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Rheological characterization of snail Pâté. II. Stress relaxation

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Abstract

Three sorts of snail pâté with different quantities of a vegetal protein (biogel) were prepared. In the third sort, without vegetal protein, the content of bacon was substituted with vegetal oil. The influence of sample temperature and the content of biogel on the viscoelastic characteristics of snail pâté were studied in this paper. These characteristics of snail pâté were derived from stress relaxation tests. The relaxation times and the elastic moduli, as viscoelastic characteristics, were calculated from the relaxation curves by non-linear regression. From above viscoelastic parameters, the viscosities were calculated. For statistical analysis, one-way analysis of variance (ANOVA) was used. Correlation between experimental relaxation curves and calculated curves were emphasized by absolute average deviation. The stiffest snail pâté contains 2.7% biogel.

Keywords: snail pâté, rheology, viscoelastic properties, stress relaxation, temperature influence.

1. Introduction

Snail, for its meat and shell, is reared in commercial farms. Snail meat is a delicacy in Japanese and Chinese. In French, North of America and Australia it is consumed also as main meal. In South Africa, land snail is also a traditional food. Snail recipes vary from cuisine to cuisine. Studies on the nutritional value of snail have reported that snail is high in protein but low in fat contents. It is estimated that snail is 15% protein, 2.4% fat and about 80% water. This makes snail healthy alternative food for people with high protein low fat diet requirements. Besides, snail is high in health benefiting essential fatty acids such as linoleic acids and linolenic acids [1]. Therefore, snail meat can be included in the feed of individuals under dietary restrictions. Snail meat (H. Aspersa maxima) is an adequate protein source with low lipid content in its essential fatty acid composition (linoleic and linolenic acids) and polyunsaturated fatty acids with more than 20 C atoms (0.45-2.66%), indicating that the use of this food is suitable for patient diet, irrespective of total lipid content [2]. Having a low content of lipids and high content of proteins, meat snail is similarly with fish meat.

Nowadays, there is an increasing demand for foodstuffs with lower fat content. However, the reduction of fat in meat products or its replacement with a more unsaturated fat might affect their technological or sensory characteristics [3], mainly in products in which fat is one of the major components of the formulae, such as frankfurters, sausages, beef patties or liver pâtés [4]. The rheology of pâté is less studied. There are same references on rheology of ham pâté [5] and of pork liver pâté by textural profile analysis and penetration tests [6]. In addition, the consistency at 23°C of the refrigerated pâtés was calculated from compression tests [3].

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There are not signaled yet the study of viscoelastic characteristics of different pâté formulae. The similar studies on snail pâté are not signaled. The stress relaxation test (or step strain test) is one of the most important evaluation tools used for determining viscoelastic properties of materials [7-12]

Three new recipes for snail pâté are proposed in a previous paper [13], snail meat being considered a substitute of other type of meat used in classical pâté. In this paper, we try to characterize the viscoelasticity of some variants of snail pâté by stress relaxation tests, realized at three different temperatures.

2. Material and Methods

2.1. Theoretical background. Many food products simultaneously exhibit obvious fluid-like (viscous) and solid-like (elastic) behavior. Manifestations of this behavior, due to a high elastic component, can be very strong and create difficult problems in process engineering [14]. In the book of Steffe [14], one of the best books for American food engineers, the subject of viscoelasticity has very well present in chapter 5. One of the most used transient tests for viscoelasticity is stress relaxation (step strain). In stress-relaxation experiments, the test specimen is compressed to a predetermined level of strain that is kept constant, during which time the viscoelastic material shows a declining trend of force/stress as a function of time [15]. No relaxation would be observed in ideal elastic materials, whereas ideal viscous substances would relax instantaneously. Viscoelastic materials are relaxed gradually, the end point depending on the molecular structure of the material being tested. Stress in viscoelastic solids decays until to an equilibrium stress, while the residual stress in viscoelastic liquids tends to be around zero [14, 16]. Mechanical models or analogues have been developed to predict different patterns of viscoelasticity. The Maxwell model has frequently been used to interpret stress-relaxation data of a viscoelastic material [17]. A Maxwell element consists of a spring and a dashpot arranged in a series (figure 1a). The model involves a parallel coupling of a Hooke's body and *n* Maxwell's bodies [18] as shown in figure 1b.

The stress relaxation equation described by this mechanical model is given by equation 1.

$$\sigma(t) = \varepsilon \cdot \left(E_o + \sum_{i-1}^{n} E_i \exp\left(-\frac{t}{\lambda_i} \right) \right) \qquad (1)$$

In this equation, $\sigma(t)$ is the stress at any time during the relaxation test, ε is the initial constant strain. Because testing was conducted in uniaxial compression, E_i is equilibrium modulus (Young's modulus) of the *n* springs from the *n* Maxwell elements, and E_o is the equilibrium modulus for the lone ideal spring element. In addition, λ_i represents the relaxation times, which is defined in terms of the ratio between viscosity of the fluid from the *n* dashpots and the elastic modulus (equation 2):

$$\lambda_i = \frac{\eta_i}{E_i} \qquad (2)$$

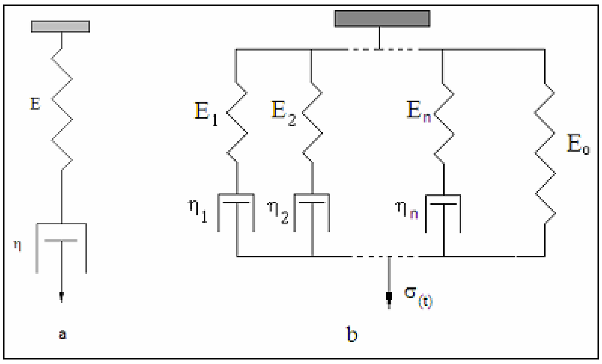

Figure 1. Mechanical models used to solve the experimental data from relaxation tests (a –Maxwell element; b – a mechanical model with *n* Maxwell elements and an ideal spring in parallel)

$$\frac{\sigma(t)}{\varepsilon} = E(t) = E_o + \sum_{i-1}^{n} E_i \exp\left(-\frac{t}{\lambda_i} \right) \qquad (3)$$

where *E(t)* is the equilibrium module of the sample from snail pâté. It exponentially decreases with the time of relaxation process.

At the initial moment, when t = 0,

$$E_{initial} = E_o + \sum_{i-1}^{n} E_i \qquad (4)$$

Finally, when $t \rightarrow \infty$, $E_{final} = E_o$

2.2. Experimental measurements. The ingredients for preparing three variants of pâté with different content of biogel, are presented in a previous paper [13]. Cans with snail pâté were storage at 4-6°C in refrigerator.

To determine the viscoelastic characteristics of snail pâté, stress relaxation had been use. The measurements were made at three different temperatures: 8°C, 14°C, and 21°C respectively. Using a cork borer, cylindrical specimens of snail pâté were prepared. The specimens had a diameter of 20 mm and their height was adjusted at 10-15 mm. The exactly height of the sample had been measured with a digital caliper Black & Decker. The temperature of samples has been measured with a portable Hanna Instruments K-thermocouple thermometer HI 935005.

To determinate the viscoelastic characteristics, by stress relaxation tests, a compression apparatus JTL Janz was used. Stress relaxation characteristics of the lubricated (using paraffin oil) cylindrical sample were determined by uniaxial compressing the sample up to a Cauchy strain of 0.30 at a compression rate of 6 mm · min^{-1}. When the compression was achieved at the desired level, the upper plate (a square with a surface of 9 cm^2) was stopped, and the pâté sample was allowed to relax for 120-180 s. The force at different relaxation times was continuously monitored. The TableCurve program has been used for non-linear and linear regressions and Origin program to plot experimental data and to draw the calculated stress-relaxation curves.

2.3. Statistical analysis. One-way analysis of variance (ANOVA) was used to determine differences among stress relaxation tests realized at different temperatures for samples of snail pâté with different content of biogel. Computations Tukey post hoc means comparisons and Levene's test for equal variance were also included. Resultant *P* values were rendered as significant ($P < 0.05$) or non-significant (ns). Degrees of significance were assessed based on $P < 0.05^{*}$ (significant), $P < 0.01^{**}$ (highly significant) and $P < 0.001^{***}$ (extremely significant).

The overall predictive capability of the mechanical model used could be commonly explained by the coefficient of determination (R^2), but R^2 is not a measure of the model's accuracy. Absolute average deviation (AAD) analysis is a direct method for verifying the model's suitability. The AAD is calculated by equation 5:

$$AAD = \left(\left(\sum_{i=1}^{n} \left(\frac{|y_{i,exp} - y_{i,cal}|}{y_{i,exp}} \right) \right) \cdot \frac{1}{n} \right) \cdot 100 \qquad (5)$$

where $y_{i,exp}$ and $y_{i,cal}$ are the experimental and calculated responses, respectively, and n is the number of the experimental runs. Evaluation of AAD and R^2 values together should check better the accuracy of the mechanical model [19].

3. Results and Discussions

A typical stress-relaxation curve for snail pâté is shown in figure 2. The stress-relaxation curves for all variety of snail pâté at the three studied temperatures are similar with the curve from figure 2. The same picture of these stress-relaxation curves for muscle tissues from Gilthead Sea Bream [7], Asian noodles [8], frozen cooked organic pasta [9], cassava dough [17], ice-stored cod [20], and acid milk gel [21] had been obtained.

Statistical tests were conducted to determine the number of Maxwell model terms (figure 1). The best correspondence between experimental data and calculated curve (eq. 1) was obtained for a model with three Maxwell elements (table 1) similar with the mechanical model from figure 1b.

For a model with one Maxwell element, the coefficient of determination (R^2) has the values (0.922 – 0.963) much less than the model with two (0.9919 – 0.9991) and three (0.9982 – 0.9997) Maxwell elements. On the other hand, the greatest values for AAD (2.53-3.75) could be observed for the model with one Maxwell element also. In the case of AAD appears a significant difference between the models with two (AAD = 0.335 – 1.148), respectively three (AAD = 0.135 – 0.375) Maxwell elements. The mechanical answer against an imposed deformation is described by equation 6, derived from equation 1:

$$\sigma(t) = \varepsilon \left(E_o + E_1 \exp\left(-\frac{t}{\lambda_1}\right) + E_2 \exp\left(-\frac{t}{\lambda_2}\right) + E_3 \exp\left(-\frac{t}{\lambda_3}\right) \right) \qquad (6)$$

The same model was used to analyze stress relaxation curves for muscle tissues from Gilthead Sea Bream (Sparus aurata L.) [7], frozen cooked organic pasta [9], and frozen stored Cape hake (M. capensis and M. paradoxus) [12].

In the stress-relaxation curve presented in figure 2, stress depends on time alone, with three zones: The initial portion shows a high slope, the third zone has the lowest slope and appears to approach a residual or an equilibrium value, whereas the second zone is intermediate between these two zones [22].

In the stress-relaxation curve of sample with 2.8% biogel, at 8°C, these three zones are showed clearly in figure 2, the first zone was in the range of 6.3–5.0 kPa, the second zone ranged from 5.0 and 4.1 kPa, and the third zone was between 4.1 to 3.4 kPa.

Table 1. R^2 and AAD values for a mechanical model with one, two and respectively three Maxwell elements

Biogel	Temperature	One Maxwell element		Two Maxwell elements		Three Maxwell elements	
		R^2	AAD	R^2	AAD	R^2	AAD
0	8	0.96119	2.769	0.99909	0.410	0.99965	0.135
	14	0.96264	2.809	0.99805	0.457	0.99815	0.383
	21	0.94966	2.650	0.99372	0.924	0.99956	0.261
1.81	8	0.94785	3.050	0.99655	0.812	0.99968	0.227
	14	0.92294	3.754	0.99192	1.148	0.99888	0.370
	21	0.95248	2.880	0.99694	0.877	0.99924	0.220
2.70	8	0.95349	2.936	0.99652	0.788	0.99956	0.280
	14	0.93926	2.534	0.99601	0.697	0.99746	0.375
	21	0.95436	2.617	0.99912	0.355	0.99943	0.174

Table 2. Viscoelastic characteristics of snail pâté calculated by non-linear regression from stress relaxation curves. A mechanic model with three Maxwell elements was used.

Biogel (%)	Temperature (°C)	□1 (s)	□2 (s)	□3 (s)	E_1 (kPa)	E_2 (kPa)	E_3 (kPa)
0	8	5.35(26) F=2.14	22.50±1.75 F=2.29	115.3±27.2 F=1.93	3.122(251) F=3.02	2.903(229) F=2.031	5.844(300) F=3.13
	14	2.73(20)***	13.23±2.46**	113.7±28.5 ns	2.990(115) ns	1.045(288)***	3.464(136)***
	21	0.33(4)***	5.44(36)***	56.1±3.9*	1.027(49)***	1.702(54)*	2.460(40)**
1.81	8	0.96(9) F=2.39	8.44(84) F=2.37	82.5±10.7 F=2.16	2.302(82) F=0.73	3.041(71) F=0.93	5.347(80) F=0.20
	14	0.69(4)**	6.15(33)**	73.3±4.7 ns	3.182(138)***	3.317(110)*	5.375(92) ns
	21	0.42(4)***	5.52(42)**	72.3±5.5 ns	2.169(122)***	2.853(130)**	4.310(101)***
2.70	8	3.96(29) F=2.13	24.31±11.56 F=3.14	161.8±16.1 F=2.86	2.424(92) F=1.65	3.497(72) F=3.07	7.410(92) F=3.33
	14	1.62(22)***	12.85±5.49 ns	109.2±4.9*	2.806(276) ns	1.752(432)*	4.356(221)***
	21	0.64(5)***	7.35(35) ns	90.5±30.8*	2.998(227) ns	2.022(885)*	3.742(587)***

Table 2.*(continued)*

Biogel (%)	Temperature (°C)	E_o (kPa)	$E_{initial}$ (kPa)	$\frac{E_o}{E_{initial}}$	□1 (kPa·s)	□2 (kPa·s)	□3 (kPa·s)
0	8	9.321(642) F=3.29	21.190	0.440	16.70(7)	65.32(40)	673.8±8.2
	14	6.488(267)***	13.987	0.464	8.16(2)	13.83(71)	393.9±3.9
	21	5.747(55)***	10.936	0.526	0.34(1)	9.26(19)	138.0(2)
1.81	8	7.946(144) F=0.71	18.636	0.426	2.21(1)	25.67(6)	441.1(9)
	14	8.005(156) ns	19.879	0.402	2.19(1)	20.40(4)	394.0(4)
	21	8.037(214) ns	17.369	0.463	0.91(1)	15.75(5)	311.6(6)
2.70	8	9.330(144) F=1.83	22.661	0.412	9.60(3)	85.01(83)	1198.9±1.5
	14	8.105(505)**	17.019	0.476	4.55(6)	22.51±2.37	475.7±1.1
	21	7.457(591)*	16.219	0.460	1.92(1)	14.86(31)	338.7±18.1

Table 3. The influence of the content of biogel on the values of relaxation times

	Temperature (°C)	Relaxation time (s)			
		0% biogel [C]		1.81% biogel	2.70% biogel
λ1	8	F=1.64	5.35(26);	0.96(9)***	3.96(29)***
	14	F=1.89	2.73(20);	0.69(4)***	1.62(22)***
	21	F=0.28	0.33(4);	0.42(4)^{ns}	0.64(5)***
λ2	8	F=3.89	22.50±1.75;	8.44(84)^{ns}	24.31±11.56^{ns}
	14	F=3.21	13.23±2.46;	6.15(33)^{ns}	12.85±5.49^{ns}
	21	F=0.16	5.44(36);	5.52(42)^{ns}	7.35(35)**
λ3	8	F=2.05	115.3±27.2;	82.5±10.7^{ns}	161.8±16.1**
	14	F=3.77	113.7±28.5;	73.3±4.7^{ns}	109.2±4.9^{ns}
	21	F=3.81	56.1±3.9;	72.3±5.5^{ns}	90.5±30.8^{ns}

[C] – control. Statistical significance is indicated relative to controls ($P < 0.05^{*}$, $P < 0.01^{**}$ and $P < 0.001^{***}$).

Table 4. The influence of the content of biogel on the values of elastic moduli

	Temperature (°C)	Elastic modulus (kPa)			
		0% biogel [C]		1.81% biogel	2.70% biogel
E1	8	F=2.62	3.122(251)	2.302(82)**	2.424(92)^{ns}
	14	F=2.30	2.990(115)	3.182(138)^{ns}	2.806(276)^{ns}
	21	F=2.71	1.027(49)	2.169(122)***	2.998(227)**
E2	8	F=3.20	2.903(229)	3.041(71)^{ns}	3.497(72)*
	14	F=2.21	1.045(288)	3.317(110)***	1.752(432)**
	21	F=3.90	1.702(54)	2.853(130)^{ns}	2.022(885)^{ns}
E3	8	F=2.93	5.844(300)	5.347(80)^{ns}	7.410(92)**
	14	F=1.89	3.464(136)	5.375(92)**	4.356(221)**
	21	F=3.86	2.460(40)	4.310(101)**	3.742(587)^{ns}
Eo	8	F=3.59	9.321(642)	7.946(144)*	9.330(144)*
	14	F=2.52	6.488(267)	8.005(156)**	8.105(505)^{ns}
	21	F=3.45	5.747(55)	8.037(214)***	7.457(591)^{ns}

[C] – control. Statistical significance is indicated relative to controls ($P < 0.05^{*}$, $P < 0.01^{**}$ and $P < 0.001^{***}$).

The parameters obtained by non-linear regression from the above mentioned model, E_1, E_2, E_3, E_o, λ_1, λ_2, and λ_3 are detailed in Table 2. From these values $E_{initial}$, $\frac{E_o}{E_{initial}}$, and the viscosities η_1, η_2, and η_3, were calculated and presented in the same table. It can be observed all values are influenced by temperature of the sample and the content of biogel in snail pâté. In addition, in table 2 are presented the degrees of statistical significance regarding the influence of sample temperature on the values of relaxation times and of the equilibrium modulus at all contents of biogel. Statistical significance is indicated relative to controls – samples with temperature of 8°C ($P < 0.05^{*}$, $P < 0.01^{**}$ and $P < 0.001^{***}$). At the same temperatures the values of the relaxation times increase in order $\lambda_1 < \lambda_2 < \lambda_3$. The first relaxation process occurs in the first portion of relaxation curve (figure 2) for $\lambda_1 < 6$ seconds, portion where is a marked reduction of the relaxation stress.

The third relaxation process happens in the final portion of the relaxation curve where a small reduction of the stress occurs. In the relaxation test, the individual variation of elastic modulus from equation 6 (E_1, E_2, E_3, and E_o) and of $E(t)$ are presented in figure 3 (for snail pâté with 2.7% biogel, at 8°C).

It can be observed the major contribution in the variation of $E(t)$ of the last term from the equation 6.

It can be observe that the relaxation time, for any of the biogel contents decrease with the increasing of temperature. The greatest statistical significance is for the first relaxation time ($P < 0.001^{***}$), and the smallest statistical significance is observed for the third relaxation time. For the third relaxation process, the influence of temperature on the values of relaxation time (λ_3) is without statistical significance.

At the same temperature of the sample, the influence of the content of biogel is better observed in the table 3.

Generally, the lowest values for the relaxation time have the samples with a content of 1.81% biogel. In the samples with biogel, the influence of the content of biogel on the values of the relaxation times is without statistical relevance.

The elastic moduli E_1, E_2 and E_3 were affected significantly by temperature, both in presence and in absence of biogel in the snail pâté (table 2). At the same temperature, the elastic moduli are not significantly influenced by the content of added biogel (table 4). The magnitude of σ_o, calculated as product of imposed deformation of the sample and the equilibrium modulus for the lone ideal spring element ($\sigma_o = E_o \cdot \varepsilon$), can be taken as a measure of the stiffness of the material [7].

Because for all samples the deformation was the same ($\varepsilon = 0.3$) the samples with the highest elastic modulus values are the stiffest materials. Appears from the table 2 the stiffest material is the snail pâté with 2.7% biogel. The absence of biogel corresponds to a less stiffness material.

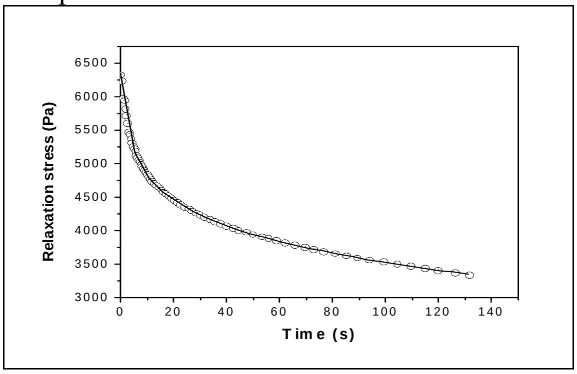

Figure 2. A typical experimental data and stress-relaxation curve for snail pâté with 2.7% biogel, at 8°C, fitted by the Maxwell model. Experimental data (○), fitted curve (continous line), $R^2 = 0.99956$, AAD = 0.280

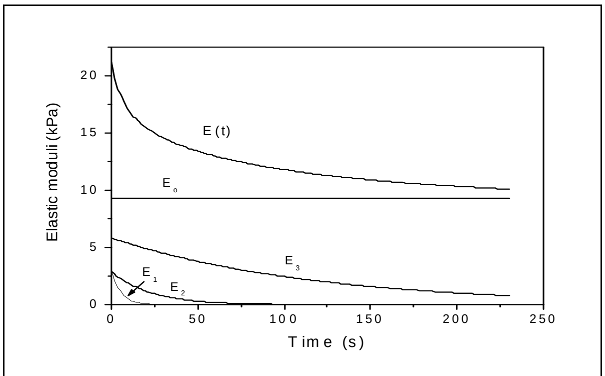

Figure 3. Variation of the elastic modului in the relaxation process, corresponding to the mechanical model with three Maxwell models, applied to the snail pâté with 2.7% biogel, at 8°C

For all varieties of snail pâté and at all studied temperatures, the calculated viscosities increase in the order $\eta_1 < \eta_2 < \eta_3$. As is expected, the viscosity decreases at the increasing sample temperature. At the same temperature, the greatest values of viscosities have the samples with 2.7% biogel. The ratio of the equilibrium modulus (E_o) to $E_{initial}$ (equation 4) could gives an information of viscoelastic characteristics of snail pâté. A less values of this ratio, means a more pregnant viscous nature of the sample. A greater value of the ratio, signify a greater elastic nature of the material [14]. Therefore, the snail pâté with 1.81% biogel is more viscous than snail pâté with 2.8% biogel or with no content of biogel.

4. Conclusions

A mechanic model with three Maxwell elements and an ideal spring in parallel is the best solution to analyze the relaxation curves. By nonlinear regression with a mathematical model corresponding to mechanical model was determined the values of the relaxation times and of the elastic moduli. From these viscoelastic characteristics was calculated the values of the viscosities. From the equilibrium modulus for the lone ideal spring element appears as the stiffest material is the snail pâté with 2.7% biogel. The snail pâté with 1.81% biogel is material that is more viscous.

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