CHARACTERIZATION OF SUNFLOWER PECTINS DEPENDING ON THE MATURITY STAGE OF PLANTS

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INTRODUCTION

Stoikoff (1948) found that the sunflower heads were rich in pectin raw material exceeding 25% polyuronide content and might be applied to the industrial production of pectin. Stoikoff (1958) established that the sunflower pectin was similar to the apple and citrus pectin in its properties. Since then an increasing interest has been continuously taken in this new pectic raw material (Monselise, 1954 ; Qudrat et al., 1972 ; Riaz and Uddin, 1972 ; Sabir et al., 1976 ; Lin et al., 1976, 1978).

It is known that the pectic substances in fruits sustained qualitative and quantitative changes in passing through various physiological stages (Tishchenko and Sapozhnikova, 1972 ; Rouse and Grandall, 1978 ; Bhattacharyya et al., 1970). The literature provides scanty and inconsistent data on the yield and the quality of sunflower pectins depending on the harvest time of sunflower heads. Andreev (1956) found that the quality of pectic substances in sunflower heads increased from 8.5% to 18.8% with the development of plant and obtained its maximum in full maturity. Upon studying the sunflower plant in three maturity stages Lüdtke (1961) established that the total amount of pectic substances in the whole plant was slightly dependent on the development stage (from 9.57 to 10.98).

According to Lüdtke, the changes in the methoxyl content were non-essential while more important changes were observed in the degree of polymerization. Stoikoff (1958) showed that the degree of esterification and the gelling power of pectin decreased strongly with passing from yellow brown to brown maturity of sunflower which was confirmed in our later studies (Stoikoff and Kratchanov, 1966). Lin et al, (1975) studied three sunflower cultivars in three maturity stages and concluded that the methoxyl number of suflower pectin was more strongly dependent on the cultivar than the maturity of heads. Campbell et al. (1978), when studying four sunflower cultivars in three development stages, confirmed the observations of Stoikoff that the degree of esterification of sunflower pectin decreased with the maturity of plant. They found that the watersoluble fraction of sunflower pectin did not give firm jellies while the gelling power of the oxalate-soluble fraction was good and depended slightly on the maturity stage of sunflower heads. These contradictory data and the problem of industrial production of sunflower pectin in our country resulted in carrying out the studies on the effect of the development stage of sunflower on the pectin content of sunflower heads and the characteristics of sunflower pectins.

MATERIALS AND METHODS

Sunflower heads of the cultivar Peredovik, crops 1975, 1976 and 1977, harvested in the experimental fields at the Vassil Kolarov Agricultural Institute in Plovdiv, were used for this study. After harvesting the heads were threshed to remove the seeds and shredded in pieces of 3—7 cm. The raw material thus obtained was dried in a laboratory forced draft air oven at 50°C to a moisture of 5—8%. The heads were ground in a laboratory mill and stored in air-tight packages. The analysis for polyuronide content and degree of esterification of the raw materials was made by a modified method of Gee, McComb and McCready (Gee et al., 1958).

The extraction of the pectins was achieved with 0.5% oxalic acid in accordance with a previously described method (Stoikoff and Kratchanov, 1966), while the precipitation was carried out with a double volume of ethyl alcohol. The coagulate was repeatedly washed with ethanol acidified with hydrochloric acid, and finally with 70% ethanol to neutral reaction.

The degree of esterification and the purity of the pectic preparations obtained were determined by neutralization on a Hinton indicator (Hinton, 1940).

The gelling power of the pectins obtained was established by the firmness of standard 65% sugar jellies according to the method and the equipment of Tarr-Baker (Baker, 1926).

The viscosity numbers were evaluated by the method of Deuel (Deuel, 1943) using the Höppler viscosimeter.

RESULTS AND DISCUSSION

We studied initially the changes in the pectic substances of sunflower heads in terms of the polyuronide content and the degree of esterification determined by the method of Gee, McComb and McCready (Gee et al., 1958). The data are shown in Table 1 as

Table 1

Polyuronide content of sunflower heads depending on the maturity degree

Sample No.	Maturity degree of sunflower heads	Polyuronide content %	Degree of esterification %
1	Floscules	19.0—20.0	64.0—66.0
2	Early wax maturity	20.0—22.0	60.0—62.0
3	Wax maturity	21.0—24.0	53.0—59.0
4	Yellow brown maturity	22.0—25.0	50.0—56.0
5	Yellow brown to brown maturity	23.0—27.0	41.0—53.0
6	Brown maturity	22.0—26.0	38.0—48.0

average values for the three crops investigated (1975, 1976 and 1977). The polyuronide content changed from 19% in floscules to about 25% at brown maturity passing through maximum at yellow brown to brown maturity. Hence, it follows that the accumulation of pectic substances in the sunflower heads occurred gradually with the development and ripening — to yellow brown maturity when the heads had their largest volume. The degree of esterification of the pectic substances contained in the heads decreased gradually with the development of the plant from 65% (in floscules) in the beginning to bellow 40% at brown maturity. It follows from the data that in the initial period of vitality the plant needed a pectin having more expressed hydrophile properties and a lower ion exchange capacity. A decrease in the degree of esterification of the pectin was also observed at ripening of citrus and apples (Tiaschenko and Sapozhnikova, 1972 ; Rouse and Grandall, 1978).

In a further series of expriments, sunflower heads of crop 1977 were subjected to extraction with 0.5% oxalic acid. Table 2 shows the characteristics of the pectic preparations obtained. It is seen that under these conditions 65—85% of pectins were extracted from the raw material. It is worth noting that the degree of esterification of the pectins obtained was generally lower than that of the polyuronides contained in the heads (Table 1), and in this instance it decreased gradually with maturity of plant. The lower values of degree of esterification can be attributed to various reasons such as extraction of the lower esterified part of pectin, partial deesterification under the conditions of extraction, etc. The gelling power of preparations reached its maximum at yellow brown maturity of heads. A strict correlation between the viscosity number Z (measure for the degree of polimerization of pectin) and the gelling power was not observed, presumably for the gelling properties depended also on other factors such as the degree of esterification, for instance. It is of great significance for studying the physiological functions of the pectin in the plant, in our opinion, that with advancing the ripeness after wax maturity the viscosity number decreased nearly in parallel with lowering the degree of esterification. It shows that processes of pectolysis have started which resulted in decreasing the water uptake of the cell system, i.e. in lessening the turgur of cells and moreover the active exchange processes in plant. These changes affected also the appearance of the sunflower heads-with passing from wax maturity to yellow brown and brown maturity especially, the moisture content of the heads decreased and a sharp reduction of their geometric size was observed as well.

Table 2

Characteristics of pectic preparations obtained from sunflower heads in various maturity stages, crop 1977

Sample No.	Maturity degree	Yield %	Purity %	Degree of esterification %	Gelling power acc. to Tarr-Baker	Viscosity number acc. to Deuel
1	Floscules	14.2	84.8	56.4	190	1.14
2	Early vax maturity	16.1	87.8	54.0	190	1.29
3	Wax maturity	18.4	94.8	51.8	200	1.44
4	Yellow brown maturity	19.2	95.8	44.5	240	1.26
5	Yellow brown to brown maturity	23.1	94.8	42.5	220	0.99
6	Brown maturity	22.4	97.8	39.7	180	0.97

The above discussed data also suggested that in selecting the harvest period of sunflower heads, it should be taken into consideration not only the technological properties of the sunflower plant as oleaginous raw material but also as a raw material for the production

of pectin. For certain applications of the sunflower pectin such as gelling agent, thickener or product for curative and preventive purposes, the sunflower should be harvested at yellow brown maturity instead of brown maturity as is practised recently in agriculture.

CONCLUSIONS

1. The polyuronide content of threshed sunflower heads varied within 19—27% passing through maximum at yellow brown to brown maturity.

2. The degree of esterification of sunflower head pectic substances decreased gradually from 66% to 38%, being the highest in the stage of floscules and the lowest in brown maturity stage.

3. The gelling power of oxalate-extracted pectic preparations was dependent on the maturity of sunflower heads while a trend toward increasing to yellow brown maturity was noted followed by decreasing.

4. The viscosity number of the pectin passed also through maximum during the development of sunflower (the highest value was reported at wax maturity) while a strict correlation between the gelling properties dependent on the degree of esterification was not observed.

5. The polyuronide content of sunflower heads, the physico-chemical properties and the chemical content of the sunflower pectin were mostly dependent on the maturity stage and to much lesser extent on the year of cultivation.

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CARACTÉRISATION DES PECTINES DU TOURNESOL EN FONCTION DU STADE DE MATURITÉ DES PLANTES

Résumé

Sur les modifications de la teneur en pectine et de ses propriétés au cours de la maturation des plantes de tournesol, les avis sont très différents. Le présent ouvrage expose les résultats de l'étude des capitules de la variété Peredovik, récoltés à différents stades de maturité des plantes, pendant les années 1975—1977. La teneur en polyuronides des capitules hachés a varié de 19% à 27% la valeur maximale se trouvant entre la maturité jaune et la maturité brune.

L'estérification des substances pectiques a diminué graduellement de 66 à 38%, étant plus importante au moment du remplissage des graines et plus faible au moment de la pleine maturité. La production de pectine suit une courbe qui débute avec 14% au moment du remplissage des graines, atteint 23% pendant la période entre la maturité jaune et la maturité brune et prend après une allure descendante. Jusqu'à la maturité brune la pureté des substances pectiques augmente régulièrement (97,8%), tandis que l'estérification baise de 56,4 à 39,7%. La capacité de gélification des substances pectiques extraites à l'acide oxalique et coagulées à l'étanol acidifié augmente jusqu'à la maturité jaunebrune. La viscosité

atteint le plus haut chiffre au cours du stade pâteux de maturité. Entre la capacité de gélification et le degré d'estérification, il n'y a aucune corrélation. Les conditions climatiques des années des essais ont eu une faible ou très faible influence sur la quantité et les propriétés des pectines.

CARACTERIZACIÓN DE LAS PECTINAS DE GIRASOL EN FUNCIÓN DEL ESTADIO DE MADUREZ DE LAS PLANTAS

Resúmen

Hay varias opiniones diferentes con respecto a los cambios del contenida de pectina y de sus rasgos, una vez maduradas las plantas de girasol. En el presente trabajo se han estudiado capítulos de la especie Peredovik, recogidos en diferentes estadios de madures de las plantas, durante el período 1975—1977. El contenido en poliuronidas de los capítulos picados varió entre 19 y 27 por ciento, pasando por el punto máximo en la fase de entre la madurez amarilla y la madurez castaña. El grado de esterificación de las sustancias pécticas disminuyó gradualmente, desde 66 hasta 38 por ciento, llegando a ser 10 màs elevado en el período de relleno de las semillas y 10 màs reducido a la sazón. La producción de pectina persiguió una curva que partió desde 14 por ciento en la fase de relleno de las semillas y alcanzó 23 por ciento en la fase de entre la madurez amarilla y la madurez castaña y luego empezó a disminuèr. La pureza de las sustancias pécticas aumentó permanentemente hasta la madurez castaña (97,8 por ciento), mientras que el grado de esterificación disminuyó desde 56,4 hasta 39,7 por ciento. El poder de gelatinización de las sustancias pécticas, extraídas con ácido oxálico y coaguladas con etanol acidifiádo fue máximo en la fase de madurez amarilla-castaña, mientras que la ligamaza tuvo el mayor número durante la fase de madurez en cera. No se ha notado correlación entre el poder de gelatinización y el grado de esterificación. Los anos han influido poco o muy poco en la cantidad y los rasgos de las pectinas.