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Production, Properties and Swelling of Copper-Pectic Gel Particles in an Artificial Gastroenteric Environment

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Abstract

The purpose of the this research was to obtain and study the properties of copper-pectic gel particles (CuPGPs) obtained from aqueous solutions of apple pectin (AP) in the concentration range of 1%-5% in the presence of Cu^{2+} ions.

Methods and Results: We used commercial AP AU701 (Herbstreith & Fox KG, Germany). CuPGPs were obtained from aqueous solutions of AP (1%, 3%, 5%) in the presence of Cu^{2+} ions (1%-10%) by the method of ionotropic gelation. The diameter and density of the CuPGPs were determined. Dry CuPGPs formed from 5% AP with all tested concentrations of copper ions have the largest diameter (0.96-1.15 mm), and gel particles formed on the basis of 1% AP have the smallest diameter (0.42-0.74 mm). CuPGPs formed from 5% AP have the highest density (1.43-1.65 mg/mm^3), and CuPGPs formed on the basis of 1% AP have the lowest density (0.65-0.92 mg/mm^3). Gel particles obtained from 1% AP swelled in simulated gastric fluid (SGF) by 161% and then completely degraded immediately upon entering in simulated intestinal fluid (SIF). CuPGPs obtained from 3% AP swelled by 166% in SGF and 148% in SIF, and completely degraded in SIF after 2.5 hours of incubation in it. Gel particles obtained from 5% AP in the presence of 10% Cu^{2+} swelled most strongly – by 173% in SGF and by 208% in SIF. And then, they degraded after 8 hours of incubation in simulated colonic fluid (SCF).(**International Journal of Biomedicine. 2021;11(1):50-52.**)

Key Words: apple pectin • cross-linking agents • copper ions • gel particles • gastroentestinal tract

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Abbreviations

AP, apple pectin; **CuPGPs**, copper-pectic gel particles; **SGF**, simulated gastric fluid; **SIF**, simulated intestinal fluid; **SCF**, simulated colonic fluid.

Introduction

Pectins are natural, biodegradable, ionic polysaccharides that are widely used in the food and pharmaceutical industries. The widespread use of pectins is based on their ability to form gels in the presence of divalent cations, such as calcium ions. Gelation occurs as a result of specific and strong interactions between calcium ions and galacturonic acid residues of pectins.[1]

Pectins differ in macromolecular structure, monosaccharide composition and degree of methyleterification of carboxyl groups of galacturonic acid residues. Pectins readily form gel particles with a wide range of physical and mechanical properties. It has been found that the key factors affecting the formation of gel particles are the degree of methyleterification of carboxyl groups, temperature, the presence of sugar, and polyvalent ions.[2]

To obtain pectic gel particles, copper ions (Cu^{2+} ions) are also used instead of calcium ions as a cross-linking agent

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because they bind pectin more strongly than calcium ions.[3] Copper is a trace element necessary for plants and animals. The main biochemical function of copper is participation in enzymatic reactions as an activator or in the composition of copper-containing enzymes.[4]

The study of the gelation of low methyl esterified pectin (apple pectin [AP]) using Cu^{2+} ions as a cross-binding agent is also relevant. In our previous article, we investigated the properties and swelling in an artificial gastroenteric environment of CuPGPs obtained from 2% and 4% AP in the presence of Cu^{2+} ions.[5]

The purpose of the present work was to obtain and study the properties of CuPGPs obtained from aqueous solutions of AP in the concentration range of 1%-5% (1%, 3%, 5%) in the presence of Cu^{2+} ions (1%-10%). The swelling of the obtained CuPGPs during incubation in an artificial gastroenteric environment was also investigated.

Materials and Methods

We used commercial AP AU701 (Herbstreith & Fox KG, Germany). CuPGPs were obtained from aqueous solutions of AP (1%, 3%, 5%) in the presence of Cu^{2+} ions (1%-10%) by the method of ionotropic gelation,[6,7] Certain weighed portions of AP were dissolved in corresponding volumes of distilled water by slow stirring with a magnetic stirrer MM-5 (Russia) for 2-5 hours at room temperature until complete dissolution

Gel particles of spherical shape were prepared by drop-by-drop injection of a pectin solution from a syringe through a needle with a hole diameter of 0.7 mm on the distance of 4-5 cm in the slowly stirred copper chloride solution and further stirring for 20 min at room temperature. The resulting gel particles were then washed three times in distilled water with stirring for 5 minutes and dried for 10-14 h at 37°C.

Further, the diameter and density of CuPGPs were determined using an optical microscope (Altami, Russia) with a camera and an image analysis program (ImageJ 1.46r program, National Institutes of Health, USA). For calibration, a linear scale was used; one pixel corresponded to 0.024 mm.

The swelling and degradation of CuPGPs were studied under conditions simulating the gastrointestinal environment: for these purposes, the simulated gastric fluid (SGF solution), simulated intestinal fluid (SIF solution) and simulated colonic fluid (SCF solution), as described previously.[8, 9]

To determine swelling and degradation, dry CuPGPs (1–2 mg) were placed in Petri dishes (diameter 3.5 cm) and subsequently incubated in 3 ml of SGF (2 h), SIF (4h) and SCF solutions with shaking in a shaker (Titramax 1000, Heidolph, Germany) at 100 rpm and at 37°C. The diameter and density of 100 randomly selected gel particles were measured as described above after certain time intervals. The experiments were performed in triplicate. The degree of gel swelling (SD,%) was determined by the formula[10]: $SD = (D_1\text{-}D_0)/ D_0 \times 100\%$, where D_1 – diameter of the particles (mm) after a certain incubation time in the medium, D_0 – initial diameter of the particles (mm).

The statistical analysis was performed using the statistical software BioStat (version 4.03) and Microsoft Office Excel 2007.

Results and Discussion

CuPGPs were obtained from aqueous solutions of AP (1%, 3%, 5%) in the presence of Cu^{2+} ions (1%-10%) by the method of ionotropic gelation, in which intermolecular cross-links arise between divalent Cu^{2+} ions and negatively charged carboxyl groups of AP. [5,11,12] Morphological (diameter) and structural-mechanical (density) characteristics of gel particles are given.

Dry CuPGPs formed from 5% AP with all tested concentrations of copper ions have the largest diameter (0.96-1.15 mm), and gel particles formed on the basis of 1% AP have the smallest diameter (0.42-0.74 mm) (Table 1). It was shown[13] that the diameter of dry calcium-pectin gel particles formed from 1% AP in the presence of 1.0 M $CaCl_2$ solution is approximately 3 times larger – 3.56 mm. That is, Cu^{2+} ions contribute to the formation of smaller pectin gel particles than Ca^{2+} ions.

Table 1.

Diameter of dry CuPGPs (M±SD, mm)

AP concentration	$CuCl_2$ concentration					
	1%	2%	3%	4%	5%	10%
1%	0.42 ± 0.05	0.58 ± 0.03	0.68 ± 0.05	0.71 ± 0.06	0.73 ± 0.07	0.74 ± 0.08
3%	0.82 ± 0.02	0.83 ± 0.05	0.87 ± 0.04	0.89 ± 0.04	0.92 ± 0.06	0.94 ± 0.04
5%	0.96 ± 0.08	0.98 ± 0.06	0.99 ± 0.06	1.00 ± 0.05	1.06 ± 0.05	1.15 ± 0.06

In several works,[3,14] it has been shown that Cu^{2+} ions, as cross-binding agents, form denser gel particles than Ca^{2+} ions. At the same time, with an increase in the concentration of AP, the density of the gel particles increases at all tested concentrations of Cu^{2+} ions. CuPGPs formed from 5% AP have the highest density(1.43-1.65 mg/mm^3), and CuPGPs formed on the basis of 1% AP have the lowest density(0.65-0.92 mg/mm^3) (Table 2). Ca^{2+} ions, as cross-linking agents, form gel particles from AP with a lower density(0.40 mg/mm^3).[13]

Table 2.

Density of dry CuPGPs (M±SD, mg/mm^3)

AP concentration	$CuCl_2$ concentration					
	1%	2%	3%	4%	5%	10%
1%	0.65 ± 0.14	0.69 ± 0.17	0.75 ± 0.18	0.81 ± 0.14	0.85 ± 0.25	0.92 ± 0.21
3%	1.40 ± 0.15	1.42 ± 0.12	1.46 ± 0.16	1.49 ± 0.12	1.50 ± 0.18	1.52 ± 0.16
5%	1.43 ± 0.20	1.46 ± 0.25	1.51 ± 0.21	1.52 ± 0.18	1.56 ± 0.22	1.65 ± 0.16

The swelling of the obtained CuPGPs in an artificial gastroenteric environment was studied. Gel particles formed from AP (1%, 3%, 5%) with 1%-5% Cu^{2+} as a cross-linking agent swelled in a simulated gastric fluid SGF by 90%-150% and degraded in a simulated intestinal fluid SIF during the first 30 minutes of incubation in it.

With an increase in AP concentration, the degree of swelling of CuPGPs increases (Fig.1). Gel particles obtained from 1% AP swelled in SGF by 161% and then completely degraded immediately upon entering in SIF. CuPGPs obtained from 3% AP swelled by 166% in SGF and 148% in SIF, and completely degraded in SIF after 2.5 hours of incubation in it.

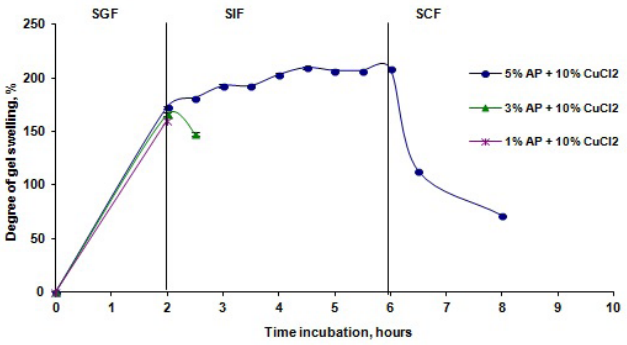

Fig. 1. *Swelling and degradation of CuPGPs formed from AP (1%, 3%, 5%) and Cu^{2+} ions (10% $CuCl_2$) in an artificial SGF, SIF, and SCF.*

Gel particles obtained from 5% AP in the presence of 10% Cu^{2+} swelled most strongly – by 173% in SGF and by 208% in SIF. And then, they degraded after 8 hours of incubation in SCF.

Thus, we can conclude that Cu^{2+} ions are stronger cross-linking agents than Ca^{2+} ions. CuPGPs formed from 5% AP in the presence of 10% Cu^{2+} have the highest degree of swelling and the least degradability in an artificial gastroenteric environment.

Competing Interests

The authors declare that they have no competing interests.

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