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OBTAINING AND CHARACTERIZATION OF THE CURCUMIN COMPLEX WITH XYLAN

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N. Cherno, Doctor of Technical Sciences, Professor
K. Naumenko, PhD Tech. Science, Associate Professor
K. Yershova, postgraduate student
Department of food chemistry, expertise and biotechnology
Odesa National University of Technology
112, Kanatna Str., Odesa, Ukraine, 65039

Correspondence:

K. Yershova
E-mail: kate21101997@gmail.com

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Abstract. Polyphenolic compounds of plant origin are known for their positive effects on the human body due to their antioxidant, anti-inflammatory, antiviral, etc. effects, but they have limitations in terms of use as preventive agents due to their low bioavailability, which is caused by their chemical instability and insolubility in water. One of the ways to increase the bioavailability of polyphenols is to use various nanocarriers (liposomes, micelles, nanoemulsions, metal complexes, nano-gels, etc.) The most commonly used biopolymers in the development of systems for stabilizing and increasing the solubility of polyphenolic compounds are biocompatible and biodegradable. In this context, non-starch polysaccharides are attracting increasing attention of researchers due to their gastrointestinal resistance, most of them are able to provide controlled release, targeted delivery and water solubility. One of the polyphenolic compounds with low bioavailability is curcumin, the stability of which can be increased by complexation with certain polysaccharides. This article is devoted to the substantiation of the possibility of increasing the stability of curcumin by its complexation with xylan. Xylan was obtained by alkaline extraction from corn cobs. The complex of curcumin with xylan was obtained by a pH-controlled method. The rational conditions of complexation were substantiated. The formation of the curcumin-xylan complex and the preservation of the curcumin structure after complexation were proved by spectroscopic methods. It is shown that curcumin in the complex has higher thermo- and photostability compared to free curcumin. Complexation with xylan has been shown to increase the stability of curcumin in vitro conditions imitating the human gastrointestinal tract. The curcumin-xylan complex can be used as a physiologically functional ingredient in food production and as a dietary supplement.

Key words: dietary supplement, food functional ingredient, curcumin, xylan.

Introduction. Formulation of the problem

In recent years, the creation and development of dietary supplements and functional food ingredients based on phenolic compounds of plant origin has become relevant. Plant polyphenols have a positive impact on human health due to their antioxidant, anti-inflammatory, antitumor, and antiviral effects. However, they have limitations in terms of use due to low bioavailability and poor water solubility [1-3].

The physiological conditions of the body lead to the degradation of phenolic compounds in the gastrointestinal tract (GIT), with only about 5–10% of their total amount being absorbed in the small intestine [2-7].

The bioavailability of polyphenols is increased by blocking metabolic processes and using various nanocarriers (liposomes, micelles, nanoemulsions, nano-gels, solid dispersions, metal complexes, etc.) The use of such approaches has demonstrated different levels of absorption enhancement. Some of them have limitations in terms of application due to the use of non-food ingredients, poor biocompatibility and insufficient stability, high material costs, complex synthesis process, and other disadvantages [8].

According to the previous, nanoparticles of natural origin attract increased attention of scientists. Compared to analogues based on metals, metal oxides and synthetic polymers, they are biodegradable, biocompatible, non-immunogenic, non-toxic, and stable [8-11].

Polysaccharides are most often used for encapsulation of polyphenolic compounds. It has been shown that most of them are capable of providing controlled release of polyphenols and their targeted delivery, as well as water solubility. Polysaccharides protect these compounds from degradation in the

stomach and upper digestive tract, and fermented in the colon, where most bioactive substances are absorbed [10,12].

Analysis of recent research and publications

For many centuries, plant polyphenols have been used to prevent and treat various diseases, improve physiological processes in the body and increase immunity. Curcumin is one of these compounds. Its natural source is turmeric (*Curcuma longa L.*). It has been widely used as a medicine in Asian countries since ancient times. The physiological effects of turmeric are primarily associated with curcuminoids – polyphenols contained in its roots in the amount of 3–5% and responsible for color. The main biologically active component of curcuminoids is curcumin (IUPAC name for curcumin is 1,7-bis(4-hydroxy-3-methoxyphenyl)-1,6-heptadien-3,5-dione) – 75–85%, and the rest is demethoxycurcumin (10–20%) and bis-demethoxycurcumin (3%) [13-15].

Curcumin is readily soluble in polar organic solvents (ethanol, methanol, dimethyl sulfoxide, etc.) and practically insoluble in water. The curcumin diketogroup has keto-enol tautomerism, so curcumin exists in keto- and enol tautomeric forms (Fig. 1). The diketo form predominates in crystalline curcumin or in acidic and neutral solutions, the enolic form is present in alkaline conditions. At pH < 1, curcumin has a red color, in the range of pH 1–7 – yellow, and at pH 8–9 and more – red [16].

Studies of curcumin have shown its safety and effectiveness as a preventive and therapeutic agent. However, its chemical instability, water insolubility, and low bioavailability limit the use of curcumin for pharmacological purposes [17-18].

Curcumin demonstrates a large number of pharmacological properties: antitumor [18,19], antioxidant [21,22], anti-inflammatory [21,23], antidiabetic [21], antimicrobial [24], antiviral [24,25], immunomodulatory [26], and others.

Increasing the bioavailability of curcumin has become a challenge for many researchers. Curcumin immobilization on various carriers is used to increase its bioavailability. It is considered that the most perspective curcumin nanosystems are based on natural biopolymers. They have several advantages and are a common component of food rations. This simplifies their use in the food industry in the form of dietary supplements, emulsifiers, foam and gel formers, antioxidants, etc. Polysaccharides are the most commonly used biopolymer matrices.

Polysaccharides are widely used in the food and pharmaceutical industries due to their unique physical and chemical properties. This has motivated scientists to intensify research on the use of polysaccharides as components of nanosystems for stabilizing biologically active substances (BAS) with low bioavailability. Such systems are used to increase the bioavailability of curcumin, in particular, because it has high gastroresistance. The stability of polysaccharides contributes to its targeted delivery to the colon, where both components of the nanosystem are fermented by the intestinal microbiota [8,10,11].

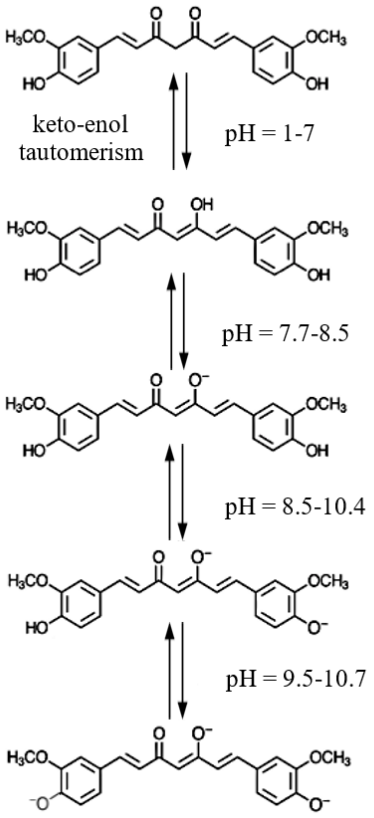

Fig. 1. Forms of curcumin depending on pH conditions [16]

The most popular component used in the formation of curcumin-based nanoparticles is starch. It is the most widely available polysaccharide with a significant source of raw materials and low cost. The size of starch particles can be controlled by its chemical or enzymatic hydrolysis, nanoprecipitation, etc. Starch is also modified to increase the efficiency of its use as a matrix that ensures the release of curcumin in the stomach and small intestine. However, such systems are not able to perform targeted delivery to the colon [11,27-29], as starch is destroyed by the enzyme systems of the GIT.

Cyclodextrins (CDs) are products of the cyclization of starch oligomeric fragments. It is known that CDs can be used to enhance intestinal absorption due to their properties of solubilization and stabilization of BAS. The authors of [30] studied the intestinal absorption of curcumin included in different CDs by the closed-cycle *in situ* method using male rats. Improved oral absorption of curcumin was shown by α-CD and dimethyl-β-CD.

Chitosan is a cationic polysaccharide that has mucoadhesive properties, in particular to the surface of

the gastrointestinal mucosa, which has a negative charge. This approach is used to target specific organs or cells and prolong the residence time of polyphenols in the intestine. Positively charged chitosan is often used together with negatively charged polysaccharides (e.g., pectin, sodium alginate, etc.) to produce nanoparticles [1,11,31,32]. Seung Won Hwang and Jae Sup [33] developed curcumin-loaded chitosan-pectin microparticles by polymeric microencapsulation using two methods. One of them included the preparation of curcumin-chitosan microparticles and their subsequent coating with pectin. According to the second method, chitosan-pectin microparticles were first formed, which were then loaded with curcumin. In the studies, Mg^{2+} and Ca^{2+} were used in parallel to crosslink pectin macromolecules. Microparticles obtained by the first method showed slower curcumin release profiles at pH 1.2 and pH 6.8 than those obtained by the second method. This is probably due to the fact that the first method produces systems in which curcumin is protected by a pectin layer, which slows down its release. In contrast, particles obtained by the second method contain curcumin on the surface of the system. This increases its accessibility to environmental factors, which contributes to its desorption from the surface. It should be noted that the particles obtained using Mg^{2+}, showed a slower release rate compared to particles made with Ca^{2+}.

Study [34] described the preparation of polyelectrolyte complexes of curcumin with chitosan, gellan, and with or without ι-carrageenan. The efficiency of curcumin encapsulation increased with decreasing crosslinking density and the amount of ι-carrageenan. The kinetics of curcumin release was studied in solutions with pH 2, 6.8, and 7.4 at 37 °C. The highest efficiency of curcumin release from all samples was at pH, the value of which is similar to the conditions of the colon. According to the authors, this determined the suitability of the obtained complexes as means of targeted delivery of curcumin to the colon.

The use of pectin aerogels and pectin aerogels coated with chitosan to increase the bioavailability of curcumin has been described [35]. Chitosan was used to slow down the dissolution of pectin and, as a result, the release of curcumin. Pectin aerogels *in vitro* studies released curcumin after 3 hours. At the same time, chitosan-coated pectin aerogels demonstrated significantly higher efficiency, as they prolonged the release of curcumin for up to 24 hours.

In another study [36], deesterified citrus pectin was used to increase the bioavailability of curcumin. Gastroresistant curcumin-pectin calcium gel balls were prepared. *In vitro* experiments showed that the gel coatings promote the transit of curcumin to the colon, preventing its premature release.

In the study [37], curcumin was encapsulated in fig pectin by vacuum spray drying. It was shown that 95 % of the total amount of curcumin was released in 24 h into the conditions imitating liquids of GIT. This method of curcumin encapsulation allowed to keep the antioxidant activity stable for 6 months. The authors concluded that it is possible to use encapsulated curcumin for targeted delivery to the colon.

The authors of [38] obtained a curcumin-loaded octenylsuccinate glucomannan nanoemulsion. It was tested in simulated gastric and intestinal liquids, and native curcumin was used for comparison. Stability and release rate were higher in simulated intestine than stomach conditions, but significantly lower compared to free curcumin. Similar results were obtained *in vivo* studies. It is concluded that the obtained nanoemulsion has the potential as a means for targeted delivery of curcumin to the colon. In addition, it was found that the thermal and storage stability of curcumin in the nanoemulsion was improved compared to free curcumin.

Study [39] demonstrated the possibility of using carboxymethylglucomannan nanogels with chitosan crosslinked with 1-ethyl-3-(3-dimethylaminopropyl)/N - hydroxysuccinimide and without it for encapsulation of curcumin. It has been shown that their application helps to prolong its release in simulated conditions of the GIT.

The authors of study [40] tried to immobilize curcumin on water-soluble dietary fibers of oats, corn, fenugreek, and celery. However, this attempt did not lead to the desired results, as the obtained substances were not stable enough.

In study [41] authors describe the application of sodium alginate to stabilize curcumin. Alginate microspheres filled with curcumin were obtained by ionic gelation, using $CaCl_2$ as a crosslinking agent. *In vitro* studies have confirmed the stability of the obtained formulations to low pH values and the ability to overcome the gastric barrier, ensuring the absorption of curcumin in the small intestine.

Cellulose nanofibers from lemongrass waste were used as a potential carrier of curcumin to slow its release in the GIT. The degree of its release was determined at different pH values of the solutions. The results showed that enzymatically produced cellulose nanofibers are an effective matrix for curcumin immobilization, which contributes to its controlled release [43].

Studies [44] demonstrate the use of cellulose nanocrystals for the encapsulation of curcumin by a pH-controlled method, which leads to an increase in its stability and solubility, and the encapsulation efficiency was more than 90%. The curcumin-loaded cellulose nanoparticles showed stability at pH 3.0–8.0, as well as an increase in stability under conditions simulating the human GIT by more than 2.5 times compared to free curcumin.

The above data indicates the significant potential of non-starch polysaccharides as agents that can be used to stabilize curcumin. The most widely found polysaccharide, which ranks second in terms of abundance after cellulose, is xylan. The field of

application of this biopolymer is quite limited, and its properties as a biocompatible and biodegradable compound have not yet been evaluated. Therefore, it is important to study the stability of curcumin using this polysaccharide as a structural and biocompatible component of the curcumin-xylan system.

The purpose of the study was to determine the conditions for obtaining and to characterize the water-soluble complex of curcumin and xylan.

The tasks were identified to achieve this goal:

- to substantiate the conditions for obtaining the complex and to characterize it;
- to determine the thermal and photostability;
- to examine the transformations of the complex in conditions imitating the GIT.

Research materials and methods

Curcumin (Curcumin for synthesis, Sigma Aldrich, Germany) and xylan from corn cobs (this raw material contains 60% xylan of the total amount of polysaccharides) were used in the study. The xylan was obtained by its alkaline extraction from the feedstock [45,46].

The monosaccharide composition of xylan hydrolysates was determined by high-performance liquid chromatography [47] using chromatograph Agilent 1100 (Agilent Technologies, Germany).

The degree of degradation (DD) of curcumin in alkaline solution was determined at different concentrations of NaOH. Curcumin was kept for a certain time in an alkaline solution, then it was neutralized with 0.1 n HCl. The precipitate was separated by centrifugation, then it was dissolved in ethanol and the amount of curcumin in the precipitate was determined by photoelectrocolorimetric method. The DD was calculated by the formula:

$$\mathrm{DD} = \frac{m}{m_{total}} \times 100\ \%,$$

where m – amount of curcumin in the precipitate;

m_{total} – total amount of curcumin.

The polysaccharide component was dissolved in an alkaline medium (0.1 n NaOH) to obtain the complexes, after curcumin was added and dissolved rather quickly. Then the pH of the mixture was adjusted to 7 with 1 n HCl, curcumin, which did not participate in complexation, precipitated and was separated by centrifugation. In the experiments, the concentration of alkaline xylan solutions and the mass of curcumin were varied. The formed water-soluble complex of curcumin with xylan was obtained by freeze-drying the liquid phase. The amount of curcumin in the complex was determined by photoelectrocolorimetric method. Chloroform was used to extract curcumin from the complex [48]. The efficiency of complexation (E) was calculated by the formula:

$$\mathrm{E} = \frac{m_e}{m_{total}} \times 100\ \%,$$

where m_e – amount of curcumin extracted with chloroform;

m_{total} – total amount of curcumin.

Fourier transform infrared (FTIR) spectroscopy was performed by FT-IR Frontier Spectrometer (Perkin-Elmer, USA) in the range of 4000–400 cm^{-1} [49]. For this purpose, tablets were formed, which were a mixture of the sample and KBr. The spectra in the range of 300-500 nm were recorded on a UV-1100 spectrophotometer in cuvettes with a layer thickness of 10 mm.

The structure of curcumin before and after its stabilization by the polysaccharide component was compared as follows [48]: curcumin from the complex was extracted with chloroform, and free curcumin was dissolved directly in chloroform at a concentration corresponding to the amount of curcumin bound to xylan. After that, the spectra of the obtained samples were taken and varied in the range of 300-500 nm using a UV-1100 spectrophotometer in cuvettes with a layer thickness of 10 mm.

The thermostability was determined by keeping the complex and free curcumin at different temperatures and varying the time of exposure, after the content of undegraded curcumin was determined [50].

The photostability of the complex was determined by irradiating it with ultraviolet light at a wavelength of 365 nm. An ultraviolet lamp with a power of 48 W was used as an radiation source, and the experiment was carried out for 60 min, recording the residual amount of curcumin at certain intervals. In parallel, free curcumin was studied under the same conditions [50].

The stability of the complexes was determined in media with pH values corresponding to the pH of gastrointestinal fluids [34]. Solutions of 0.02 n HCl and $NaHCO_3$ were used for the experiments. In parallel, experiments were performed on free curcumin.

Results of the research and their discussion

The following data are presented to determine the feasibility of increasing the solubility and stability of curcumin, as factors determining its bioavailability, by complexation with xylan, the most common polysaccharide component of hemicelluloses. Xylan was obtained by alkaline extraction from corn cobs. The results of the study of its monomeric composition indicate that it is structured from xylose, arabinose, glucose, galactose, and glucuronic acid in the ratio Xyl:Ara:Glu:Gal:GlucA 51.4:17.0:12.3:10.0:3.0, which determines it to the category of heteroxylans. The presence of glucuronic acid in the monomeric composition allows it to be classified as an acidic heteroxylan.

The conditions of the experiment on the formation of a curcumin-xylan complex were determined based on information about the physicochemical properties of curcumin, namely its solubility in aqueous medium at different pH values, so it was necessary to determine the

rational limits of the concentration of NaOH solution, which will be used for complexation. It is known that curcumin is prone to degradation in an alkaline medium, but data on the degree of its destruction in these conditions are contradictory [50-52]. Thus, the authors of [51] state that at pH 10, less than 10 % of curcumin is retained after 1 hour. At the same time, Zhiyu Li et al. indicate that according to the results of their research, under the same conditions, the degree of curcumin degradation was more than 50%, and at pH 12 – almost 80% of its total amount in 1 hour [50]. According to the authors of the patent US 4999205A "Curcumin complexed on water-dispersible substrates" (Kalamazoo Holdings Inc., 17.08.1989), the degradation of curcumin can be approximately 7% per hour in an alkaline environment. In order to determine the degree of curcumin degradation, an experiment was performed in which a sample of curcumin was kept in an alkaline solution by varying the concentration of NaOH and then the content of undegraded curcumin was determined (Table 1).

Table 1 – Degree of degradation of curcumin in alkaline solution

№	Processing time, min	DD in 0.1 n NaOH, %	DD in 0.05 n NaOH, %
1.	5	4.6	4.6
2.	10	5.4	5.4
3.	20	6.8	6.3
4.	40	8.4	7.2
5.	60	9.0	8.5
6.	120	10.8	10.2
7.	180	15.2	14.8
8.	240	21.0	18.7
9.	300	27.2	21.1

According to the results of the experiment, in an alkaline medium with a pH of 14 (0.1 n NaOH and 0.05 n NaOH), a gradual degradation of curcumin occurs within 300 minutes. In the first 10 minutes, the degree of degradation of curcumin is the same in both alkali solutions, and then it gradually increases: in 0.1 n NaOH it becomes 0.5-1 % higher compared to 0.05 n NaOH at a certain point in time, after 4 hours the difference reaches more than 2 %, and after 5 hours – more than 6 %. Based on the data presented in Table 1, for further experiments on the complexation of curcumin with xylan, a 0.1 n NaOH solution was used, since curcumin dissolved faster in it compared to a 0.05 n solution, and the degree of its degradation was the same.

The complex of curcumin with xylan was obtained by dissolving curcumin in an alkaline solution of xylan with 0.1 n NaOH solution, after the pH of the mixture was immediately adjusted to 7 (the pH value at which free curcumin becomes insoluble and precipitates) and curcumin remains in the supernatant (liquid phase) as a soluble complex with xylan. By varying the concentrations of each of the components and their ratios, data illustrating the degree of curcumin complexation with xylan were obtained and they are shown in Table 2. It should be noted that the data characterize the composition of soluble complexes, since partial precipitation (probably an insoluble curcumin-xylan complex or a mixture of xylan and free curcumin) occurred during the study. The complex after lyophilization was well soluble in water and insoluble in ethanol and chloroform, in contrast to free curcumin.

Therefore, as a result of the complexation of curcumin with xylan, a highly efficient immobilization of curcumin to xylan (about 90 %) can be achieved. An increase in the concentration of xylan led to the formation of a precipitate and a decrease in the content of curcumin in the soluble complex. The complex obtained at the minimum concentration of curcumin at a ratio of curcumin : xylan 1 : 3 showed the highest efficiency of complexation, but the use of such concentrations of components is technologically unjustified, and ratio curcumin : xylan 2 : 1 and curcumin concentration 1.0 mg/cm3 are the most rational conditions in the context of this issue. If the purpose of using a complex of curcumin and xylan is consuming the maximum amount of water-soluble curcumin, then it is technologically feasible to use a 2 : 1 ratio of curcumin : xylan with a concentration of curcumin of 1.0 mg/cm3, since in this case the resulting product will contain more than 50 % curcumin in its composition. To obtain a product with a more pronounced prebiotic effect, which will contain curcumin in an amount of more than 20 %, it is more advantageous to use a ratio of 1...2 : 1...3. Thus, it is important to note that, based on the results obtained, the composition of the complex can be controlled by changing the conditions for obtaining the complex.

The obtained curcumin-xylan complex was characterized by spectroscopic methods. Figure 2 shows the Fourier transform infrared spectra of curcumin (2.1), xylan (2.2), the complex (2.3), and a physical mixture of curcumin and xylan (2.4) in a mass ratio corresponding to the ratio of curcumin and xylan in the complex.

The spectra of curcumin (Fig. 2.1) shows characteristic absorption bands at 3510 cm-1 corresponding to the valence vibrations of phenolic hydroxyls, at 1628 cm^{-1} to the C=O bond, at 1510 cm^{-1} to the C=C bonds of aromatic structures, and at 1429 cm^{-1} to the C–C-valent aromatic skeletal vibrations combined with C–H asymmetric vibrations (valence vibrations of the benzene ring). The peak at 1283 cm^{-1} refers to the vibrations of the carbonyl group –CO, while the peaks at 963, 857, and 815 cm^{-1} characterize the C–H vibrations of the alkene group. These bands were observed by other authors in the spectra of curcumin and its complexes with biopolymers [53-55].

Table 2 – Efficiency of curcumin complexation with xylan depending on the conditions

Mass ratio curcumin : xylan	Curcumin concentration, mg/cm^3	Xylan concentration, mg/cm^3	Efficiency of complexation (E), %	Amount of curcumin in the complex, %.
2 : 1	0.25	0.13	26.5	34.6
	0.50	0.25	42.1	46.7
	1.00	0.50	54.5	**52.2**
1 : 1	0.25	0.25	76.0	43.2
	0.50	0.50	64.4	39.2
	1.00	1.00	65.3	39.5
1 : 2	0.25	0.50	78.6	28.2
	0.50	1.00	73.0	26.7
	1.00	2.00	67.8	25.3
1 : 3	0.25	0.75	**86.5**	22.3
	0.50	1.50	80.4	21.1
	1.00	3.00	76.5	20.3
1 : 5	0.25	1.25	78.0	13.5
	0.50	2.50	**85.6**	14.6
	1.00	5.00	73.2	12.8
1 : 7	0.25	1.75	75.2	9.7
	0.50	3.50	80.5	10.3
	1.00	7.00	68.0	8.9
1 : 10	0.25	2.50	74.3	6.9
	0.50	5.00	77.8	7.2
	1.00	10.00	60.1	5.7
1 : 15	0.25	3.75	73.0	4.6
	0.50	7.50	69.6	4.4
	1.00	15.00	53.4	3.4
1 : 20	0.25	5.00	72.3	3.5
	0.50	10.00	59.5	2.9
	1.00	20.00	48.1	2.3

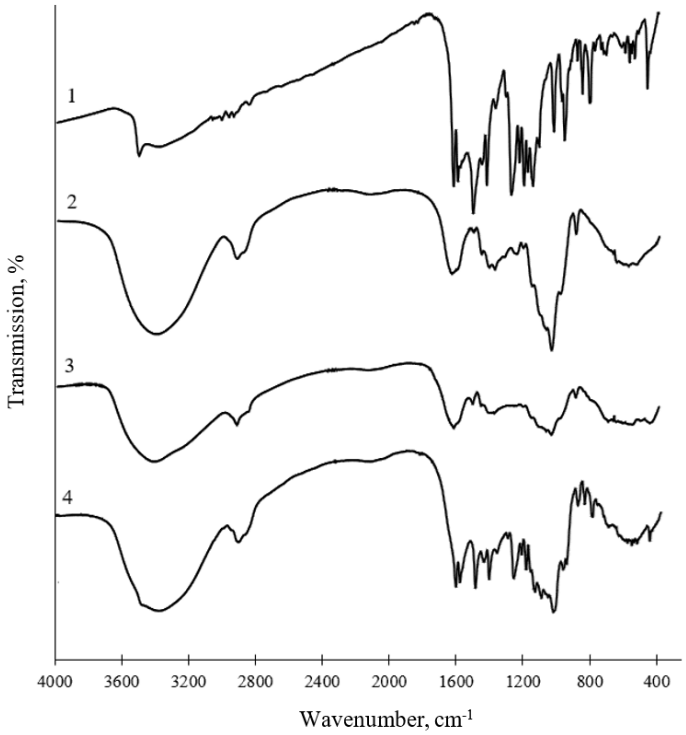

Fig. 2. FTIR-spectra for free curcumin (1), xylan (2), complex (3) and mixture of curcumin and xylan (4)

The bands in the FTIR-spectra of the polysaccharide component sample (Fig. 2.2) are typical for heteroxylan: a characteristic peak of a wide band of the hydroxyl group at 3402 cm^{-1} and a low absorption in the range of 3000-2800 cm^{-1} corresponding to the C–H valence vibrations of CH_2, CH_3, and CH groups with a maximum absorption at 2925 cm^{-1}, the peak at 1642 cm^{-1} is characteristic of the H–O–H deformational vibrations of crystallization water, the peaks at 1417 and 1384 cm^{-1} are for the symmetrical valence vibrations of the carboxyl group of glucuronic acid and the deformational vibrations of the terminal CH_3 in acyl groups respectively. The pronounced band at 897 cm^{-1} is determined by the β-glycosidic bond between xyloses, and the peak at 1045 cm^{-1} is explained by the valence vibrations of the hydroxyl group bonding to the C–OH carbon atom. The weak band at 1510 cm^{-1} may be due to the presence of residual lignin in the xylan sample [56,57].

The IR-spectra of the complex (Fig. 2.3) has a band in the range of 3700-3000 cm^{-1} corresponding to O–H valence vibrations with a maximum at 3419 cm^{-1}, the peak at 2925 cm^{-1} corresponds to C–H valence vibrations of CH_2, CH_3, and CH groups, the peaks at 1417 and 1384 cm^{-1} are characteristic of the carboxyl group of glucuronic acid and terminal CH_3 in acyl groups, respectively, the peak at 1043 cm^{-1} corresponds to the H–O–H bond. All of these maxima correspond to the absorption maxima in the xylan spectra. The absorption maximum at 1627 cm^{-1} corresponds to the C=O bond, which is characteristic of curcumin.

The spectra of the physical mixture of curcumin and xylan (Fig. 2.4) contains characteristic bands of both curcumin and xylan; for example, in the curcumin spectra, the peak at 3510 cm^{-1} is explained by the vibration of the OH group in the benzene ring, and the peaks at 3402 cm^{-1} and 1044 cm^{-1} are typical for xylan. The FTIR-spectra of the curcumin-xylan complex showed certain changes in their character compared to the spectra of the mixture, in particular, the peak at 3510 cm^{-1} and most of the curcumin peaks completely cover the spectra of xylan. This could indicate that curcumin does not exist in the free form. In addition, the peak at 3402 cm^{-1} (OH vibration) has a changed configuration (shifted to 3419 cm^{-1}) compared to the spectra of the mixture of curcumin and xylan, which could be an indication of a change in the interaction between curcumin and xylan, i.e. complexation due to hydrogen bonds. An increasing absorption intensity at 3419, 1628, and 1045 cm^{-1} showed that hydrogen bonds led to the formation of a complex of curcumin with xylan. [34,43,58,59].

Figure 3 shows the UV-Vis absorption spectra of the complex in the wavelength range of 300–500 nm.

The complex of curcumin with xylan (aqueous solution) has a single absorption maximum at 410 nm, which differs from the absorption maxima of curcumin in ethanol – 430 nm and in chloroform – 420 nm. It is obvious that it is impossible to compare these data, because, on the one hand, the complex is insoluble in organic solvents, and on the other hand, curcumin is insoluble in water. Xylan had no absorption maxima in the specified range.

One of the approaches used to determine the identity of the structure of free curcumin and curcumin in the complex is to compare their absorption spectra. In Fig. 4 shows the spectra of curcumin extracted from the complex and free curcumin.

It was found that the absorption peak of curcumin did not change, and the absorption intensity almost did not change as a result of complexation with xylan. This indicates that the structure of curcumin is preserved after complexation.

Thus, the formation of a complex involving curcumin and xylan, as well as the preservation of the structure of curcumin after complexation with xylan, was proved by spectroscopic methods.

Curcumin has low thermal and photostability. For example, the authors of [50] report that free curcumin degraded by 15% at 0°C in 7 h, and at 80–100°C – almost completely during the same period. The results of the study of the thermostability of curcumin in the complex in comparison with that of free curcumin are shown in Fig. 5.

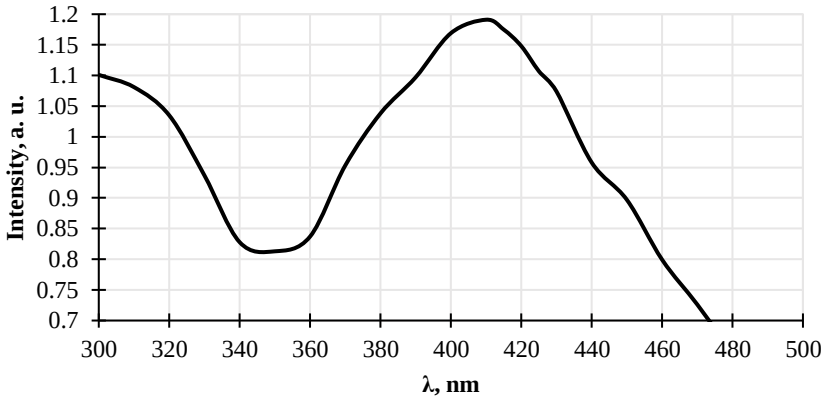

Fig. 3. UV-Vis absorption spectra of curcumin complex (in water)

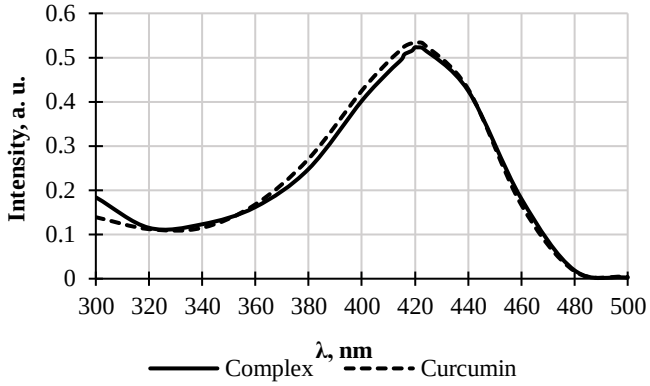

Fig. 4. UV-Vis absorption spectra of native curcumin and curcumin, extracted from the complex (in chloroform)

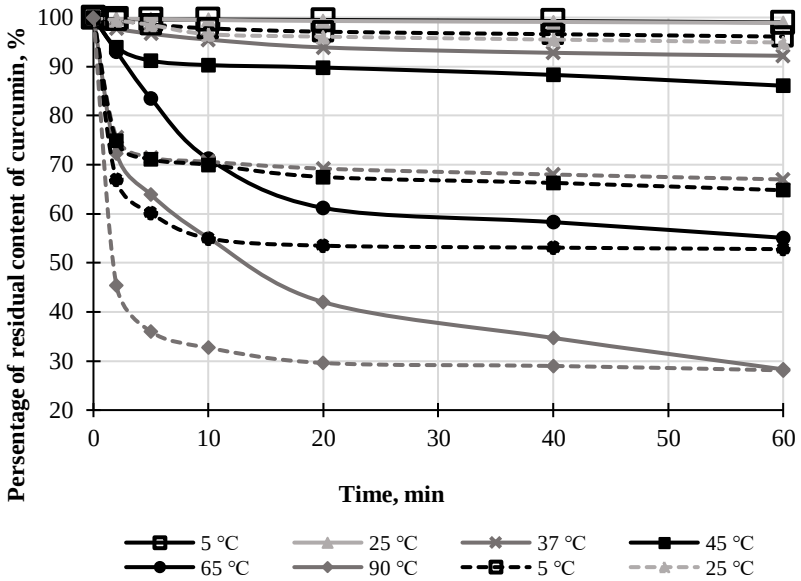

Fig. 5. Thermostability curves of free curcumin and curcumin in the complex at different temperatures

The data show that at 5°C and 25°C, the curcumin included in the complex is completely stable, but free curcumin decreased by 5 % after an hour. At 37°C and 45°C, at the end of the experiment, the amount of curcumin in the complex remained 25 % and 21% higher, respectively, compared to free curcumin under the same conditions. At 65°C, 2.5 times less curcumin was destroyed in the complex during the first five minutes compared to native curcumin. At the same time, prolongation of the exposure period led to the degradation of about half of the curcumin in both cases. The most obvious evidence of the increase in the thermostability of curcumin in the complex is provided by the experimental data obtained as a result of processing the samples for 2 min at 90°C, when, compared to native curcumin, more than 27% curcumin was retained in the curcumin-xylan complex. After 5 minutes at 90°C, almost 2 times more curcumin was retained in the complex, although more than 70% was destroyed in both samples after an hour. Thus, the stabilization of curcumin on the xylan matrix contributes to the increased thermal stability of curcumin compared to free curcumin, but an increase in temperature adversely affects the complex, which should be taken into account when determining the technological parameters and storage conditions.

Figure 6 shows curves characterizing the dependence of the amount of free curcumin and curcumin in the complex on the duration of UV radiation treatment.

As can be seen from Figure 6, in the first minutes of the experiment, intensive destruction of free curcumin occurs, unlike that protected by the xylan matrix. 10 minutes after the start of the experiment, the amount of curcumin in the complex remained 1.6 times more compared to native curcumin under the same conditions, but it continued to decrease over time. After an hour of UV treatment, 16.6 % more curcumin remained in the complex than free curcumin. These data indicate an increase in the photostability of curcumin in the complex compared to free curcumin, but illustrate its rather high vulnerability to radiation and indicate that

this factor should be taken into account during the storage of the complex.

Studies were conducted to simulate the conditions of the GIT to predict the bioavailability of the obtained complex in comparison with native curcumin. It has been shown that under these conditions, free curcumin and curcumin in the complex gradually degrade, but free curcumin degrades at a higher rate (Fig. 7). For example, the degree of its destruction in two hours (conditional period of stay in the stomach, pH 2.0) is 31%, while curcumin protected by the xylan component is destroyed only by 5.7%. The next three hours, which correspond to the conditional period of stay in the small intestine at pH 6.8, show a rapid destruction of free curcumin by still 52.4%. Curcumin in the complex under such conditions was degraded by more than 42% of the original amount, but almost 52% of it was retained. Thus, the amount of curcumin in the complex that can theoretically reach the colon is more than 3 times higher than that of free curcumin. After another 4 hours (conditional residence time of curcumin in the colon, pH 7.4), free curcumin remained almost 3 times less than curcumin in the complex.

The complexation with xylan is advisable because it significantly increases the stability of curcumin in conditions that simulate the environment of the stomach and intestines. Thus, more than 50 % of curcumin in the complex can probably reach the large intestine *in vitro*, where xylan can be metabolized to short-chain fatty acids, and curcumin can be metabolized to tetrahydrocurcumin, hexahydrocurcumin, and octahydrocurcumin, which have physiological effects similar to curcumin. Thus, the results of this experiment show an increase in the stability of curcumin due to complexation with xylan.

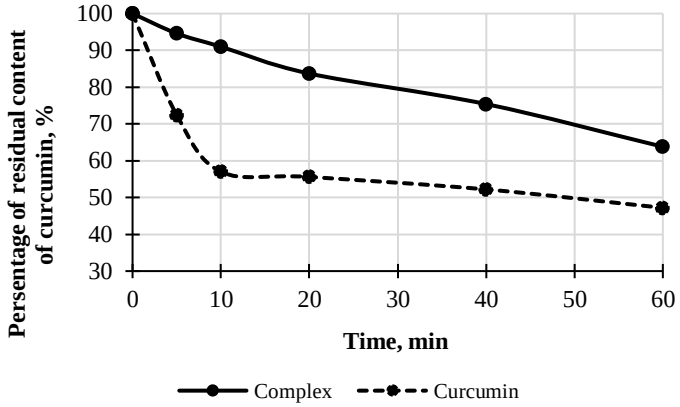

Fig. 6. Curves of photostability of free curcumin and curcumin in the complex under UV treatment

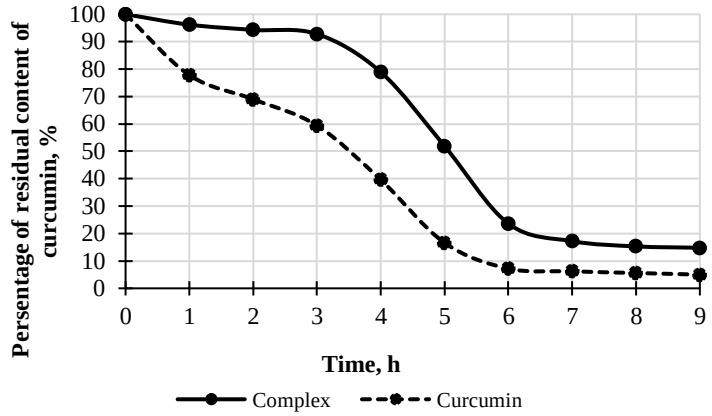

Fig. 7. Stability curves of free curcumin and curcumin in the complex under conditions simulating the gastrointestinal tract

Conclusion

The possibility of using xylan for the purpose of complexation with curcumin, which leads to an increase in the stability and solubility of curcumin – properties that determine the degree of its bioavailability has been shown. Complexation has been shown to increase the stability of curcumin under the influence of radiation and temperature. A significant increase in its stability *in vitro* conditions simulating the human GIT was found. Taken together, the above allows us to predict an increase in the bioavailability of curcumin as a result of its complexation with xylan and determines the prospects for its use as a dietary supplement or functional and physiological ingredient.

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ОТРИМАННЯ ТА ХАРАКТЕРИСТИКА КОМПЛЕКСУ КУРКУМІНУ З КСЛИЛАНОМ

Н. Черно, д-р техн. наук, професор, *E-mail*: cherno.onaft@gmail.com
К. Науменко, канд. техн. наук, доцент, *E-mail*: shapkinak@gmail.com
К. Єршова, аспірант, *E-mail*: kate21101997@gmail.com
Кафедра харчової хімії, експертизи та біотехнологій
Одеський національний технологічний університет
вул. Канатна, 112, м. Одеса, Україна, 65039

Анотація. Поліфенольні сполуки рослинного походження відомі своїм позитивним впливом на організм людини завдяки їхній антиоксидантній, протизапальній, противірусній та ін. діям, але вони мають обмеження щодо використання у якості профілактичних засобів через низький рівень біодоступності, яка зумовлена їхньою хімічною нестабільністю та нерозчинністю у воді. Одним із шляхів підвищення біодоступності поліфенолів є використання різноманітних наноносіїв (ліпосоми, міцели, наноемульсії, комплекси з металами, нано-гелі та ін.). Найбільш часто при розробленні систем для стабілізації та підвищення розчинності поліфенольних сполук використовують біополімери, які відрізняються біосумісністю та біоразкладністю. У цьому контексті некрохмальні полісахариди привертають все більшу увагу дослідників через їхню гастрорезистентність, більшість з них здатні забезпечувати контрольоване вивільнення, адресну доставку та розчинність у воді. Однією з поліфенольних сполук з низьким рівнем біодоступності є куркумін, стабільність якого може бути підвищена за рахунок комплексоутворення з деякими полісахаридами. Дана стаття присвячена обґрунтуванню можливості підвищення стабільності куркуміну за рахунок його комплексоутворення з ксиланом. Ксилан отримували шляхом лужної екстракції зі стрижнів кукурудзи. Комплекс куркуміну з ксиланом отримували pH-керованим методом. Обґрунтовано раціональні умови комплексоутворення. Спектроскопічними методами доведено утворення комплексу куркумін-ксилан, а також збереження структури куркуміну після комплексоутворення. Показано, що куркумін у складі комплексу, має більшу термо- та фотостабільність у порівнянні з вільним куркуміном. Доведено, що комплексоутворення з ксиланом збільшує стабільність куркуміну в умовах in vitro, що імітують шлунково-кишковий тракт людини. Комплекс куркумін-ксилан можливо використовувати як фізіологічно-функціональний інгредієнт у виробництві харчових продуктів та як дієтичну добавку.

Ключові слова: дієтична добавка, харчовий фізіологічно-функціональний інгредієнт, куркумін, ксилан.