties

partially controlled by the molecular structure of amylopectin (unit chain length, extent of branching, molecular weight, and polydispersity), amylose to amylopectin ratio and granule architecture (crystalline to amorphous ratio) (Tester & Morrison, 1990).

Differential scanning calorimetry (DSC) is known as an extremely valuable tool to characterize and control the gelatinization phenomenon of starch (Stevens & Elton, 1971) because it provides a quantitative measurement of the enthalpy, Δ*H*, which means the energy required to take place the gelatinization process, and a determination of temperature range where gelatinization occurs as well.

DSC curves recorded for all starches showed an endotherm peak associated to gelatinization phenomenon and thus, based on it, the temperatures involved in this process and gelatinization enthalpy were determined. It was noticed that the temperatures and gelatinization enthalpy were different for each investigated starch and, as we mentioned previously, these differences are due to the different chemical composition, granule morphology and structure of starches.

It was expected that the possible radioinduced changes in starch structure and suggested by previous analyzed properties to lead also to changes of thermal characteristics of starches. The DSC studies reported (Ciesla & Eliasson, 2002; Ciesla & Eliasson, 2003) in the literature regarding the influence of gamma radiation on gelatinization properties of potato and wheat starches can be added here. The recorded DSC curves for samples treated with electron beam indicated that the height of the peak attributed to gelatinization decreased with dose increasing suggesting a decrease of the gelatinization enthalpy. Also, it was noticed the peak shift which indicates structural changes appeared within macromolecule.

Table 5 shows the temperatures and enthalpy characterizing the gelatinization process for all analyzed starches. We noticed that the e-beam treated starches presented a decrease of the peak gelatinization temperature (T_p) as irradiation dose increasing, excepting the potato starch where this temperature slightly increased. The onset gelatinization temperature (*To*) and conclusion temperature (*Tc*) of gelatinization process were affected also with the increase of the irradiation dose. Therefore, the onset temperature for cereal starches shifted to lower values simultaneously with the increase of temperature range of gelatinization. On the other hand, the onset temperature of potato starch increased while the temperature range narrowed as irradiation dose increasing.

The gelatinization enthalpy value decreased with the increase of the irradiation dose for all studied samples even the specific temperature range of gelatinization phenomenon modified according the starch source.

Most of the times, the enthalpy is directly associated to the granule cristallinity degree. Taking into account this aspect and the tabled values of gelatinization enthalpy, it might assume that electron beam treatment of analyzed starches led to the reduction of crystalline regions.

We reminded previously that the gelatinization begins in the amorphous regions and after that it affects crystalline regions. Therefore, one can consider the enthalpy of whole gelatinization process as a sum of the enthalpies attributed to amorphous region gelatinization, Δ*Hamorph*, and crystalline region, Δ*Hcrystalin*, respectively according the following relation (2):

$$
\Delta H = \Delta H_{amorph} + \Delta H_{crystalin} \tag{2}
$$

Consequently, in our opinion, the gelatinization enthalpy should be carefully used as a cristallinity index because it is practically the quantity required to the phenomena occurring

during gelatinization process like intermolecular bond disruption, granule hydration and swelling, and crystallite melting.

 T_o – onset temperature; T_p – peak temperature; T_c – conclusion temperature; *Tc* - *To* – temperature range of gelatinization; Δ*H* – enthalpy of gelatinization Table 5. Starch thermal characteristics observed from DSC curves

3.7 Molecular weight

Molecular weight is a characteristic of polysaccharides which influences most of their physicochemical properties. Polysaccharides contain generally chains of different constituent monosaccharides conferring certain molecular weight distribution. This distribution is variable depending on different factors like the biosynthesis or extraction conditions used to isolate the polysaccharide.

Number average molecular weight (*Mn*), weight average molecular weight (*Mw*), z-average molecular weight (*Mz*) were determined for studied samples in order to reveal the influence

of the e-beam treatment on molecular weights and distribution. Table 6 shows the molar mass distribution of the samples treated with electron beam in comparison with the control samples.

Irradiation dose	$M_n \times 10^{-4}$	$M_w \times 10^{-5}$	$M_z \times 10^{-5}$	PI		
[kGy]	[g/mol]	[g/mol]	[g/mol]			
	Corn starch					
$\boldsymbol{0}$	5.52 ± 0.45	3.07 ± 0.22	8.36 ± 0.31	5.56 ± 0.23		
10	4.19 ± 0.14	2.34 ± 0.02	6.43 ± 0.03	5.58 ± 0.12		
20	3.62 ± 0.37	1.78 ± 0.08	5.43 ± 0.02	4.92 ± 0.27		
30	3.42 ± 0.21	1.50 ± 0.03	4.75 ± 0.12	4.41 ± 0.37		
40	2.87 ± 0.03	1.16 ± 0.02	3.82 ± 0.10	4.04 ± 0.28		
50	2.84 ± 0.13	1.12 ± 0.05	3.48 ± 0.39	3.95 ± 0.34		
		Wheat starch				
$\boldsymbol{0}$	6.46 ± 0.27	3.43 ± 0.01	7.67 ± 0.01	5.31 ± 0.11		
10	3.53 ± 0.02	1.87 ± 0.01	5.87 ± 0.01	5.30 ± 0.04		
20	2.58 ± 0.24	1.33 ± 0.01	4.62 ± 0.18	5.20 ± 0.53		
30	2.46 ± 0.08	0.94 ± 0.05	3.13 ± 0.14	3.83 ± 0.09		
40	2.13 ± 0.20	0.79 ± 0.02	2.37 ± 0.05	3.69 ± 0.34		
50	2.31 ± 0.01	0.71 ± 0.07	1.79 ± 0.13	3.07 ± 0.27		
		Rice starch				
$\mathbf{0}$	7.10 ± 0.03	3.80 ± 0.04	9.14 ± 0.02	5.36 ± 0.10		
10	5.38 ± 0.15	2.62 ± 0.09	7.86 ± 0.20	4.86 ± 0.45		
20	4.74 ± 0.09	2.24 ± 0.19	6.69 ± 0.33	4.72 ± 0.40		
30	4.41 ± 0.43	2.04 ± 0.09	6.17 ± 0.02	4.64 ± 0.27		
40	4.31 ± 0.24	1.96 ± 0.03	5.90 ± 0.08	4.55 ± 0.19		
50	4.11 ± 0.02	1.86 ± 0.09	5.71 ± 0.25	4.53 ± 0.41		
	Potato starch					
$\boldsymbol{0}$	8.85 ± 0.04	4.41 ± 0.04	9.81 ± 0.06	4.98 ± 0.20		
10	6.72 ± 0.54	3.67 ± 0.23	9.38 ± 0.17	5.46 ± 0.45		
20	5.57 ± 0.44	2.98 ± 0.09	8.50 ± 0.28	5.35 ± 0.54		
30	4.16 ± 0.11	2.22 ± 0.01	7.60 ± 0.08	5.33 ± 0.13		
40	3.38 ± 0.15	1.83 ± 0.10	6.57 ± 0.07	5.40 ± 0.39		
50	3.20 ± 0.14	1.73 ± 0.05	6.01 ± 0.23	5.41 ± 0.50		

Table 6. Molecular weight distributions of starch samples

It was noticed the decrease of the molecular weights as the irradiation increase for each studied starch, indicating the degradation phenomenon of the macromolecules. The decreasing evolution of the molecular weights with the irradiation dose suggests the break of polymeric chain and formation of the fragments with different molecular weights, which modify the mass molecular distribution of each sample. Even *Mⁿ* and *Mw* decreased concomitantly, though their evolution was differently influenced by irradiation and it was reflected by polydispersity index as well. The polydispersity decreased by e-beam treatment of cereal starches showing the change of their molecular weight distribution so that the *M^w* decreased faster than *Mn* with the irradiation dose. For potato starch, it was observed a slight increase of the polydispersity after e-beam treatment showing that *Mn* decreased faster than *Mw*. The molecular fractions with low molecular weights appeared with a higher

percentage than the fractions with high molecular weight as a consequence of the e-beam treatment.

3.8 Factor analysis

The factor analysis was used in order to find a correlation between the most important properties of the irradiated starches in respect of anticipation of the radio-induced change degree. This statistical method is generally used to analyze large numbers of dependent variables to detect certain aspects of the independent variables (factors) affecting those dependent variables – without directly analyzing the independent variables.

The statistic processing of our experimental data pursued the interdependence relationship of physicochemical properties of starches, among them and with the irradiation dose, being considered as system variables. This analysis assumes the volume reduction (condensation) of the initial variables and a new set of independent variables named factors, with a minimal loss of information. The variables are modeled as linear combinations, plus "error" terms.

In the first analysis stage, taking into account the experimental data introduced in the statistic analysis software, the correlation matrix of those 15 variables (Table 7) was calculated so that to highlight the relationship relevance between every two variables.

Thus, for each studied starch, it was globally noticed that, for instance, the apparent viscosity is strongly positive correlated both with number average molecular weight (*r* = 0.869) and mass average molecular weight ($r = 0.857$), less correlated with pH ($r = 0.617$) and gelatinization enthalpy ($r = 0.684$), at the same time showing strongly negative correlation with irradiation dose $(r = -0.801)$ and reduced one with gel consistency $(r = -0.687)$. Also, properties like pH, swelling power, gelatinization enthalpy are positive correlated with number and mass average molecular weights, while they present negative correlation with the irradiation dose. Unlike these, solubility, paste clarity or other visual attributes present positive correlation with irradiation dose and negative correlation with number and mass average molecular weights. It was noticed as well the correlation among thermal properties and colorimetric aspects emphasized especially by strong positive correlation of gelatinization temperature and paste hue $(r = 0.802)$, while it presents strong negative correlation with chroma (*r* = - 0.827) and reduced one with clarity (*r* = - 0.441) and lightness $(r = -0.432)$ of pastes. The gelatinization temperature showed also quite good positive correlation with polidispersity, despite of its quite weak correlation with number and mass average molecular weights.

In the second stage of statistic analysis, based on the variable correlation matrix, it was considered that every analyzed variable represents a factor so that the table of individual values of factors was further generated (Table 8). The factor individual value, known as *eigenvalue,* represents the specific value of variable variance explained by factor and it is given by saturations square sum from this factor.

It was observed that first 3-4 factorial axis bring a high percent of explicative factors of variance, ~90%, while the contribution of the other axis is insignificant so that their omission leads to minor loss of information. To reduce the number of factors, *Kaiser's criterion* was considered. According to this principle the contribution of factors is significant when the eigenvalue is higher than 1. Therefore, the total number of factors (independent artificial variables) was simplified to four, covering ~90% of variance with the following proportions: Factor 1 with \sim 47%, Factor 2 with \sim 26%, and Factor 3 and 4 with \sim 21%.

Table 7. Correlation matrix of variables

	Factor	Eigenvalue	$\%$ of variance	$\%$ cumulative
	1	7.029	46.861	46.861
	$\overline{2}$	3.843	25.618	72.479
	3	2.080	13.864	86.343
	4	1.111	7.407	93.750
	5	0.423	2.823	96.573
	6	0.175	1.165	97.739
	7	0.164	1.093	98.832
	8	0.076	0.504	99.336
	9	0.042	0.280	99.616
	10	0.026	0.172	99.788
	11	0.015	0.100	99.889
	12	0.012	0.078	99.967
	13	0.003	0.021	99.988
	14	0.002	0.010	99.998
	15	0.000	0.002	100.000

Table 8. Explained variance (eigenvalues)

Extraction method: principal component analysis, 4 extracted components Table 9. Unrotated factor loadings

Next stage consisted in generation of matrix of factor loadings (Table 9) which indicate the correlation level existing between each variable and correspondent factor. For a better "outlook" of data and simplification of their interpretation, it was used the rotation method *varimax*, which minimize the number of variables with factor loadings. The matrix of factor loadings after rotation is showed in Table 10. The positive values of coefficients indicated an increase of variable contribution simultaneously with the coefficient increase, while their negative values indicated the variable contribution decrease with the coefficient increase. For instance, the coefficient positive values of apparent viscosity, swelling power, pH, gelatinization enthalpy and average molecular weights indicated the contribution increase of these variables to the increase of contribution decrease of these two variables to Factor 1 with the coefficient increase.

Extraction method: principal component analysis

Rotation method: Varimax with Kaiser normalization

Rotation completed in 7 iterations

Table 10. Rotated factor loadings

The casewise factor scores (Table 11) of those four factors extracted previously were obtained in the last stage of the statistic analysis. The score matrix showed the contribution of each factor to the explanation of each sample behavior.

Graphical representation of samples and attributes as against principal components allowed the synthetic correlation among those and the analysis of the sample similitude. All previous remarks can be found again in the mentioned plot. To exemplify, there is displayed the plot for the first two factors (Fig. 6).

Sample distribution in all quadrants showed their differentiation as a whole. Nevertheless, the corn and wheat starch samples treated with irradiation doses over 20 kGy are well grouped and therefore similar, while the rice starch samples are rather isolated from the other samples and distributed each in only one quadrant, being similar only between them.

The potato starch samples kept the group identity even when they were in the same quadrants with other samples.

c – corn starch, w - wheat starch, r – rice starch, p – potato starch

10….50 – irradiation dose [kGy]

Table 11. Casewise factor scores

4. Conclusion

The treatment of starch with accelerated electron beam can produce several structural and functional changes. For the studied starch types, these modifications have generally similar tendency, but different quantitatively, depending on the specificity of each starch.

Fig. 6. Factor score plot for Factor 1 and 2

The acidity and solubility increased with the irradiation dose, while the swelling power and gel consistency decreased with the irradiation dose increase. The viscosity – the most important property of starch in applications – showed exponential decrease as irradiation dose increasing with a high radiosensitivity for tuber starch. The paste clarity and its stability improved at the same time with the delay of retrogradation phenomenon as the irradiation dose increase. The gelatinization temperature and enthalpy reduced in a dosedependent manner. All these radioinduced changes depended on the vegetal source of starch and are globally confirmed also by the changes of the paste colorimetric characteristics.

Also, the radioinduced changes are strongly correlated with the structural organization of starch, especially amylopectin, the component with complex branched structure. Thus, for cereal starches with short chains, the molecular weight distribution was affected especially by formation of the fractions with higher molecular weight than the formation of the fractions with low molecular weight. For potato starch with long chains, the molecular weight distribution is just slightly changed by e-beam treatment, the scissions in fractions with high molecular weight being closer to that of the fractions with low molecular weight.

The statistic interpretation using principal component analysis of studied properties pointed out that the multitude of these properties depended on four independent variables which cover 94% of variance. The different evolution of starch properties could be explained mainly by the contribution of Factor 1 containing the apparent viscosity, gel consistency, pH, swelling power, gelatinization enthalpy, number and weight average molecular masses, and irradiation dose. The change of one of these variables led implicitly to similar changes of the others. The modification of one variable leads inherently to other modifications.

The behavior of the structural and physicochemical properties of treated starches highlights the effectiveness of the applied treatment in order to modify them. Both the choice of the starch type and the degree of the treatment depend on the desired characteristics for the proposed application, so that the final product can meet user's specific needs.

5. Acknowledgement

This work has been partially funded by project PN II 51-007/2007.

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Biopolymers Edited by Magdy Elnashar

ISBN 978-953-307-109-1 Hard cover, 612 pages **Publisher** Sciyo **Published online** 28, September, 2010 **Published in print edition** September, 2010

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