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Assessment of the Structure of Maize Starch and Flaxseed Gum Interaction by the Zener Fractional Rheological Model

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ABSTRACT: *The effect of changes in temperature and concentration of flaxseed gum on viscoelastic properties of paste made from maize starch is discussed in the paper. It is indicated that the structure can be assessed during the interaction of maize starch paste and flaxseed gum using a rheological fractional standard linear solid model. Rheological parameters of this model for different values of temperature and concentration of flaxseed gum in the starch paste were determined and differences in the properties of such mixture structure were shown.*

Keywords: *fractional standard linear solid model, flaxseed gum, maize starch*

I. INTRODUCTION

Substances commonly added to food are hydrocolloids, i.e. food gums. These are water-soluble polysaccharides with thickening and/or gelling properties. As food components they cause a decrease of its energy value and due to binding large amounts of water they can be used as fat replacers. Used as food additives they improve the quality and durability of food products ensuring their storage stability. This broad scope of applicability of hydrocolloids in food production causes that recently especially much attention is paid to the group of polysaccharides extracted from seaweed (alginates, carrageenans), rare plants (guar gum, carob powder) and also from bacteria produced in industrial-scale bioreactors (xanthan, dextran, curdlan, gellan). However, the best known polysaccharide is starch. Due to industrial importance of starch used in fruit and vegetable industry, confectionery and meat processing, the rheological properties of starch pastes are a subject of continuous studies. Systems composed of starch and non-starch hydrocolloid are very important texture-forming agents in many food products. Hence, particularly significant are the mutual interactions between starch and food gums [1,2].

At the same time, the demand of food industry for thickeners and stabilizers is so large that new sources of such substances are still searched for. Researchers are interested in the extracts from seeds of the following plants: *Alyssum homolocarpum* [3], *Lepidium sativum* [4], *Lepidium perfoliatum* [5], *Ocimum basilicum* [6], *Sinapis alba* [7,8] and *Linum usitatissimum* [9-13].

Extracts from flaxseed, the so called flaxseed mucilage or flaxseed gum, have enjoyed significant interest from both food and pharmaceutical industry. Flaxseed mucilage is a source of water-soluble dietary fiber which can be used as replacers of commonly applied gelling agents. The extensive use of flaxseed gum made that the effect of its concentration, pH of solution and electrolyte temperature on its rheological properties was investigated by many researchers [14-18]. Nevertheless, until present, only one study trying to explain the mutual interactions between maize starch and non-starch polysaccharide hydrocolloid such as flaxseed gum was published [18]. Wang [18] analyzed also the rheological properties of a mixture of flaxseed gum and maize starch and found that they formed a gel whose strength increased with the concentration of flaxseed gum in the mixture. Additionally, flaxseed gum added to maize starch pastes increased their viscoelastic properties. However, such an analysis appeared insufficient to estimate viscoelastic properties of the formed structure.

The authors of this study decided to explain the mutual interactions between flaxseed gum and maize starch using a rheological fractional standard linear solid model. The proposed method for interpretation of the results of rheological measurements allowed for a comprehensive assessment of the tested biomaterial structure.

II. MATERIALS AND METHODS

The analysis of the effect of changes in flaxseed gum temperature and concentration on viscoelastic properties of the paste made from maize starch was based on the results of Wang's rheological studies [18]. The researchers described the curves of storage G' and loss G" moduli in the range of oscillation frequencies from 0.1 to 10 s^{-1} at constant deformation of 2%, using the power-law model [19] in the following form:

$$G' = k' \cdot \omega^{n'} \tag{1}$$

$$G'' = k'' \cdot \omega^{n''} \tag{2}$$

The values of parameters k', k", n' and n" of this model for Wang's studies are given in Table 1.

Table 1. Results of Wang's oscillation studies for the mixture of flaxseed gum and 3% maize starch paste.

Flaxseed gum concentration [%]	G'		G"	
	n' [-]	k' [Pas^n]	n" [-]	k" [Pas^n]
Temperature 25°C				
0	0.082	10.35	0.391	1.71
0.1	0.096	16.83	0.234	3.02
0.3	0.100	27.41	0.230	4.91
0.5	0.093	40.74	0.185	6.52
Temperature 50°C				
0	0.068	11.26	0.357	1.46
0.1	0.098	16.51	0.217	2.79
0.3	0.101	24.09	0.224	4.04
0.5	0.101	33.46	0.163	5.47
Temperature 75°C				
0	0.069	10.86	0.316	1.37
0.1	0.098	13.28	0.234	2.23
0.3	0.112	16.03	0.232	3.03
0.5	0.123	21.12	0.165	4.44

The analysis of viscoelastic properties and mutual interactions of the mixture composed of flaxseed gum and maize starch covers quick and slow dissipation processes. Quick dissipation is a result of movements which appear in structural segments of the network between its nodes, while slow dissipation processes correspond to movements across the network nodes [20]. The Zener standard linear solid model, Fig. 1, seems to be most appropriate for a comprehensive description of these two processes in shaping the structure of maize starch with flaxseed gum.

Fig. 1 shows a standard linear solid model. It is composed of a spring and a system of elements of the Maxwell model, i.e. the spring connected in series with a dashpot in which the dashpot is replaced by the Scott-Blair element called a viscoelastic element – a springpot.

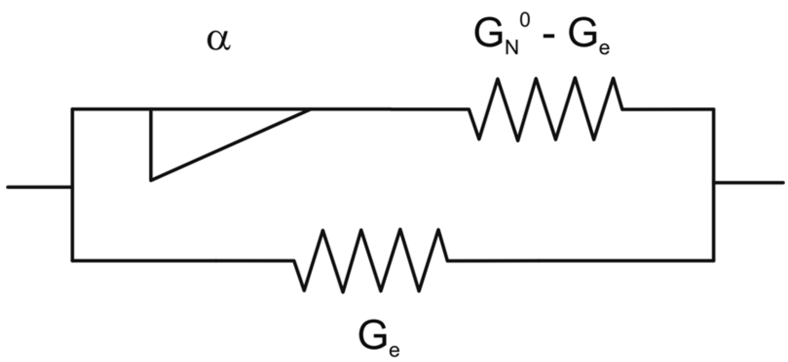

Fig. 1. Standard linear solid model (SLSM) Zener model.

It seems that viscoelastic properties of biomaterials can be described more precisely using the so called fractional derivative rheological models and fractional calculus than when classical simple rheological models of viscoelastic fluids are applied. The advantage of the fractional rheological models is that it can describe dynamic behavior by means of a single equation which contains a number of constant parameters determining viscoelastic properties of the tested material.

Dinzart and Lipiński [21] described several fractional rheological models used to determine viscoelastic properties of the tested media. In particular, a standard linear solid model, a so called fractional Zener model, is worth mentioning, cf. Fig. 1 [22,23]. The values of storage G' and loss G" moduli for this model can be described using trigonometric functions which lead to the following equations:

$$G' = G_e \frac{(1+k)(\omega\tau_0)^{2\alpha} + (\omega\tau_0)^{\alpha}(2+k)\cos\left(\frac{\pi\alpha}{2}\right) + 1}{\left[1 + (\omega\tau_0)^{\alpha}\cos\left(\frac{\pi\alpha}{2}\right)\right]^2 + \left[(\omega\tau_0)^{\alpha}\sin\left(\frac{\pi\alpha}{2}\right)\right]^2} \quad (3)$$

$$G'' = G_e \frac{k(\omega\tau_0)^{\alpha}\sin\left(\frac{\pi\alpha}{2}\right)}{\left[1 + (\omega\tau_0)^{\alpha}\cos\left(\frac{\pi\alpha}{2}\right)\right]^2 + \left[(\omega\tau_0)^{\alpha}\sin\left(\frac{\pi\alpha}{2}\right)\right]^2} \quad (4)$$

$$k = \frac{G_N^0 - G_e}{G_e} \quad (5)$$

where: G_e is the equilibrium modulus, G_N^0 is the plateau modulus, τ_0 is the relaxation time, α is the fractional exponent.

The Zener model – eq. (3) to (5), has five rheological parameters, i.e. G_e, G_N^0, τ_0, α and k. These parameters represent the following properties of the tested materials [24,25]:

– equilibrium modulus G_e represents the overall network elasticity, and its reciprocal is susceptibility in the state of equilibrium J_e. High values of modulus G_e indicate an increase of elastic properties of the material.
– plateau modulus G_N^0 is identified with the structure cross-linking power. The higher the value of this modulus the higher the structure cross-linking. The reciprocal of this modulus is susceptibility of the structure at cross-linking J_N^0.
– characteristic relaxation time τ_0 defines the time after which stress relaxation will occur. Short relaxation times indicate strong elastic properties of the material.
– parameter α indicates characteristic behaviors of elastic bodies if its value is close or equal to zero; when it is close or equal to one it indicates behaviors characteristic of viscous liquids.
– parameter k is the damping factor of network oscillations. The higher its value, the more the oscillations are damped by the formed network of a given biopolymer.

The knowledge of these parameters allows us to get additional information on the tested material properties. We can learn the value of dispersion modulus *f*, cross-linking density ω_0 and gel stiffness S. Relations which make it possible to determine the values of three new parameters on the basis of the five parameters already existing in the Zener fractional model are described by the following equations:

- dispersion modulus *f*

$$f = \frac{G_N^0}{G_e} \quad (6)$$

- cross-linking density ω_0

$$\omega_0 = \frac{1}{\tau_0} \quad (7)$$

- gel stiffness S [26]

$$S = G_N^0 \cdot \tau_0^{\alpha} \quad (8)$$

The Zener standard linear solid model holds if the following conditions are satisfied:

$$G_e \geq 0$$
$$G_N^0 \geq 0$$
$$\tau_0 \geq 0$$
$$0 \leq \alpha \leq 1$$

As a result, 8 rheological parameters presented above are obtained. They are used in a comprehensive analysis of viscoelastic properties of the tested material.

III. RESULTS AND DISCUSSION

Wang [18] tried to determine rheological properties of a mixture composed of flaxseed gum at three different concentrations and a 3% maize starch paste at different temperature and concentration of flaxseed gum in the mixture. The use of a simple power-law model by Wang [18], cf. eq. (1) and (2) [19], does not allow for a comprehensive assessment of the mechanical state of a structure, neither it explains the reasons of such a state.

In the present study, an attempt was made to relate the structure to mutual interactions between flaxseed gum and maize starch using the Zener model. Having the experimental data on rheological properties of flaxseed gum shown in Wang's study [18], we described them by the Zener fractional rheological model. The obtained parameters are given in Table 2.

The application of Zener standard linear solid model was also illustrated graphically in the form of relations of experimental values of storage G' and loss G" moduli as a function of ω with curves obtained from the Zener model given in eq. (3) to (5) – cf. Fig. 2 to 4.

Diagrams in Fig. 2 to 4 show very good agreement of Wang's experimental data [18] with the curves obtained by means of the Zener fractional standard linear solid model. The mean error in the description of experimental data for all cases was ±3.0%. The maximum error of description of the experimental data by means of the Zener fractional model was ±6.0% for most of the analyzed cases, only for 3% maize starch paste without flaxseed gum addition at 25°C at higher oscillation frequency it was higher and amounted to +9.2%.

Table 2. Rheological parameters of the Zener model calculated from Wang's experimental data for the mixture of flaxseed gum and 3% maize starch paste.

Flaxseed gum concentration [%]	G_e [Pa]	G_N^0 [Pa]	τ_0 [s]	α [-]	k [-]	*f* [-]	ω_0 [s^{-1}]	S [Pas]	R^2 [-]
Temperature 25°C									
0	7.659	138.09	$9.63\cdot10^{-5}$	0.409	17.03	18.03	$1.04\cdot10^{+4}$	3.16	0.998
0.1	9.239	139.39	$3.42\cdot10^{-5}$	0.266	14.09	15.09	$2.92\cdot10^{+4}$	9.00	0.999
0.3	14.889	228.52	$2.98\cdot10^{-5}$	0.262	14.35	15.35	$3.35\cdot10^{+4}$	14.84	0.999
0.5	20.617	266.07	$2.28\cdot10^{-5}$	0.223	11.90	12.90	$4.38\cdot10^{+4}$	24.51	1.000
Temperature 50°C									
0	8.722	139.11	$3.13\cdot10^{-5}$	0.372	14.95	15.95	$3.19\cdot10^{+4}$	2.94	0.999
0.1	8.928	139.23	$1.44\cdot10^{-5}$	0.246	14.59	15.59	$6.95\cdot10^{+4}$	8.94	1.000
0.3	13.780	143.58	$1.28\cdot10^{-4}$	0.269	9.42	10.42	$7.79\cdot10^{+3}$	12.92	1.000
0.5	15.888	143.23	$3.83\cdot10^{-4}$	0.229	8.02	9.02	$2.61\cdot10^{+3}$	23.67	1.000
Temperature 75°C									
0	8.180	138.49	$9.63\cdot10^{-6}$	0.330	15.93	16.93	$1.04\cdot10^{+5}$	3.06	0.999
0.1	7.547	137.91	$7.06\cdot10^{-6}$	0.256	17.27	18.27	$1.41\cdot10^{+5}$	6.61	1.000
0.3	8.391	138.54	$3.32\cdot10^{-5}$	0.265	15.51	16.51	$3.01\cdot10^{+4}$	9.05	1.000
0.5	6.523	136.22	$8.26\cdot10^{-5}$	0.216	19.88	20.88	$1.21\cdot10^{+4}$	17.92	1.000

Analysis of the experimental data by means of the Zener fractional rheological model, cf. Table 2, allows us to state that in all analyzed cases, both for the pure maize starch paste without the addition of flaxseed gum and for the maize starch paste with flaxseed gum at the concentration 0.1%, 0.3% and 0.5%, a medium with the structure typical of viscoelastic quasi-solid bodies is formed. This is confirmed by quite high values of the viscoelastic modulus of plateau G_N^0.

At the temperature 25°C, cf. Table 2, the moduli responsible for the overall network elasticity G_e and cross-linking power G_N^0 increase with the concentration of flaxseed gum in the mixture with maize starch to maximum values for flaxseed gum concentration equal to 0.5%. The characteristic relaxation time τ_0 and fractional exponent α decrease with an increase of flaxseed gum concentration in the mixture and reach the lowest values for flaxseed gum concentration equal to 0.5%, leading as a consequence to higher stiffness of the gel formed for this flaxseed gum concentration.

The cross-linking density ω_0 of flaxseed gum and maize starch mixture increases with flaxseed gum concentration in the mixture and reaches the highest value for the concentration equal to 0.5%, cf. Table 2. It is also important that the values of cross-linking density are within the same decade of oscillation frequencies (10^{+4} s^{-1}) which additionally can indicate that the type of structure is similar. While considering cross-linking density ω_0 it was also observed that its highest values corresponded to the highest value of the viscoelastic modulus of plateau G_N^0 which showed similarity of the elementary units forming the tested medium network.

The highest dispersion modulus *f* was observed for pure 3% maize starch paste without the addition of flaxseed gum which confirmed wide distribution of molecular mass and a possibility of reorganizing the structure during the oscillation studies; no effect of flaxseed gum on blocking starch granules was reported – cf. Table 2.

A growing flaxseed gum concentration in the mixture with maize starch at 25°C leads to the formation of a medium with strong elastic properties and strong structure which can slow down the processes of physical ageing of such a system in time. The addition of 0.5% flaxseed gum to maize starch which is manifested by an increase of dispersion modulus *f* and decrease of the damping factor of network oscillations k shows that the flaxseed gum begins to dominate in the mixture being also responsible for an increase of its elastic properties.

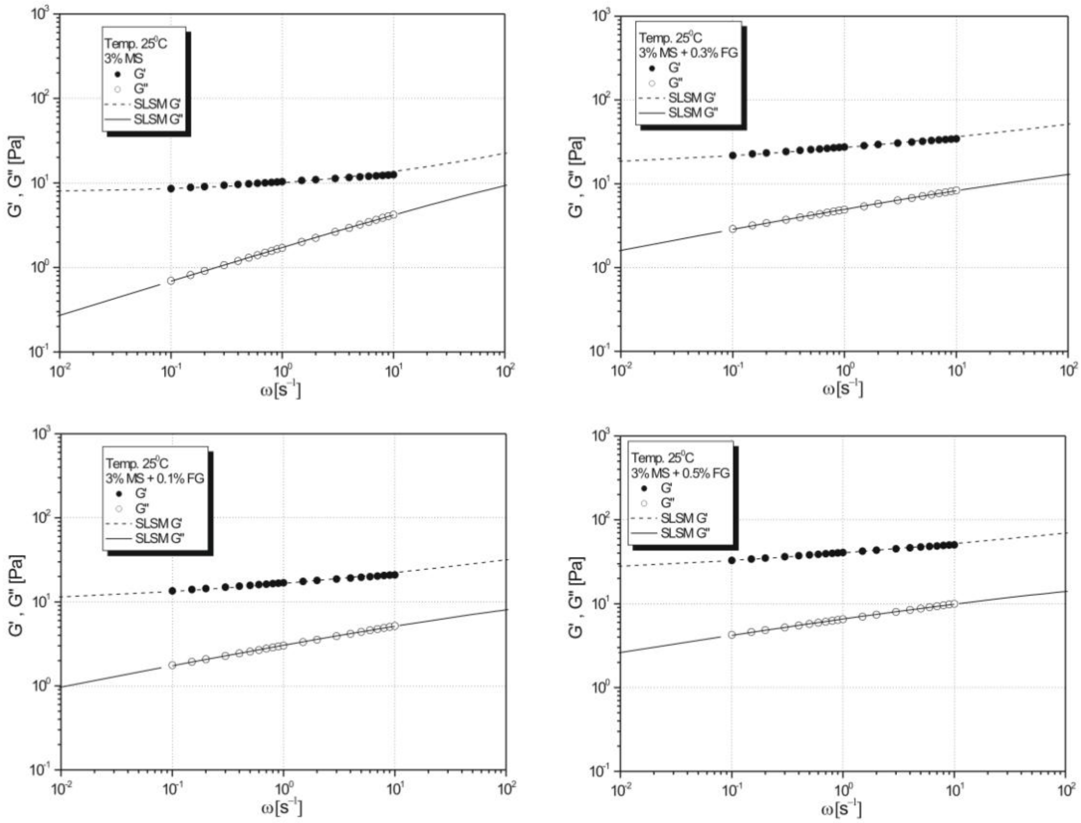

Fig. 2. Frequency dependence of G' and G" for flaxseed gum, temp. 25°C. The curves throughout the experimental data are calculated using the standard linear solid model (SLSM – Zener model).

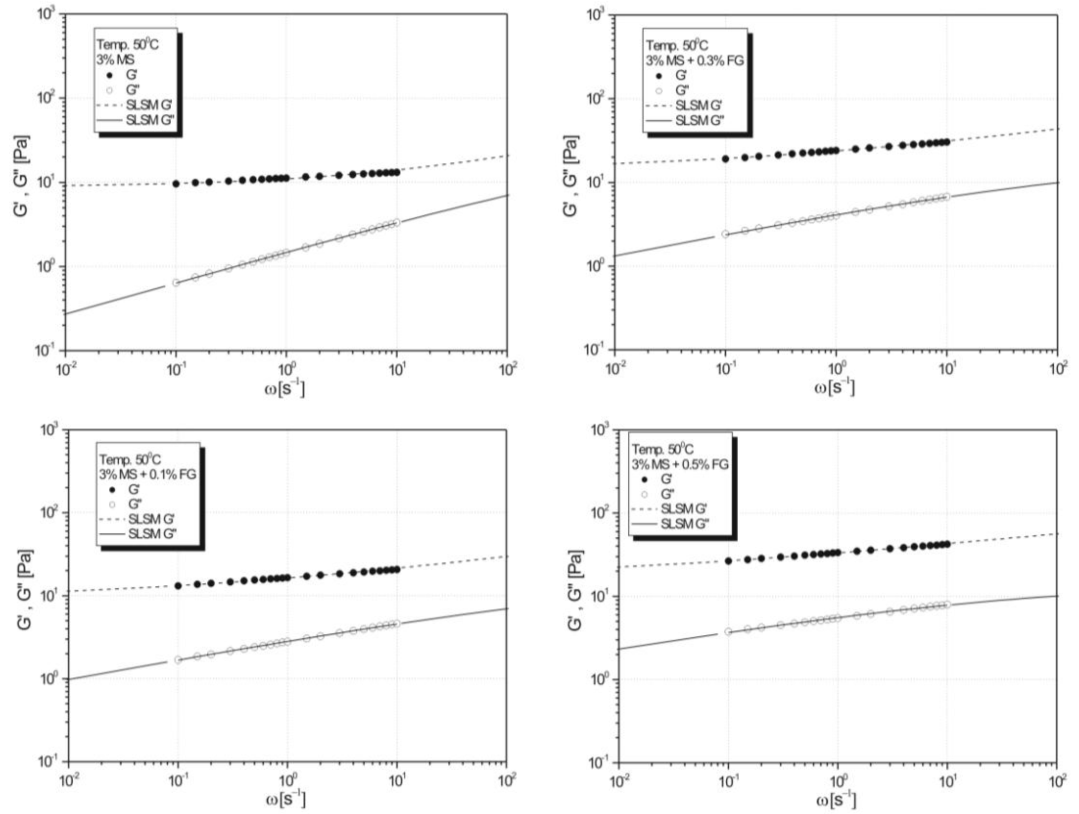

Fig. 3. Frequency dependence of G' and G" for flaxseed gum, temp. 50°C. The curves throughout the experimental data are calculated using the standard linear solid model (SLSM – Zener model).

At the temperature 50°C, cf. Table 2, the equilibrium modulus G_e responsible for the overall network elasticity increases systematically with the flaxseed gum concentration in the mixture with maize starch and reaches the highest value at 0.5% concentration. It is worth mentioning, however, that up to the flaxseed gum concentration of 0.1%, its effect on the overall network elasticity at this temperature is relatively small. A similar relation is observed when the network strength represented by the viscoelastic modulus of plateau G_N^0 is considered. The network strength does not increase significantly up to 0.1% concentration of the flaxseed gum. Only the gum addition at the concentrations 0.3% and 0.5% causes a slight increase of the network strength reaching ca. 3%. The characteristic relaxation time τ_0 achieves a maximum value for pure maize starch which indicates quite strong elastic properties of this medium. Like the ability of damping network oscillations, molecular mass distribution represented by the dispersion modulus *f* decreases with an increase of flaxseed gum concentration in the mixture with starch, reaching the highest value for pure maize starch paste. This confirms the possibility of structure reorganization during oscillation studies at this temperature and lack of any effect of flaxseed gum on the blocking of starch granules.

The addition of flaxseed gum to maize starch at 50°C results in the formation of gel with growing stiffness S, which is maximum for flaxseed gum concentration equal to 0.5%, cf. Table 2. There is an interaction between the added flaxseed gum and maize starch which is manifested by blocking starch granules by flaxseed gum. As a result, this leads to lower polydispersity of the system and at the same time to smaller density of structure cross-linking ω_0. This is a contradiction of the synergistic effect of increasing stress on the swelling starch granules described by Christianson [27].

At the temperature 75°C, cf. Table 2, the equilibrium modulus G_e responsible for the overall network elasticity is similar both for pure maize starch paste and for the paste with the addition of flaxseed gum. On a similar level are the values of the viscoelastic modulus of plateau G_N^0. The cross-linking power of a medium tested at this temperature is similar, for two extreme values the difference is about 28.6%. The values of characteristic relaxation time τ_0 show that the effect of flaxseed gum on elastic properties of the mixture is observed up to the gum concentration of 0.3%. At the same time polydispersity of the mixture expressed as the dispersion modulus *f* is the highest for the mixture of maize starch and 0.5% flaxseed gum, which shows that at this temperature and flaxseed gum concentration the starch granules are coated with flaxseed gum. This is also revealed by lower overall network elasticity. A bigger contribution of flaxseed gum to structure formation results in the growth of gel stiffness and possibility of damping the network oscillations.

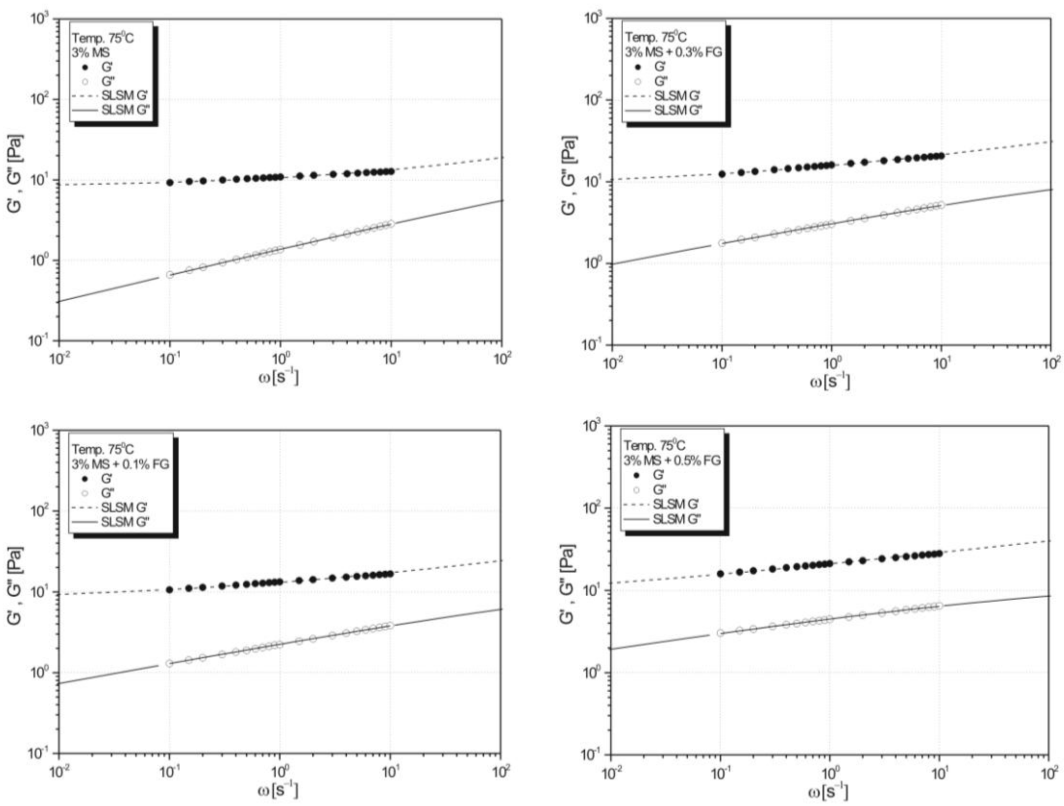

Fig. 4. Frequency dependence of G' and G" for flaxseed gum, temp. 75°C. The curves throughout the experimental data are calculated using the standard linear solid model (SLSM – Zener model).

Due to the blocking of starch granules, the addition of flaxseed gum to maize starch paste at 75°C leads to smaller density of the cross-linking of such mixture structure – cf. the values of cross-linking density ω_0 in Table 2.

To better illustrate differences in the structure of flaxseed gum and maize starch mixture, the curves of storage G’ and loss G” moduli are shown additionally in the form of so called reduced curves in the system of coordinates $G'/G_N^0=f(\omega/\omega_0)$ and $G''/G_N^0=f(\omega/\omega_0)$ – cf. Fig. 5 to 6.

The reduced curves confirm that:

- at the temperature 25°C practically three types of structure are obtained, cf. Fig. 5a: a very similar type of structure at flaxseed gum concentrations of 0.1% and 0.3% in the mixture with maize starch (very good superposition of curves $G'/G_N^0=f(\omega/\omega_0)$ and $G''/G_N^0=f(\omega/\omega_0)$ in Fig. 5a), a similar but different type of structure for the addition of 0.5% flaxseed gum to maize starch, and the third structure for pure maize starch paste quite different from the other types;
- at the temperature 50°C the situation is similar, cf. Fig. 5b, but higher flaxseed gum concentration in the maize starch paste results in the unification of structure type, so the type of structure for the flaxseed gum concentration equal to 0.5% is more and more similar to the type of structure at the gum concentrations 0.1% and 0.3%, quite different structure type is formed by pure maize starch paste;
- at the temperature 75°C, cf. Fig. 5c, the type of structure formed at flaxseed gum concentration 0.1% and 0.3% is very similar, a 0.5% addition of flaxseed gum forms the type of structure for which the flaxseed gum is more responsible than maize starch which forms a separate structure type itself;
- pure 3% maize starch paste at all three analyzed temperatures forms a separate but at the same time not very similar type of structure, cf. Fig. 6a;

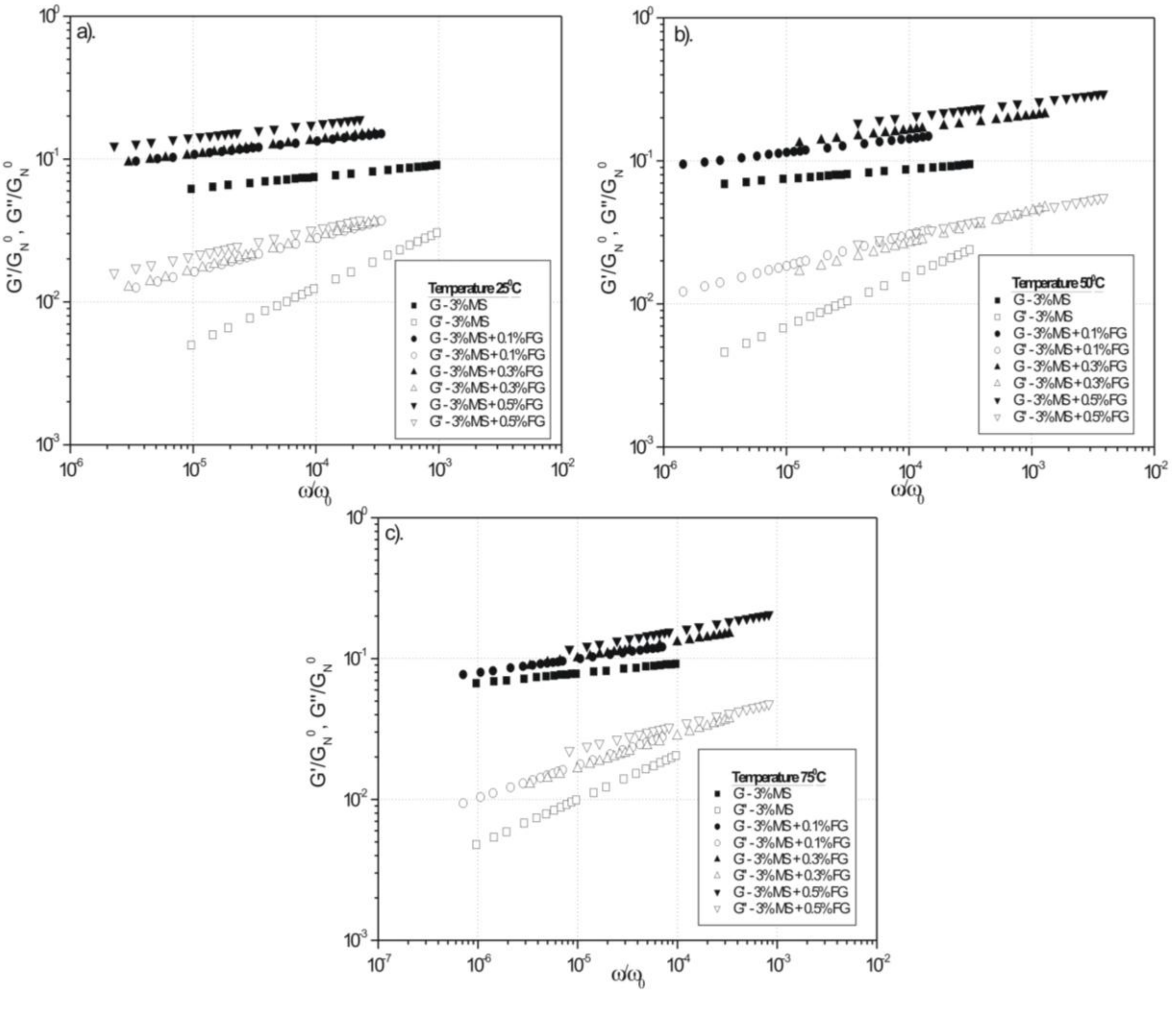

Fig. 5. Reduced curves of maize starch and flaxseed gum mixture at changing flaxseed gum concentration.

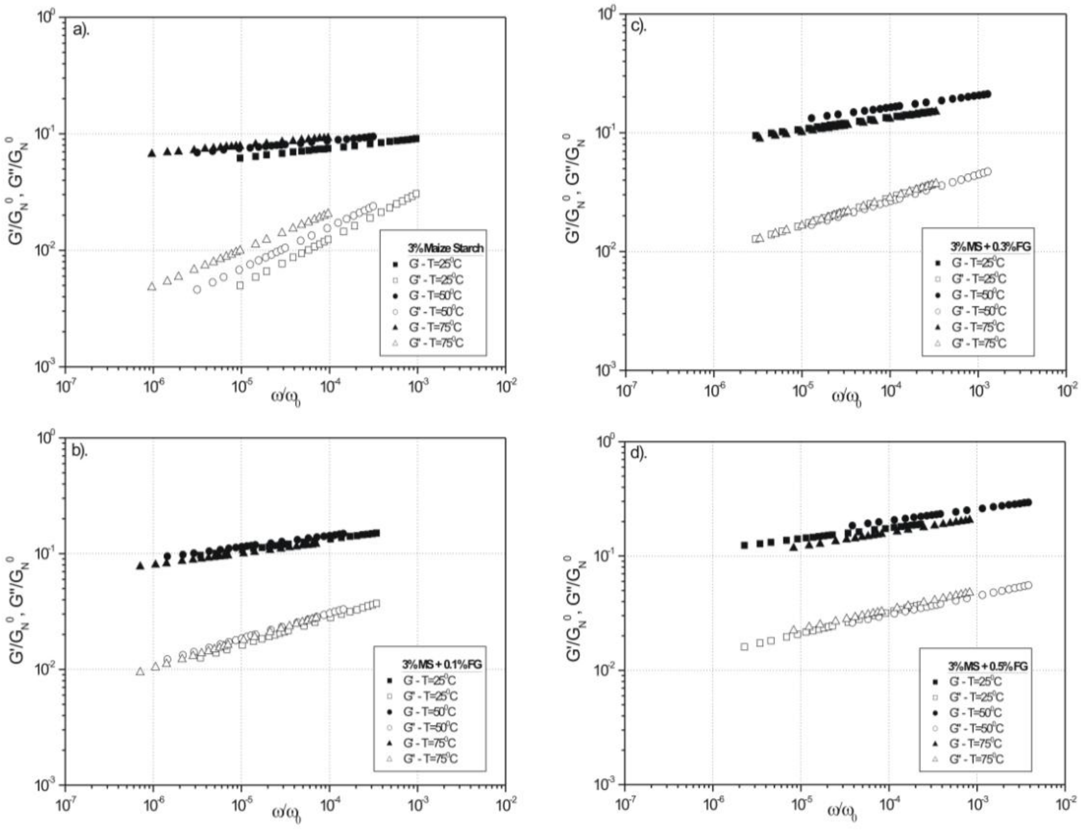

Fig. 6. Reduced curves of maize starch and flaxseed gum mixture at changing medium temperature.

IV. CONCLUSIONS

It was proved that the fractional rheological model in the description of biomaterial structure could be used in a comprehensive analysis and estimation of the properties of structure of these materials, including their viscous, elastic or viscoelastic properties. The fractional rheological models provide more information on the structure of biomaterials than the previously applied classical mechanical models such as Maxwell or Kelvin-Voight models.

The analysis of data obtained by means of the Zener fractional rheological model allowed us to state that for pure starch paste and a mixture of this paste with flaxseed gum a medium is formed with a structure representing the behavior typical of viscoelastic quasi-solid bodies. The values of parameters of the Zener fractional rheological model show that:

- an increasing flaxseed gum concentration in the mixture with maize starch at 25°C leads as a result to obtaining a medium with strong elastic properties, strong structure enabling slow-down of physical ageing of such a system in time, and having a differentiated type of structure,
- an increasing flaxseed gum concentration in the maize starch paste at 50°C leads to the unification of structure type, formation of a gel with higher stiffness but smaller cross-linking density,
- an increasing flaxseed gum concentration in the mixture with maize starch at 75°C results in an increase of gel stiffness and a possibility of damping network oscillations and smaller overall network elasticity, a new type of structure is formed for which flaxseed gum is more responsible than maize starch.

In summary, the comprehensive analysis of the mechanical state of biomaterial structure by means of fractional rheological models can be useful to control or form appropriately the structure of biomaterials for the needs of product which is especially important from the point of view of materials science.

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