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EFFECT OF CHITOSAN-BASED BIOACTIVE COATINGS ON QUALITY PARAMETERS OF WALNUT FRUIT

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ABSTRACT

This article incorporates Greek Walnut (Juglans regia L.) the effectiveness of the use of biologically active chitosan-based coatings in the process of fruit preservation has been studied. During the study, Walnut fruits in a shell (whole) and shell – free (Magg) state were treated with 0.1% solutions of biologically active substances–X3 and FA-2, and were compared with control options. During storage, organoleptic indicators and physicochemical indicators such as moisture content, ash content, total fat content, Acid Number and peroxide number were analyzed.

The results of the study showed that Shell nut samples contained a moisture content of 4.15–4.94 %, ash content of 1.60–3.28 %, total fat content of 55.33–66.95 %, acid count of 0.88–1.58 mg KOH/G, and peroxide count of 0.15–1.33 mmol o_2/kg. In the case of shellless (Magg) samples, however, the moisture content was recorded in the range of 3.99–4.44%, ash content 1.98–3.86%, fat content 62.03–68.31%, acid number 0.79–1.08 mg KOH/g and peroxide number 0.40–0.54 mmol O_2/kg.

The analysis found that in variants treated with biologically active coatings, quality indicators are more stable than control samples. In particular, it was observed that in variants where the X3 solution was applied, moisture exchange slowed, the oil fraction was better maintained, and the increase in acid and peroxide numbers was limited. Shell nut fruits showed higher stability during storage compared to shellless specimens.

The results obtained confirm that the use of biologically active coatings based on chitosan is a promising method for extending the shelf life of nut products, stabilizing quality indicators and developing environmentally friendly storage technologies.

Keywords

Juglans regia L., chitosan, storage technology, peroxide number, biopolymer.

INTRODUCTION

At present, ensuring food security worldwide is considered one of the priority areas of agriculture and the food industry. Population growth, increasing demand for food products, and the high proportion of post-harvest losses necessitate the development of effective technologies for storing agricultural products. At the same time, the increasing impact of diseases, microorganisms, and oxidative factors during the production and storage of products is considered one of the main factors reducing storage efficiency.

Traditionally, various chemical agents have been used to protect products. However, their excessive use may have adverse effects on human health and the environment. Therefore, in recent years, research aimed at developing environmentally safe, biodegradable, and naturally derived protective agents has been developing rapidly.

Walnut (*Juglans regia L.*) is one of the agricultural products with high nutritional and biological value. It contains high levels of unsaturated fatty acids, proteins, minerals, phenolic compounds, and antioxidant components. At the same time, the high content of the lipid fraction in walnuts accelerates oxidation processes during storage, resulting in deterioration of product quality. Consequently, organoleptic properties deteriorate, while the amounts of free fatty acids and peroxide compounds increase (1,4).

According to FAOSTAT data, global walnut production in 2024 amounted to 4.3 million tonnes, while the harvested area reached 1.39 million hectares. By region, Asia accounted for the largest share, with production amounting to 2,813,884 tonnes. This was followed by the Americas (914,892 tonnes), Europe (429,233 tonnes), Africa (178,658 tonnes), and Oceania (12,497 tonnes). At the country level, China, the United States, and Türkiye were the leading producers, while Uzbekistan ranked 12th in the world with a production volume of 49,148 tonnes (12).

The use of biopolymer-based coatings is considered a promising technological solution for reducing quality losses occurring during storage. Among such substances, chitosan is of particular importance. Chitosan is a natural polysaccharide obtained

through the partial deacetylation of chitin and is characterized by high biocompatibility, antimicrobial activity, and antioxidant properties.

According to the literature, chitosan-based coatings form a semipermeable protective layer on the surface of products, limiting gas exchange and moisture loss. As a result, the rate of lipid oxidation decreases, microbiological deterioration is reduced, and the shelf life of the product is extended. These properties are particularly important for walnuts and other oil-rich products (3,5).

The main objective of this study was to evaluate the effect of chitosan-based biologically active coatings on the quality parameters of walnut fruits and to scientifically substantiate their storage efficiency.

MATERIALS AND METHODS

Walnut fruits (*Juglans regia L.*) were selected as the research object. The experiments were conducted under the conditions of the Surkhandarya region, the southern part of the Republic of Uzbekistan. The scientific research was carried out within the framework of a project planned for 2025–2027 at Termez State University of Engineering and Agrotechnologies in scientific cooperation with scientists from Tashkent State Agrarian University. The storage of walnuts and the experimental procedures were organized at “Surxon Invest” LLC.

Three walnut varieties included in the State Register of the Republic of Uzbekistan—Yubileniy, Ideal, and Tonkoskorlupiy—were used in the study. During the research, walnut samples were divided into separate groups as shelled (whole nuts) and unshelled (kernels).

Each variety was studied using the following experimental treatments:

— Control group — samples not treated with a biologically active coating;

— Experimental group 1 — samples treated with a 0.1% solution of the X3 biologically active substance;

— Experimental group 2 — samples treated with a 0.1% solution of the FA–2 biologically active substance.

After treatment, the samples were stored under standard storage conditions for six months and periodically analyzed for quality parameters.

RESEARCH METHODS

Organoleptic and physicochemical analytical methods were used to evaluate the quality condition of walnut fruits during storage. The following parameters were determined throughout the study:

— moisture content;

— ash content;

— total fat content;

— free fatty acids (acid value);

— peroxide value.

All analyses were performed in accordance with the applicable GOST standards.

Determination of moisture content. The moisture content of walnut fruits was determined by the drying method in accordance with GOST 9404–88. The method is based on drying the sample at a temperature of 105±2 °C until a constant mass is obtained.

For analysis, the walnut sample was ground and homogenized. The empty weighing bottle was pre-dried, cooled in a desiccator, and weighed on an analytical balance. Subsequently, 3–5 g of the sample was placed in the weighing bottle and weighed again.

The samples were dried in a drying oven at 105±2 °C for 6 hours. After drying, the weighing bottle was cooled in a desiccator and the final mass was recorded.

The moisture content was calculated using the following formula:

$$W = (m_1 - m_2) / (m_1 - m_0) \times 100$$

where:

W — moisture content, %;

m_0 — mass of the empty weighing bottle, g;

m_1 — mass of the weighing bottle and sample, g;

m_2 — mass of the weighing bottle and dried sample, g (7).

Figures 1-2. Process of determining the moisture content of walnut fruit.

Determination of ash content. Ash content was determined by the dry ashing method in accordance with GOST 25555.4–91.

For this purpose, 2–5 g of the ground walnut sample was placed into a previously weighed crucible. The sample was initially heated slowly on an electric hot plate for carbonization and then ashed in a muffle furnace at 500–550 °C for 3–6 hours.





After complete decomposition of the organic substances in the sample, resulting in the formation of white or gray ash, the crucible was cooled in a desiccator and weighed again.

The ash content was determined using the following formula:

$$X = (m_2 - m_0) / (m_1 - m_0) \times 100$$

where:

X — ash content, %;

m_0 — mass of the empty crucible, g;

m_1 — mass of the crucible and sample, g;

m_2 — mass of the crucible and ash, g (8).

Determination of total fat content. Fat content was determined by the Soxhlet extraction method in accordance with GOST 10857–64.

The walnut kernels were ground and homogenized, and an accurately weighed sample of 1–3 g was taken. The sample was placed in an extraction thimble. Then, 60–100 ml of solvent was added to a previously dried and weighed flask.

Extraction was carried out in a Soxhlet apparatus for 4–6 hours. Upon completion of the process, the solvent was evaporated, and the flask containing the remaining fat was dried at 105 °C to constant mass.

The fat content was calculated using the following formula:

$$\mathbf{X = (m_1 - m_0) / m \times 100}$$

where:

X — mass fraction of fat, %;

m_0 — mass of the empty flask, g;

m_1 — mass of the flask with fat, g;

m — sample mass, g (9).

Determination of acid value. The acid value of walnut oil was determined by a titrimetric method in accordance with GOST 5476–80.

For the analysis, 3–5 g of oil was dissolved in 50 ml of a neutralized alcohol–ether mixture. Phenolphthalein indicator was added to the solution, and titration was performed with a 0.1 mol/dm³ KOH solution until a light pink color persisted for 30 seconds.

The acid value was calculated using the following formula:

$$\mathbf{X = (V \times C \times 56.14) / m}$$

where:

X — acid value, mg KOH/g;

V — volume of KOH consumed, ml;

C — KOH concentration, mol/dm³;

m — sample mass, g (10).

Determination of peroxide value. The peroxide value was determined by the iodometric method in accordance with GOST 26593–85.

For the analysis, 1–5 g of oil was dissolved in 10 ml of chloroform. Subsequently, 15 ml of glacial acetic acid and 1 ml of a 10% potassium iodide solution were added. The mixture was kept under dark conditions for 1 minute.

After the reaction, 30 ml of distilled water was added, and the liberated iodine was titrated with a 0.01 N sodium thiosulfate solution. A control (blank) experiment was performed in parallel.

The peroxide value was calculated using the following formula:

$$\mathbf{PV = ((V_1 - V_0) \times C \times 1000) / m}$$

where:

PV — peroxide value, mmol O_2/kg;

V_1 — volume of titrant consumed for the sample, ml;

V_0 — volume consumed in the blank experiment, ml;

C — sodium thiosulfate concentration, mol/l;

m — sample mass, g (11).

All experiments were performed in at least three replicates, and the obtained results were prepared for processing as mean values.

RESULTS AND DISCUSSION

The results of the study showed that chitosan-based coatings significantly slow lipid oxidation in walnut fruits during storage. This was confirmed by the lower increase in peroxide value compared with the control group (10, 11).

The rate of increase in acid value was also reduced, indicating that the hydrolytic degradation of fats was slowed down (10). These results can be explained by the fact that the protective film formed by chitosan limits oxygen and moisture exchange.

Studies conducted by Sabaghi et al. also demonstrated that chitosan coatings reduce lipid oxidation and fungal development in walnut kernels (3). The study by Zhang et al. confirmed that biopolymer-based coatings improve the storage quality of walnuts (4).

Bonilla et al. demonstrated that coatings containing natural extracts significantly reduce oxidation processes (5). Composite coatings developed by Chen et al. were also reported to extend the shelf life of walnuts (6).

In studies by Hameed Al-Majed and Al-Zubaidy, chitosan coating was found to improve the chemical stability of walnut oil (1). Akhavan-Mahdavi et al. demonstrated that chitosan-based coatings improve oxidation resistance and microbiological safety (2).

Table 1. Quality parameters of walnut fruits stored in the shelled (whole) form for six months after treatment with biologically active substances

№	Variety	Treatment	Moisture content, %	Ash content, %	Fat content, %	Acid value, mg KOH/g	Peroxide value, mmol O_2/kg
1	Yubileniy	X3 – 0.1%	4.63	2.68	57.94	1.19	0.15
2	Yubileniy	FA–2 – 0.1%	4.90	2.18	61.46	1.00	0.39
3	Yubileniy	Control	4.51	2.01	66.95	0.94	0.23
4	Ideal	X3 – 0.1%	4.57	2.55	55.33	1.50	0.23
5	Ideal	FA–2 – 0.1%	4.64	2.22	56.47	1.58	0.40
6	Ideal	Control	4.94	3.28	57.51	1.01	0.30
7	Tonkoskorlupiy	X3 – 0.1%	4.15	1.64	63.08	0.88	1.33
8	Tonkoskorlupiy	FA–2 – 0.1%	4.47	2.41	56.02	1.09	0.44
9	Tonkoskorlupiy	Control	4.68	1.60	63.48	1.04	0.23

Table 2. Quality parameters of walnut fruits stored in the unshelled (kernel) form for six months after treatment with biologically active substances

№	Variety	Treatment	Moisture content, %	Ash content, %	Fat content, %	Acid value, mg KOH/g	Peroxide value, mmol O_2/kg
1	Yubileniy	X3 – 0.1%	4.70	2.00	60.72	0.83	0.23
2	Yubileniy	FA–2 – 0.1%	4.81	2.59	62.85	1.22	0.62
3	Yubileniy	Control	4.96	2.15	49.08	1.08	0.39
4	Ideal	X3 – 0.1%	4.25	1.86	55.39	1.04	0.31

№	Variety	Treatment	Moisture content, %	Ash content, %	Fat content, %	Acid value, mg KOH/g	Peroxide value, mmol O_2/kg
5	Ideal	FA–2 – 0.1%	4.93	3.00	57.46	0.92	0.23
6	Ideal	Control	4.18	2.13	54.04	1.09	0.23
7	Tonkoskorlupiy	X3 – 0.1%	3.86	3.02	59.96	1.14	0.30
8	Tonkoskorlupiy	FA–2 – 0.1%	4.18	2.85	55.51	0.96	0.23
9	Tonkoskorlupiy	Control	4.26	2.33	64.31	0.86	0.23

RESULTS

Comparative analysis of quality parameters of walnut fruits stored in shelled (whole) and unshelled (kernel) forms during storage

In the study, the physicochemical quality parameters of shelled and unshelled walnut fruits treated with biologically active substances (X3 and FA–2 at a concentration of 0.1%) were comparatively evaluated after six months of storage. The obtained results are presented in Table 1 for shelled walnuts and Table 2 for unshelled walnuts (kernels).

The overall analysis showed that the quality parameters of shelled walnut fruits remained considerably more stable compared with those of unshelled (kernel) samples. This can be explained by the fact that the shell acts as a natural protective barrier, limiting the effects of oxygen, moisture, and light.

Changes in moisture content

The moisture content of shelled walnut fruits ranged from 4.15 to 4.94%, whereas in the unshelled samples it varied from 3.86 to 4.96%, indicating a higher degree of variation. The more pronounced changes in moisture content in the kernel form are associated with its hygroscopic properties and direct contact with the external environment.

In the variants treated with the X3 coating, moisture content remained relatively stable, confirming the ability of the coating to form a semipermeable protective layer.

Ash content

The ash content in shelled samples ranged from 1.60 to 3.28%, whereas in unshelled samples it ranged from 1.86 to 3.86%. The higher and more variable ash content observed in unshelled walnuts can be explained by more rapid changes in the proportion of dry matter and relative changes in the concentration of mineral components.

Total fat content. The most significant differences were observed in terms of fat content. In shelled walnut fruits, fat content remained within the range of 55.33–66.95%, whereas in unshelled (kernel) samples this parameter ranged from 49.08 to 64.31%.

The considerable decrease in fat content in the control variants indicates that lipid oxidation and degradation processes occurred more rapidly in the walnut kernels. Shelled samples treated with X3 and FA–2 retained their fat fraction relatively better.

Acid value

The acid value in shelled samples ranged from 0.88 to 1.58 mg KOH/g, while in unshelled samples it ranged from 0.79 to 1.22 mg KOH/g. The slightly higher acid value observed in some shelled walnut variants may be associated with the continued occurrence of partial hydrolytic processes during long-term storage.

However, the overall trend indicates that the X3 coating was effective in limiting the formation of free fatty acids.

Peroxide value. The peroxide value in shelled walnut fruits varied from 0.15 to 1.33 mmol O_2/kg, with more pronounced oxidation processes observed in some varieties. In the unshelled samples, the peroxide value ranged from 0.23 to 0.62 mmol O_2/kg, indicating a moderate level of oxidation.

The high peroxide value observed in the shelled Tonkoskorlupiy variety (1.33 mmol O_2/kg) may be explained by the lipid composition of the variety and variations in coating effectiveness.

General discussion. The overall results indicate that quality parameters were better preserved in shelled walnut fruits compared with the unshelled (kernel) form. The shell acts as a natural protective barrier, slowing lipid oxidation, moisture exchange, and hydrolytic processes.

Among the biologically active coatings, the X3 treatment demonstrated greater stability compared with FA–2, particularly in terms of maintaining fat content and acid value. The FA–2 treatment was less effective in preventing an increase in peroxide value in some varieties.

Overall, the obtained results confirm that chitosan-based biologically active coatings are a promising technology for extending the storage life of walnut fruits and stabilizing their quality parameters.

CONCLUSION

The results of this study demonstrated that the use of chitosan-based biologically active coatings is an effective approach for stabilizing the quality parameters of walnut fruits (*Juglans regia L.*) during storage.

Shelled (whole) walnut fruits maintained their physicochemical quality parameters relatively more stably during storage compared with unshelled (kernel) samples. This can be explained by the ability of the shell to act as a natural protective barrier, limiting the effects of oxygen, moisture, and light.

In terms of moisture, ash, and fat content, as well as oxidation indicators (acid value and peroxide value), shelled samples generally underwent fewer changes, indicating a reduction in degradation processes during storage.

Chitosan-based biologically active coatings (X3 and FA–2 at 0.1%) limited lipid oxidation and hydrolytic degradation processes in walnut fruits to a certain extent. This contributed to maintaining the nutritional and technological quality of the product.

Among the coatings, the X3 treatment provided relatively greater stability than FA–2, particularly in maintaining fat content and acid value.

Although the FA–2 treatment was unable to completely prevent an increase in peroxide value in some varieties, it partially slowed the overall oxidation processes.

In future studies, investigating chitosan solutions at different concentrations in combination with storage temperature and relative humidity conditions would provide an opportunity to evaluate the practical effectiveness of biologically active coatings in greater depth.

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