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AN ATTEMPT TO DEVELOP EDIBLE PACKAGING FILMS BASED ON VEGETABLE OUTGRADES®

Próba wytworzenia folii jadalnych na bazie wysortu warzywnego®

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Key words: edible films, vegetable outgrades, apple pomace, apple pectin.

The article presents the results of research on the method of recycling the vegetable outgrades of cauliflower, broccoli, yellow and green beans in the form of edible packaging films. The prepared vegetable purees after drying in the form of thin layers did not show a continuous structure, therefore an attempt was made to use dried apple pomace, also a waste material, as a film-forming agent. Studies have shown that they do not fulfill their function due to the low gelling efficiency and a film-forming solution of apple pectin was used, which showed very good gelling capacity in combination with vegetable purées. The tests concerned the determination of appropriate concentrations of the film-forming substance, preparation and addition of vegetables, and drying parameters. It was found that vegetable films with acceptable functional properties were obtained with the use of a film-forming solution based on apple pectin at a concentration of 2.5% in combination with vegetable purees in the ratio of 50:50 and drying at a temperature of 60˚C for 4 hours.

Słowa kluczowe: folie jadalne; wysort warzywny, wytłoki jabłkowe; pektyna jabłkowa.

W artykule przedstawiono wyniki badań dotyczących sposobu zagospodarowania wysortu warzywnego z kalafiora, brokułu, żółtej i zielonej fasolki szparagowej w postaci opakowań jadalnych. Przygotowane purée warzywne po wysuszeniu w postaci cienkich warstw nie wykazywało struktury ciągłej, dlatego podjęto próbę zastosowania suszonych wytłoków jabłkowych, materiału odpadowego, jako substancji żelującej. Badania wykazały, że badane puree warzywne nie spełniło swojej funkcji z uwagi na niską efektywność żelowania i zastosowano foliotwórczy roztwór pektyny jabłkowej, która wykazała bardzo dobre właściwości żelujące w połączeniu z purée warzywnym. Próby dotyczyły określenia odpowiednich stężeń substancji powłokotwórczej, przygotowania i dodatku warzyw oraz parametrów suszenia. Na podstawie przeprowadzonych badań stwierdzono, że folie warzywne o akceptowalnych właściwościach użytkowych otrzymano z zastosowaniem roztworu powłokotwórczego na bazie pektyny jabłkowej o stężeniu 2% w połączeniu z warzywami w stosunku 50:50 oraz suszeniu w temperaturze 60˚C przez 4 godziny.

INTRODUCTION

The large amounts of waste from fruit or vegetable processing are the result of processing or production of juices. Among them skins, seeds, stalks, pomace, husks, leaves can be listed [6]. Improperly stored or utilized fruit and vegetable waste may pose a microbiological hazard and have a negative impact on the natural environment [2]. The main direction of recycling waste from fruit and vegetable industry is the recovery of valuable ingredients. After proper processing, potato skins become a source of fiber, necessary for the proper functioning of the human digestive system. Polyphenols being a bioactive ingredient, are recovered from the peels of citrus, apples and grapes, which often become waste after obtaining the juice. Lycopene and β-carotene can also be recovered

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from the waste obtained from tomatoes [1]. Pectin is produced from apple pomace and orange peels, which can be used to develop edible films and coatings [17]. Banana peels, which make up about 35% of the fruit's weight, are used, among others, in for the production of wine. Pineapple peel can produce ethanol, and properly processed tomato waste can be used as a substitute for wheat flour [1]. Waste from the fruit and vegetable industry can also be used to obtain enzymes. For example, banana peel can be used to produce α-amylase, while strawberry pulp has been used as a substrate for the production of polygalacturonase, which can be used in the clarification of wines and juices, as well as in the production of jams [23].

The discarded fragments and residues of fruit and vegetables are rich in components necessary for a balanced human diet, which can be recovered by different methods from waste and intended for consumption. Among them polyphenols, flavonoids, carotenoids, phytosterols and anthocyanins can be obtained. The main issue that must be taken into account when planning the recovery of valuable components from waste are the profitability and costs [1]. Vegetable outgrades are the parts of vegetables that did not meet certain quality standards allowing for sale, that mean parts rejected during production due to inadequate dimensions. During the production process, some parts, e.g. cauliflower or broccoli, are considered too small or too large and do not meet the classification requirements. However, those parts are still the same vegetable that can be consumed [20]. In this case, cauliflower, broccoli or other vegetables can be used in the production of edible packaging films in the form of sheets or wrappings, which together with the product are the source of vitamins and minerals. In addition, they provide carbohydrates, proteins, dietary fiber and organic acids [13]. Novel food called ‘‘vegetable paper” or ‘‘edible paper”, kind of convinient food, seems to be more popular recently. This is a thin layer obtained by processing vegetables into a puree or past with the addition of various functional substances and then drying. This kind of food is characterised not only as green and nutrient rich components, but also show low sugar, sodium or fat content, which can be ready-to-eat crisp [12]. Vegetable or fruit waste can be used for the production of novel edible packaging films, which can also be a good carrier of nutrientional componds or other functional ingredients [9]. Previous studies showed that edible films such as fruit leather or pestil with a flexible structure can be obtained by hot air drying of fruit purees or juice concentrates with or without the addition of other ingredients. In addition, a lot of research has been carried out on the method of production, parameters and properties of this type of product made of various types of fruit [1, 2, 6, 9, 10, 11]. Edible films or coatings are thin layers of edible material that are formed directly onto the product, while edible coatings are obtained mostly by spraying or immersing the product in a film-forming solution formed by a structured matrix. Edible packaging films are in the form of separately formed sheets, which are then placed on the surface of the food or between the layers of the product [7, 8]. In addition, edible coating can be used as a pre-treatment in osmotic dehydration or drying for fruit or vegetables [15].

The aim of the study was an attempt to produce edible vegetable films from the vegetable outgrades of broccoli, cauliflower, green and yellow beans with the use of apple pomace and apple pectin as gelling substances. The tests concerned the determination of appropriate concentrations of the film-forming substance, preparation and addition of vegetables, and drying parameters. Dry matter and colour were tested and the surface structure was observed.

MATERIALS AND METHODS

Frozen vegetables outgrades were supplied by Unifreese Sp. z o.o. (Górzno, Poland). Apple pomace was obtained in laboratory conditions by drying at 60°C for 14 hours the waste left after squeezing. A low methylated apple pectin was pourchased from Agnex (Białystok, Poland). Glycerol (Avantor Performance Materials Poland S.A.Gliwice, Poland) was used as a plasticizer.

Technological methods

The frozen vegetables were boiled in water to getting soft (broccoli 7 min, cauliflower and green beans 10 min) and were ground to a smooth mass in a knife mill GM 200 (Retsch, Katowice). Each type of vegetable has been cooked and processed separately. The film-forming solutions were prepared in different variations by combining distilled water with powdered dried apple pomace at the concentration of 2, 4, 5 or 6% or apple pectin at the concentration 2 and 2,5%. The solutions were heated and stirred for 30 minutes using a RCT basic IKAMAG magnetic stirrer (IKA Poland, Warsaw) at the level of 200 rpm at a temperature of 60°C. After cooling, glycerol was added to the solutions in an amount representing 50% of the added apple pomace or apple pectin, and calcium lactate (Avantor Performance Materials Poland, Gliwice) in an amount representing 1% of the added film-forming substance. The vegetable purees and the film-forming solutions were combined in a mass ratio of 50:50 and poured on byko-charts test cards with a thickness of 0.35 mm (model A4 PA-2824, Eurotom Sp. z o.o., Warsaw) and spread using a slotted applicator using an automatic layering table (ZAA 2300, Zehntner Testing Instruments, Sissach, Switzerland) travel speeds 90 mm/s. The thickness of the applied layers was 2500 μm. The samples were dried from 35 to 60°C in a laboratory dryer SUP-65W (Wamed Wytwórnia Aparatury Medycznej SSP, Warsaw). The dryed vegetavble films were conditioned in a climate chamber model KBF 240 (Binder GmbH, Tuttlingen, Germany) at 25°C and relative humidity of 50% for 48 hours prior testing.

Colour

The colour test was performed using the CR-300 model colorimeter (Minolta, Japan) in the CIE $L^*a^*b^*$ system (L^* - lightness, a^* - green to red colour, b^* - blue to yellow colour). The measurement was performed in ten repetitions. For a better interpretation, the the total colour difference (ΔE) between the film and the white standard (L^*=97.12±0.10; a^*=0.05±0.01; b^*=1.84±0.04) was calculated according to the method described by Sobral, dos Santos & Garcia [22]:

$$\Delta E = \sqrt{(L^* - L)^2 + (a^* - a)^2 + (b^* - b)^2}$$

where: ΔE – total colour difference;
L*, a*, b* - parameters for white standard;
L, a, b – parameters for films.

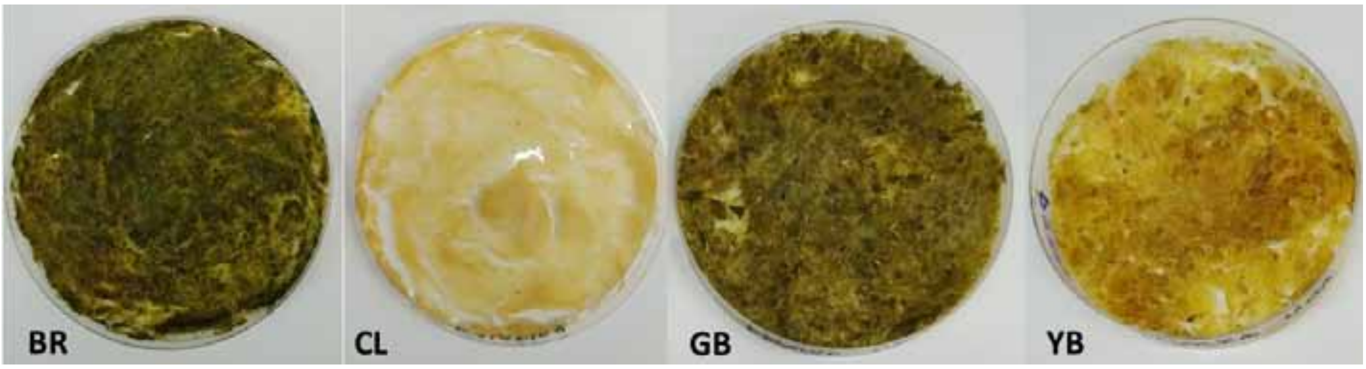

Fig. 1. Vegetable purees dryed at the temperature of 35˚C; BR – broccoli; CL - cauliflower, GB - green bean, YB - yellow beans.

Rys. 1. Wysuszone w temperaturze 35°C puree warzywne; BR – brokuł; CL – kalafior, GB – zielona fasolka szparagowa, YB – żółta fasolka szparagowa.

Source: The own study

Źródło: Badania własne

Dry matter

The film dry matter was determined at least in three repetitions through the weight loss undergone by the film after 24 h oven drying at 105±1°C.

Microstructure

The observations of the film microstructure were performed using the FEI Quanda 200 scanning electron microscope (Brno, Czech Republic) at low vacuum from 0.35 to 1 Torr and the magnification 100x.

Statistical analysis

Statistica 11 (StatSoft Inc., Tulsa, OK, USA) was used to analyze the obtained results. The analysis of variance (ANOVA) at a significance level of 0.05 was performed with Tukey's post hoc test to detect significant differences in film properties.

RESULTS AND DISCUSSION

Several studies have been carried out on the development of films or coatings based on fruits and vegetables [1, 2, 6, 9]. These investigations relied mostly upon combining different kind film-forming hydrocolloids with fruit and vegetable purees. Moreover, fruit and vegetable are sources of nutrients and antioxidants that may be ingested in form of edible films made from them [19]. The different types of vegetable films are determined from the different raw materials. Nowadays, the vegetable layers are developed mostly at home and have a single vegetable as the raw material. However, there is also a posibility to prepare a composite vegetable films based on the mixture of multiple vegetables. Nevertheless, most research is still at the laboratory stage [12]. Some fruits contain enough pectins that make them possible to produce films without the addition of gelling agents, such as apple, black currant or plum, which was presented in our previous study [11]. However, vegetables are characterized by different compositions and vegetable purees that were used in this study, from cauliflower, broccoli, yellow and green beans, did not contain a sufficient amount of gelling compounds to create thin layers with film-forming capacity (Fig 1). Therefore apple pomace, as waste material and apple pectin were used in different concentrations to form vegetable films. A summary of the tests carried out and the results achieved in this study are presented in Table 1. In general, in many studies, polysaccharides had been used as biopolymer materials, including alginate, carrageenan, starch and xanthan gum, which can form edible films or coatings to reduce petroleum-based packaging [18].

Films obtain from apple pomace and glycerol were brittle and did not show continuous structure, which was improved by the addition of calcium lactate. Fig. 2 shows the comparison of such films prepared at the concentration of 6% of apple pectin and dryed at 35°C. It can be clearly concluded that the addition of calcium lactate is necessary to maintain a homogeneous film structure. Then, different concentration and temperature as well as proportions of film-forming solutions and vegetable purees were used to obtain films with the desired film capacity. The main trials and results were presented in Table 1.

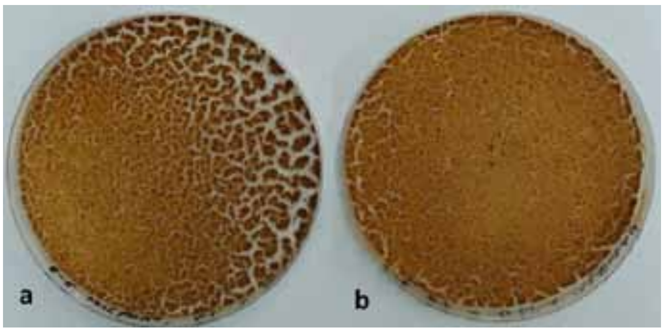

Fig. 2. Pure films prepared based on apple pomace without (a) and with calcium lactate (b).

Rys. 2. Folie wytworzone z wytłoków jabłkowych bez warzyw oraz bez dodatku (a) i z dodatkiem (b) mleczanu wapnia.

Source: The own study

Źródło: Badania własne

The first trial consisted in the production of films with a minimal addition of gelling substances in order to obtain so-called clean label with the most content of vegetable purees. The test was carried out to compare apple pomace and pectin-based films, since pectin is widely used biopolymer

Table 1. The most important technological trials of preparation films with vegetable purees with the obtained results
Tabela 1. Zestawienie najważniejszych prób technologicznych wytworzenia folii z purée warzywnego i osiągnięte wyniki

Type of film-forming agent	Concentration of film-forming agent [%]	Drying temperature [°C]	Drying time [h]	Observations
Apple pomace	2	60	24	lack of the desired gelling capacity and continuous structure; films too dry
	4	35	48	continuous structure, impossible to peel off the films
	5	60	24	lack of continuous structure, some parts burnt
	6	35	48	lack of desired gelling and continuous structure
Apple pectin	2	60	24	desired geling capacity, continuous structure, long time of drying, some parts burnt
	2,5	60	24	continuous and homogeneous structure, easy to peel off the films

Source: The own study
Źródło: Badania własne

in production of edible films or coatings and their properties meet the mechanical resistance requirements for packaging films [17]. Different temperatures were used from 35 to 60°C resulting in varied drying times. Relatively good results were observed when apple pectin was used at 2%, while films prepared with the addition of apple pomace showed rather noncontinuous structure due to the low content of pectin. The photograps of obtained vegetable films are presented in Fig. 3. Tarko et al. [24] observed that apple pomace contains pectins at the level of 0.6/100 g, however its content varies depending on the fruit type, method of processing and cultivator of fruit pomace [10, 14]. Therefore, increased concentations from 2 to 6% of apple pomace were used and different observations were noted indicating that there is no possibility to obtain smooth vegetable films with apple pomace as gelling agent due to the lack of film-forming capacity.

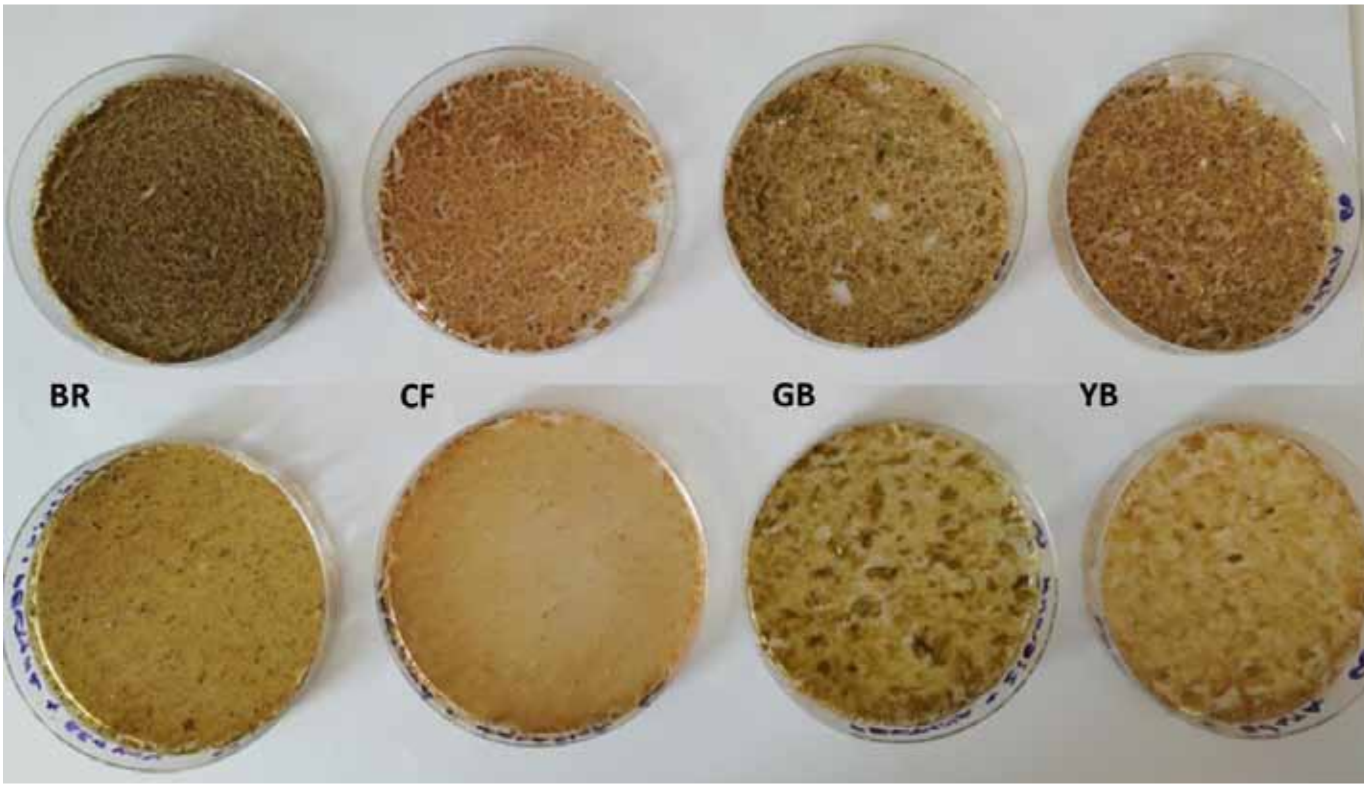

Fig. 3. Films prepared with the addition of apple pomace (top row) or apple pectin (lower row) based on vegetable puree from broccoli (BR), cauliflower (CF), green beans (GB), and yellow beans (YB).
Rys. 3. Folie wytworzone z dodatkiem wytłoków jabłkowych (powyżej) lub pektyny jabłkowej (poniżej) z puree warzywnego z brokułu (BR), kalafiora (CF), zielonej fasolki szparagowej (GB) i żółtej fasolki szparagowej (YB).

Source: The own study
Źródło: Badania własne

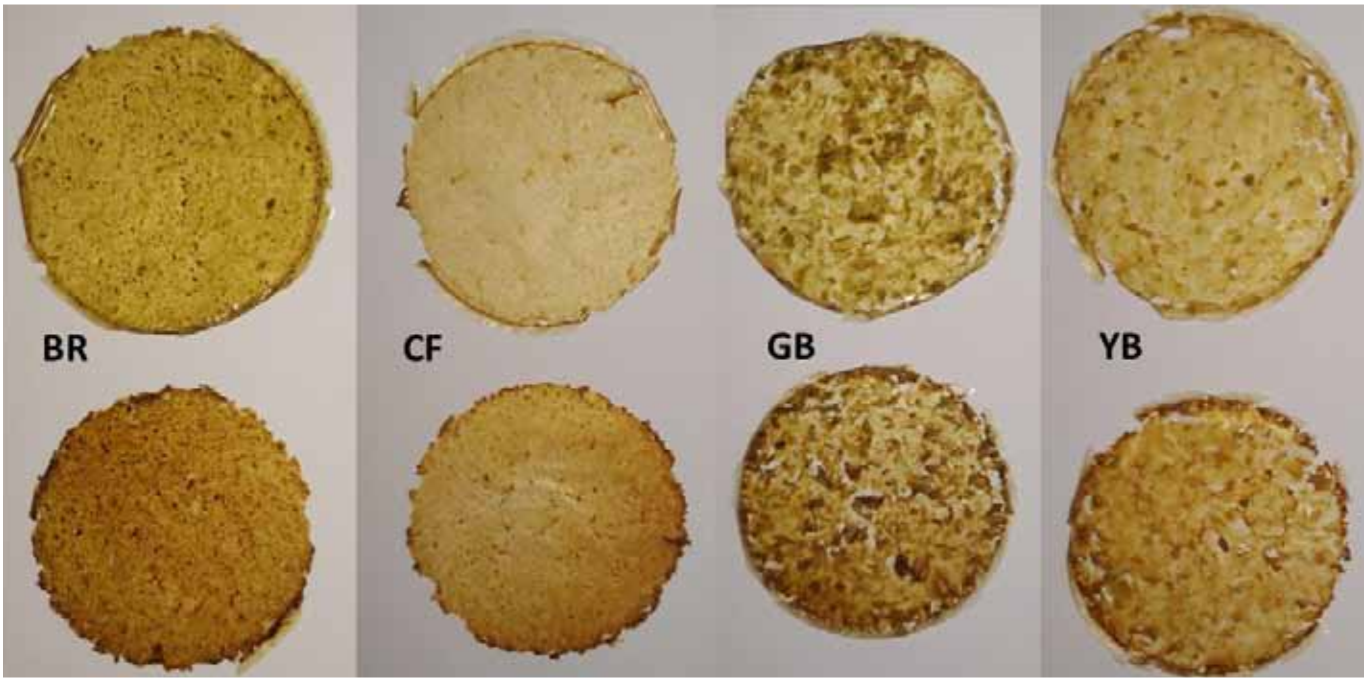

Fig. 4. Films prepared with apple pectin dryed at the temperature of 35°C (top row) and 60°C (lower row) (lower row) based on vegetable puree from broccoli (BR), cauliflower (CF), green beans (GB), and yellow beans (YB).

Rys. 4. Folie wytworzone z dodatkiem pektyny jabłkowej suszone w temperaturze 35°C (powyżej) i 60°C (poniżej) z puree warzywnego z brokułu (BR), kalafiora (CF), zielonej fasolki szparagowej (GB) i żółtej fasolki szparagowej (YB).

Source: The own study

Źródło: Badania własne

The addition of apple pectin was finally changed to lightly higher content (2.5%) to obtain films with better mechanical resistance. Different drying conditions were applied to determine the most relevant drying conditions to obtain films with desired functional properties. Fig. 4 shows the comparison of films dryed at 35°C for 72 h and 60°C for 24 h. It can be observed that higher temperature resulted in darker films with more compact structure, while films dryed at lower temperature were more humid with softened structure.

In the development process of edible films based on vegetables, it is necessary to protect the colour and retain its original colour [12]. The colour of the films is a crucial parameter, which need to be control and taken into account in order to minimize their change during the film preparation. The control films were transparent with lighty yellow colour connected to the pure pectin powder nature, while vegetable films showed colour similar to the vegetable that were used in the proces.

The results of colour parameters are presented in Table 2. Control films prepared without the addition of vegetables showed highest lightness (parametr *L**), 89.51, which is similar to the values obtained for pectin films by others [21]. Vegetable films were characterized by much lower *L** values, from 49.70 for films contaning green bean puree to 63.52 for films contianing yellow bean purees. Lighter vegetables, the lighter films were obtain, which can be observed for cauliflower and yellow bean purees contaning films (63.23–63.52). All vegetable films showed statistically significant lower lightness. All films had positive *a** (green-red) and *b** (blue-yellow) parameters. A statistically significant increase of the *a** parameter was observed, from 0.23 for control films to 11.54 for films containing cauliflower purees. Among vegetable films, the lowest values of parameter *a** were observed for films with the purees based on broccoli and green beans, 4.01 and 2.35, respectively. Films containing yellow beans showed *a** value of 8.84. The obtained data indicates that higher value of parameter a* means colour toward to red, however the values are relively low and it does not correspond with the natural colour of the raw vegetables. This is probably attributed to the changes of pigments that can occur durring the technological and drying proces, as well as the interactions between the biopolymer (apple pectin) and vegetables. Regarding the values of parameter *b**, it is noted that all films were positive values, from 13.06 for control films to 34.07 for films contaning puree from blocolli, indicating the colour toward yellow. Statistically significant increases in values were observed for films contanings purees from broccoli, cauliflower and yellow beans, which *b** values were similar, from 31.54 to 34.07, whereas films contaning green beans showed aproximate value to control films (16.69). The changes in colour parameters are due to the colour of pure apple pectin, which also affects the film appearance.

To better understand the colour changes, the total colour difference (ΔE) between the analyzed films and the white standard was calculated. The ΔE values ranged from 14.20 for control films to 51.12 for films contaning puree from broccoli. Statistically signifiact increase in ΔE values was observed for all films with vegetables, which indicates that the changes in colour are relatively high.

Table 2. L*, a*, b* colour parametres, total colour differece (ΔE) and dry matter of control films and films based on vegetable puree from broccoli (BR), cauliflower (CF), green beans (GB), and yellow beans (YB)

Tabela 2. Parametry barwy L*, a*, b*, całkowita różnica barwy (ΔE) i zawartość suchej substancji w foliach kontrolnych i z na bazie puree warzywnego z brokułu (BR), kalafiora (CF), zielonej fasolki szparagowej (GB) i żółtej fasolki szparagowej (YB)

Film	*L**	*a**	*b**	Δ*E*	Dry matter [%]
Control	89.51 ± 5.73^{c}	0.23 ± 0.03^{a}	13.06 ± 3.16^{a}	14.20 ± 3.66^{a}	3.05 ± 0.02^{a}
BR	58.23 ± 5.80^{b}	4.01 ± 1.66^{b}	34.07 ± 4.74^{b}	51.12 ± 1.44^{d}	6.63 ± 0.33^{bc}
CF	63.23 ± 2.53^{b}	11.54 ± 0.79^{d}	33.05 ± 1.05^{b}	47.54 ± 1.34^{bc}	5.12 ± 0.43^{b}
GB	49.70 ± 1.89^{a}	3.25 ± 0.64^{b}	16.69 ± 3.16^{a}	49.90 ± 1.49^{cd}	8.63 ± 0.92^{d}
YB	63.52 ± 2.02^{b}	8.84 ± 0.48^{c}	31.54 ± 1.54^{b}	45.74 ± 1.45^{b}	7.67 ± 0.75^{cd}

Mean values ± standard deviations. Different superscripts letters ($^{a-d}$) within the same column indicate significant differences between the films ($p < 0.05$).

Source: The own study

Źródło: Badania własne

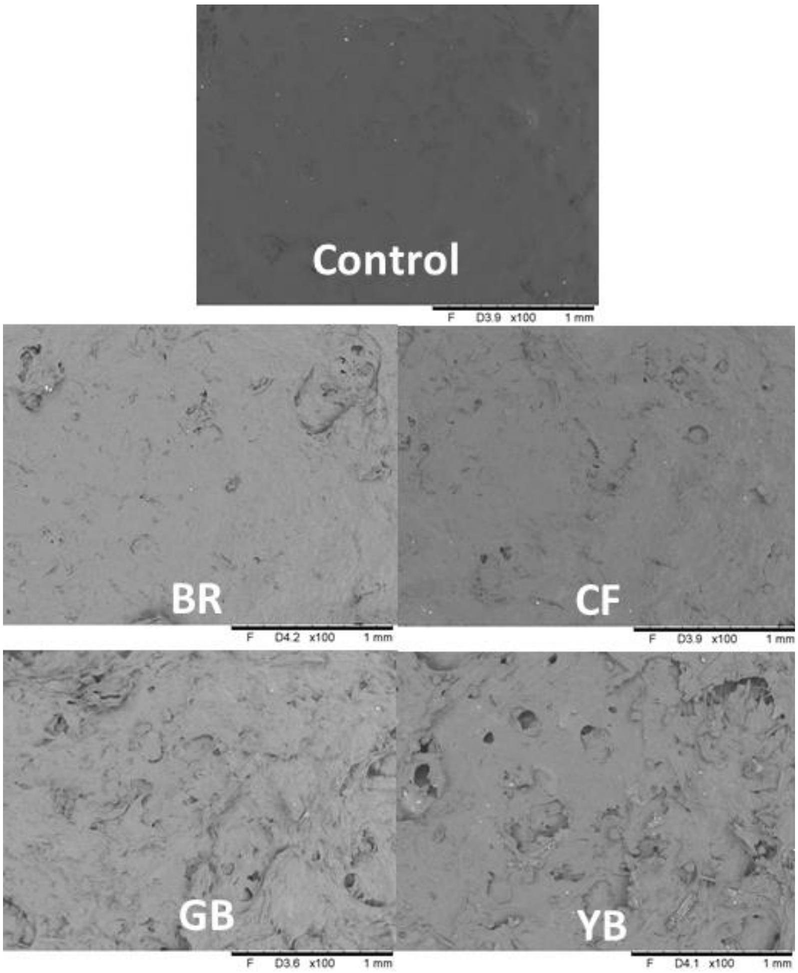

Fig. 5. Scanning electron micrographs of control films prepared only from apple pectin and films based on apple pectin and vegetable puree from broccoli (BR), cauliflower (CF), green beans (GB) and yellow beans (YB).

Rys. 5. Zdjęcia mikroskopowe folii wytworzonych z dodatkiem pektyny jabłkowej (control) i z dodatkiem puree warzywnego z brokułu (BR), kalafiora (CF), zielonej fasolki szparagowej (GB) i żółtej fasolki szparagowej (YB).

Source: The own study

Źródło: Badania własne

In general, those values are high and can be observed visually, based on the clasiffication presented by International Commission of Illumination (CIE), values of $\Delta E > 3.5$ are distinctly perceivable [21]. In addition, total colour difference is a good determinant of colour for films since it takes into account all three color parameters *L**, *a**, and *b** [8]. The study conducted by Chakravartula et al. [3] gave similar results, where the total colour difference of the 3% apple pectin films was 11.3. Similar results were obtained by Siracusa et al. [21].

The dry matter of analyzed films ranged from 3.05% for control films to 7.67% for films containing puree from yellow beans (Table 2.) Statistically significant increse in the values was observed for all vegetable films. A tendency of higher values (7.67–8.63%) can be noted for films based on green and yellow green beans, which can be connected with the higher content of solid parts and probably dietary fiber, which were observed in the films with heterogeneous structure. Therefore, more smooth films contaning cauliflower or broccoli purees were smoother and showed lower values of dry matter (5.12–6.63). Morover, taking into account that the film-forming solutions were mixed with vegetable purees at the proportion of 50:50 the values of dry matter of films are much lower that those noted for cauliflower [5] and broccoli [4], 9.3% and 11%, respectively.

The microstructure is the main characteristic of the film, which represents its surface morphology and internal structure [25]. Scanning electron micrographs of analyzed films are presented in Fig. 5. It can be observed that control films are characterized by homogeneous and continuous, both on a micro and macro scale. The surface is smooth, without pores or cracks, which is typical for most biopolymer films [8, 16]. All vegetable films showed rough, heterogeneous surface with pores, but continuous structure. There is a lightly difference between films and it can be noted, that films contaning puree from cauliflower or broccoli are more homogeneous in comparison to the films prepared with the addition of green and yellow beans. This is attributed to the structure of the vegetables and their compositions, mostly dietary fiber content and other not soluble fractions. These observations are in line with the results of dry matter (Table 2), higher values corresponed with more heterogeneous structure.

PODSUMOWANIE

W artykule przedstawiono wybrane wyniki badań nad otrzymaniem folii jadalnych na bazie wytłoków jabłkowych oraz pektyny jabłkowej w połączeniu z puree z warzyw pochodzących z mrożonego wysortu warzywnego z kalafiora, brokułu, żółtej i zielonej fasolki szparagowej. Próby dotyczyły określenia odpowiednich stężeń substancji powłokotwórczej, przygotowania i dodatku warzyw oraz parametrów suszenia powłok. Zastosowane warzywa bez dodatku substancji żelujących nie miały właściwości powłokotwórczych. Wytłoki jabłkowe nie wykazały pożądanych właściwości żelujących. Zawartość pektyn o stężeniu 2 i 2,5% w roztworze powłokotwórczym była wystarczająca do otrzymania folii o akceptowalnych właściwościach użytkowych. Dodatek mleczanu wapnia był niezbędny do uzyskania jednolitej struktury. Najlepsze właściwości użytkowe wykazały folie warzywne otrzymane poprzez połączenie warzyw w stosunku masowym 50:50 z roztworem powłokotwórczym zawierającym 2.5% pektyny, w obecności glicerolu (50% względem pektyny) i mleczanu wapnia (1% w stosunku do pektyny). Optymalne warunki suszenia folii warzywnych to temperatura 60°C i czas 4 godziny. Wytworzone folie warzywne o zróżnicowanej barwie i strukturze mogą znaleźć zastosowanie w projektowaniu nowych produktów m.in.: jako bezglutenowe przekąski warzywne lub powłoki funkcjonalne do batonów owocowo-warzywnych.

SUMMARY

The article presents the selected results of the study on the preparation of edible films based on apple pomace and apple pectin in combination with vegetable puree obtained from a frozen vegetable outgrades of cauliflower, broccoli, yellow and green beans. The analyzes concerned the determination of appropriate concentrations of the film-forming solutions, preparation and addition of vegetables as well as parameters for film drying. The vegetables used without the addition of gelling substances showed no film-forming capacity. Apple pomace did not show the desired gelling properties. The pectin content of 2 and 2.5% in the film-forming solutions was sufficient to obtain a film with acceptable performance properties. The addition of calcium lactate was necessary to obtain a homogeneous structure. The best functional properties were demonstrated by vegetable films with acceptable functional properties, obtained by combining of vegetable puree and film-forming solutions based on apple pectin at 2.5% in a weight ratio of 50:50 and in the presence of and calcium lactate. The optimal conditions for drying the vegetable films were determined at the temperature of 60°C and the time of 4 hours. The analyzed vegetable films with different colours and structures can be used in the new product development e.g. as gluten-free vegetable snacks or functional coatings for fruit and vegetable bars.

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