

## VISCOELASTICITY OF THE EDAM CHEESE DURING ITS RIPENING

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### Abstract

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Series of the indentation of the ball (10 mm in diameter) by the constant speed into blocks of Edam cheese has been conducted. The indentation tests were performed at different speeds (1, 5, 10, 20 and 100 mm/min), and the corresponding force–displacement responses were fitted with an analytical solution to obtain the time-dependent constants and the instantaneous force–displacement response. The measurement has been performed for the cheeses of different stages of their maturity. The dependence of the indentation force on the penetration depth has been evaluated. This dependence can be fitted by a polynomial. The indentation force decreases with cheese fat content. It increases with the loading rate. Its value also decreases with the time of the cheese ripening. The recently proposed method for the indentation of the ball into viscoelastic solids has been used for our data analysis. This procedure, which needs the use of the numeric methods, enables to obtain stress relaxation moduli, which describe the viscoelasticity of the tested materials. The obtained moduli describe the stage of the cheese maturity.

Edam cheese, indentation, penetration, depth, relaxation, modulus, viscoelasticity

Cheese ripening is a complex process involving many physicochemical changes such as a change in pH, a progressive breakdown of the proteins to smaller polypeptides and the gradual accumulation of amino acids (Fox et al., 1993). Cheese texture may also vary with a change in the physical state of the fats that are already present in the cheese (Dufour et al., 2000; Watkinson et al., 1997). These changes can be described e.g. by the rheological properties of cheese. Rheological characterization of cheese is important as a means of determining body and texture characteristics and also for examining how these parameters are affected by composition, processing techniques and storage conditions (Konstance and Holsinger, 1992).

The most common method employed to study the mechanical properties of cheeses is the uniaxial force-compression: a constant rate of compression is applied to the sample and the resulting stress is continuously recorded. The method is suitable for the study of the rheological parameters of cheese on ripening and has been employed in French cheeses

(Antoniou et al., 2000), Cheddar cheese (Hort et al., 1997), Parmigiano Reggiano cheese (Noël et al., 1996), Swiss-type cheese (Bachmann et al., 1999) and Gouda cheese (Spangler et al., 1990). The former tests are used as standards for conventional structural materials because the measured forces and displacements can be converted into the stress–strain properties using simple theories, but they can be tedious and difficult when applied to soft foods because of the need to prepare specimen of specific size and shape. This problem can be solved using of the indentation tests.

Tests based on the indentation technique are popular in food texture evaluation because they do not require samples with strict shape requirements (Ozkan et al., 2002). However, because of the nonuniformity in the strain distribution, only limited theories that relate the indentation force–displacement response to the stress–strain properties are available (e.g. Sneddon, 1965; Sakai, 2002). As a result, indentation force–displacement measurements for many foods are interpreted empirically (Anand and Scan-

lon, 2002). However, there is a need for converting data from existing “empirical” tests into the fundamental material properties so that sensory assessment of foods can be improved (Bourne, 1994).

The objective of this study was to obtain viscoelastic properties of the Edam cheese from the indentation test. Spherical indenter, which simulates the actions of a cheese grader when “thumbing” a cheese, was investigated in this paper.

## MATERIALS AND METHODS

Commercially produced Edam cheeses (29 cm × 10 cm × 10 cm blocks) were manufactured by a company located in Jihlava. Cheeses have been produced on January 27, 2008. It was stipulated to the suppliers that the cheese should be typical of their normal production. Samples of cheese arrived at the Department at February 4, 2008. On reception at the Department, the cheese was stored at 12 °C. The blocks of cheese have been tested at 43<sup>th</sup> (March 10, 2008), 61<sup>th</sup> (March 28, 2008), 79<sup>th</sup> (April 15, 2008), and 108<sup>th</sup> (May 14, 2008) day after the production. The fat, moisture, salt content and pH of the cheese were evaluated by common procedures (Table I). In our experiments Edam cheeses with fat contents of 30 and 45 % in matter have been used.

Experiments were performed at 23 °C using the TIRATEST testing machine (Germany). Axisymmetric indentation tests were performed at five constant speeds of 1, 5, 10, 20 and 100 mm/min using

spherical indenter 10 mm diameters,  $D = 2R$ , respectively.

## EXPERIMENTAL RESULTS

In the Fig. 1 an example of the experimental record indentation force  $P$  versus the penetration depth  $h$  is displayed. The detail analysis shows that the experimental data can be fitted by the function:

$$P(t) = ah^3 + bh^2 + ch + d, \quad (1)$$

where parameters  $a, b, c, d$  are given in the Tables II–V.

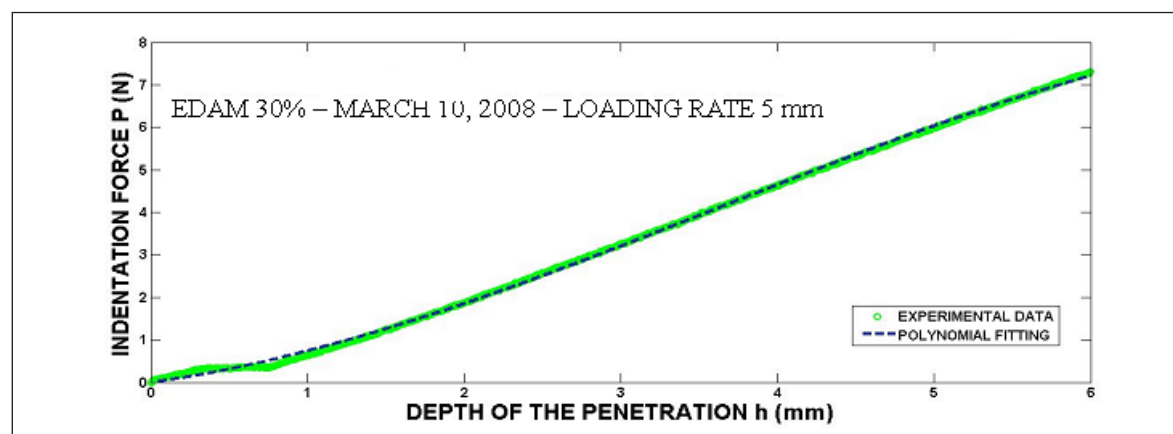
The indentation force increases with the decrease in the fat content – see example in the Fig. 2.

The indentation force increases with the loading rate – see example in the Fig. 3. The influence of the ripening time is shown in the Figs. 4–8. It is evident that for all loading rates the indentation force exhibits a decrease with the time of the cheese ripening. There are some exceptions at the early time of the ripening. In order to explain this effect some additional experiments are needed.

Now it is necessary to focus on the use of these data for the evaluation of the cheese viscoelastic properties. This problem is discussed in the next chapter.

I: Edam cheese composition

Edam type	Date	Dry matter [%]	pH	Fat [%]	NaCl [%]
30%	March 10, 2008	52.05	5.46	16.00	1.43
45%	March 10, 2008	56.46	5.58	25.75	1.29
30%	March 28, 2008	51.97	5.44	16.45	1.57
45%	March 28, 2008	57.49	5.58	26.50	1.37
30%	April 15, 2008	52.93	5.53	16.25	1.73
45%	April 15, 2008	55.63	5.63	25.63	1.79
30%	May 14, 2008	53.00	5.52	16.25	2.17
45%	May 14, 2008	57.11	5.57	26.75	2.10



1: The dependence of the indentation force on the penetration depth

II: Parameters of the polynomial fitting of the  $P(h)$ . Cheese tested on March 10, 2008.

EDAM type	Loading rate	a	b	c	d	R <sup>2</sup>
30%	1	-0.02100	0.2204	0.3068	-0.03257	0.9910
	5	-0.02479	0.2680	0.4851	0.00574	0.9950
	10	-0.02239	0.2452	0.8220	-0.11910	0.9960
	20	-0.02056	0.3164	0.1554	0.00667	0.9940
	100	-0.03702	0.4474	0.5287	0	0.9998
45%	1	-0.01017	0.1045	0.1886	0	0.9987
	5	-0.01860	0.1875	0.3156	-0.00997	0.9991
	10	-0.01108	0.1249	0.2305	0.04396	0.9993
	20	-0.01402	0.1590	0.2893	0.04491	0.9940
	100	-0.01197	0.1917	0.1644	0.09416	0.9998

III: Parameters of the polynomial fitting of the  $P(h)$ . Cheese tested on March 28, 2008.

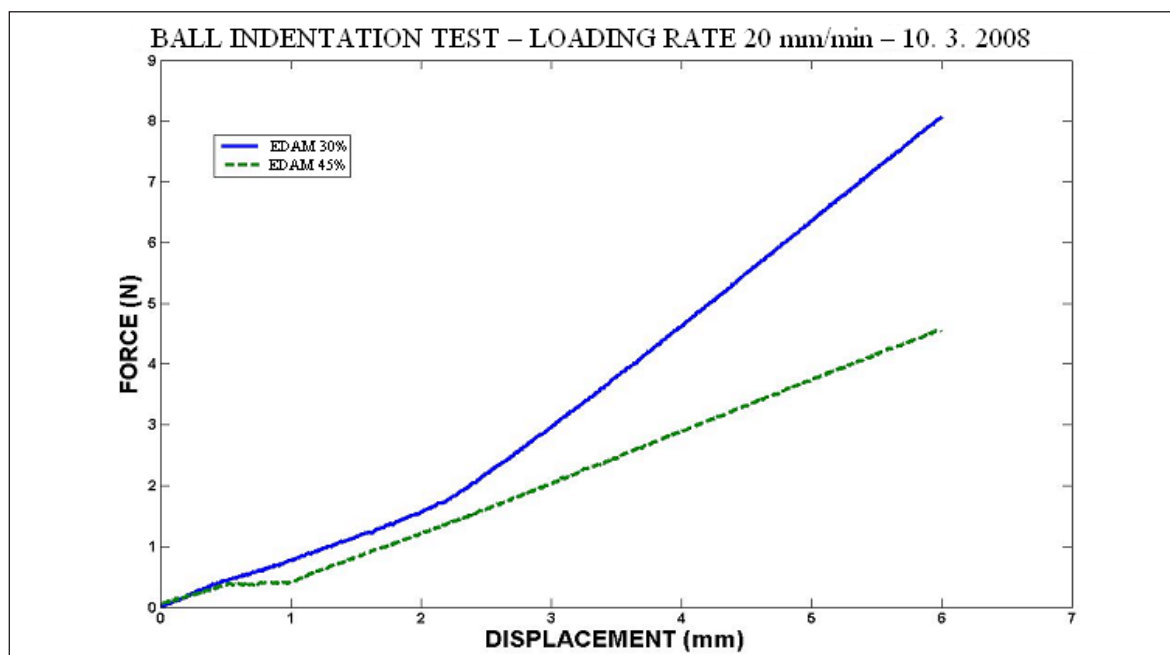
EDAM type	Loading rate	a	b	c	d	R <sup>2</sup>
30%	1	0.003958	-0.03751	0.8984	-0.1446	0.9985
	5	0.001974	-0.005166	1.1690	0.02030	0.9989
	10	-0.008963	0.13490	0.06969	-0.52850	0.9963
	20	-0.005421	0.10060	0.6557	-0.02214	0.9987
	100	0.0009304	-0.009417	0.5961	-1.96300	0.9999
45%	1	0.0009304	-0.009417	0.5961	0.01533	0.9999
	5	0.006919	-0.06142	0.9293	-0.05719	0.9990
	10	-0.032090	0.42880	-0.5979	0.11700	0.9959
	20	-0.004775	0.04302	0.6046	-0.01660	0.9959
	100	-0.029670	0.38460	-0.4066	0.02172	0.9960

IV: Parameters of the polynomial fitting of the  $P(h)$ . Cheese tested on April 16, 2008.

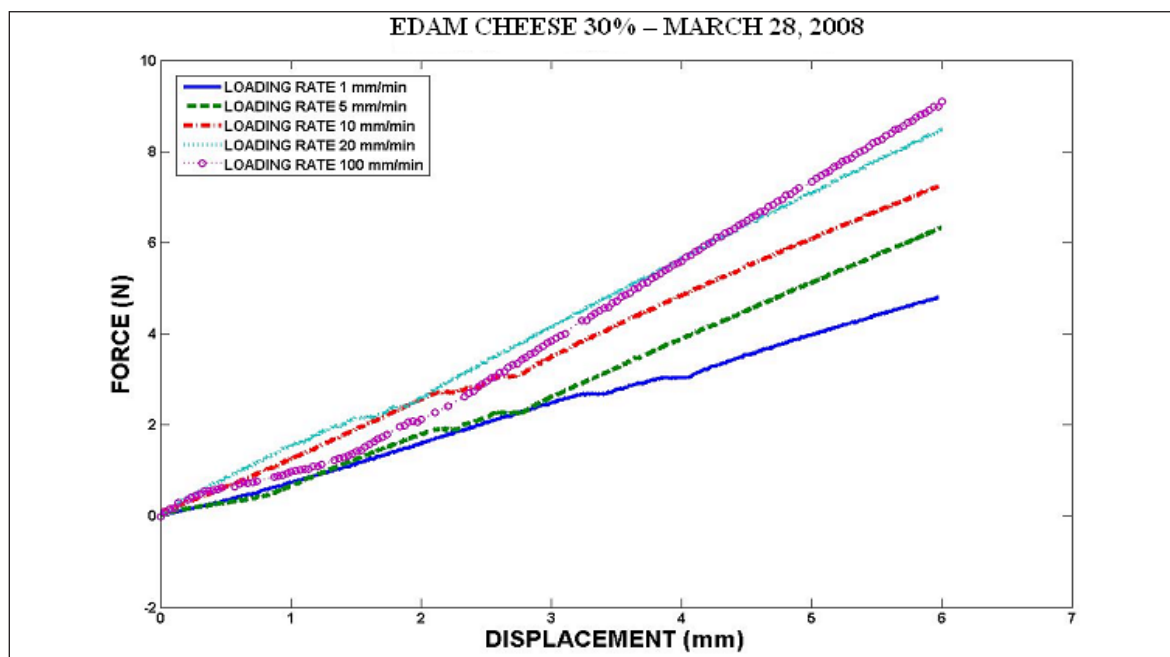
EDAM type	Loading rate	a	b	c	d	R <sup>2</sup>
30%	1	-0.005655	0.04701	0.7981	-0.04141	0.9999
	5	-0.002492	0.01076	1.2760	0.07194	0.9999
	10	-0.01005	0.10090	1.0680	-0.09581	0.9999
	20	-0.01162	0.15250	0.6751	0.24910	0.9997
	100	-0.01222	0.13040	1.0370	-0.19390	0.9999
45%	1	-0.003466	0.01606	0.5809	-0.05697	0.9998
	5	-0.003058	0.01024	0.8089	0.10680	0.9999
	10	-0.003507	0.02419	0.5933	-0.01446	0.9999
	20	-0.002595	0.01733	0.6883	0.03484	0.9999
	100	-0.002359	0.01266	1.4040	0.15780	0.9999

V: Parameters of the polynomial fitting of the  $P(h)$ . Cheese tested on May 14, 2008.

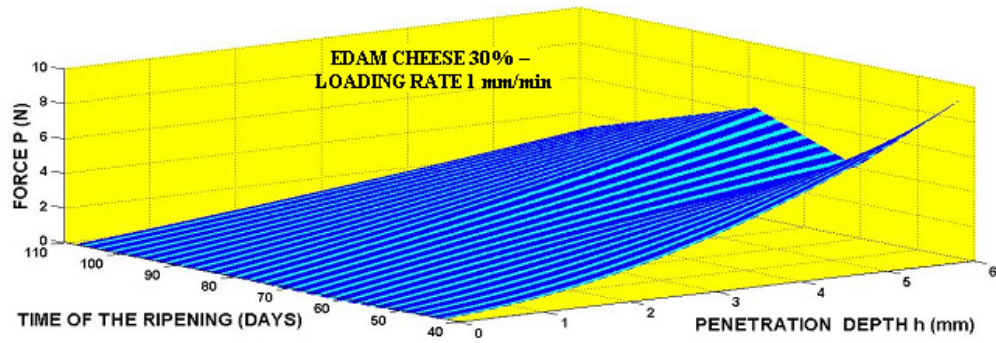
EDAM type	Loading rate	a	b	c	d	R <sup>2</sup>
30%	1	-0.00245	0.01858	0.4407	-0.00708	0.9998
	5	-0.00734	0.07599	0.5494	-0.09191	0.9999
	10	-0.00453	0.02904	0.9724	-0.0280	0.9999
	20	-0.00453	0.01174	0.9724	0.09387	0.9995
	100	-0.00886	0.0794	1.0690	-0.3153	0.9999
45%	1	-0.00882	0.07382	0.5018	-0.08236	0.9998
	5	-0.02033	0.1931	0.3686	0.15650	0.9995
	10	-0.02314	0.1591	0.9182	-0.02164	0.9999
	20	-0.01602	0.0810	1.2280	0.08917	0.9999
	100	-0.01521	0.1209	1.2320	-0.07458	0.9999



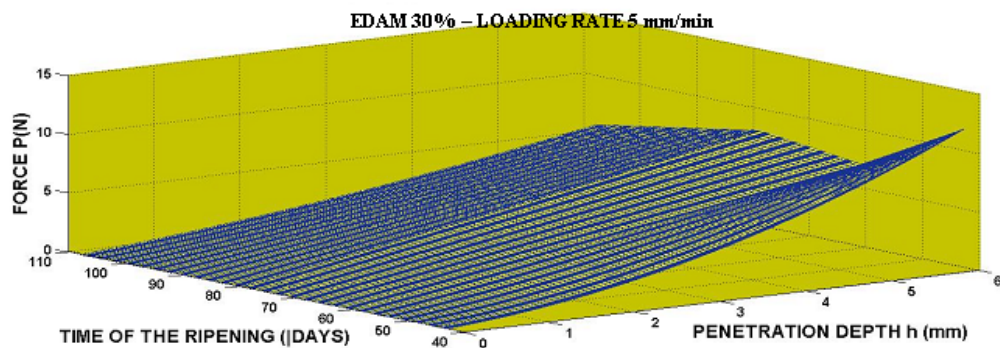
2: The influence of the fat content on the indentation force  $P$



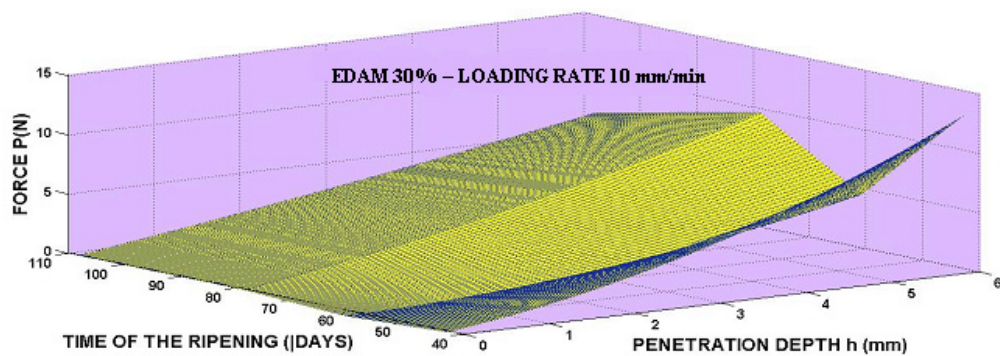
3: The influence of the loading rate on the indentation force



4: The influence of the time of the ripening on the indentation force

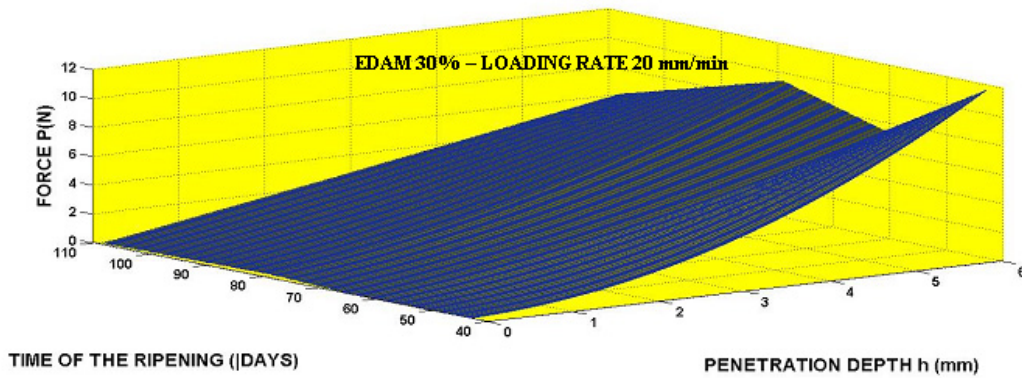


5: The influence of the time of the ripening on the indentation force

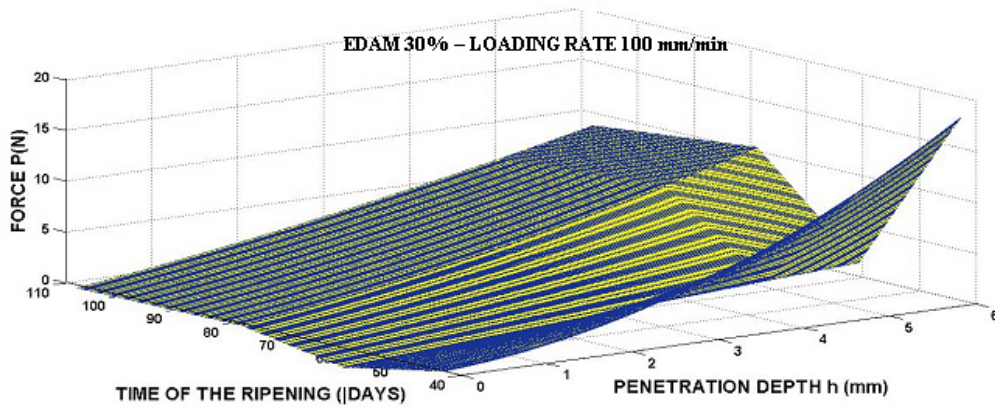


6: The influence of the time of the ripening on the indentation force





7: The influence of the time of the ripening on the indentation force



8: The influence of the time of the ripening on the indentation force

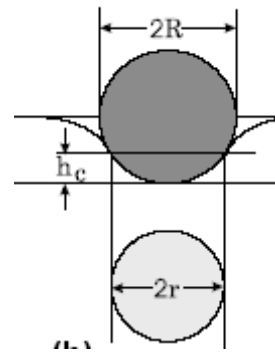
## DISCUSSION

The viscoelastic descriptions are made of time – dependent indentation load  $P(t)$  and the penetration depth  $h(t)$  resulting from any prescribed program of penetration and indentation loading, respectively.

The contact stress and strain in indentation problems, even for an elastic contact, are highly concentrated in the contact region, where extremely inhomogeneous deformations are taking place. Such a complex mechanical fields makes difficult to describe the constitutive relations of the applied force  $P$  to the internal stress, as well as the penetration depth  $h$  to the adjoin strains, however. In order to overcome these difficulties Meyer's principle of geometrical similarity can be used (Tabor, 1953). This procedure introduces the mean contact pressure  $\sigma$ , given by the ratio of the indentation load  $P$  to the projected contact area  $A_c$ ,  $\sigma = P/A_c$ . For a spherical indenter the representative stress is given by:

$$\sigma = \frac{P}{\pi r^2} \approx \frac{\gamma_s P}{2\pi R h},$$

where  $R$  is the radius of the spherical indenter,  $h$  represents the total penetration depth which is related to the contact depth  $h_c$  by  $h = \gamma_c h_c$  – see Fig. 9.



9: Schematic of the indentation geometry

The representative strain is defined as

$$d\varepsilon = k_s \frac{dr}{R},$$

where  $dr$  is the infinitesimal increment of the radius of circle of contact  $r$  – see Fig. 9. The frontal coefficient  $k_s$  is an indenter constant which can be obtained from the elastic solution (Sneddon, 1965). Its value is  $4/\pi$ . The value of  $\gamma$  is 2.

Let us consider an indenter pressed into contact with a linear viscoelastic body. During indentation loading penetration indent and its contact area both growths with time. The viscoelastic properties are described by the relaxation modulus  $E(t)$  instead of the Young modulus  $E$  for purely elastic materials. The stress increment is than expressed as

$$d\sigma(t) = \frac{1}{(1-\nu^2)} E(t-t') d\varepsilon(t'),$$

where use has been made of the assumption that Poisson ratio  $\nu$  of viscoelastic material is independent of time, for simplicity.

Substituting the representative stresses and strains and integrating the resultant equation with respect to  $t'$  in a hereditary manner from  $t' = 0$  to  $t' = t$ , the time dependent indentation load  $P(t)$  at the present time  $t$  resulting from any prescribed program of penetration can be obtained.

The equation has a form:

$$P(t) = \frac{\pi\sqrt{R}}{1-\nu^2} \frac{k_s}{2} \left(\frac{2}{\gamma_s}\right)^{\frac{3}{2}} \int_0^t E(t-t') \left[ \frac{dh^{\frac{3}{2}}(t')}{dt'} \right] dt'.$$

For a constant rate of penetration  $v_o$ , this equation can be expressed as:

$$P(t) = \frac{\pi\sqrt{R}}{1-\nu^2} \frac{k_s}{2} \left(\frac{2}{\gamma_s}\right)^{\frac{3}{2}} \int_0^t E(t-t') t'^{\frac{1}{2}} dt'. \quad (2)$$

The use of the Laplace transform leads to the solution for the stress relaxation modulus  $E(t)$ :

$$E(t) = \frac{4(1-\nu^2)}{\sqrt{R}k_s} \left(\frac{\gamma_s}{2\pi v_o}\right)^{\frac{3}{2}} L^{-1} \left[ s^{\frac{3}{2}} P(s) \right], \quad (3)$$

where  $P(s)$  is the Laplace transform of indentation load  $P(t)$  with the transform variable  $s$ . This transform is defined as

$$L[P(t)] = P(s) = \int_0^\infty P(t) e^{-ts} dt.$$

And the inverse transform is given as

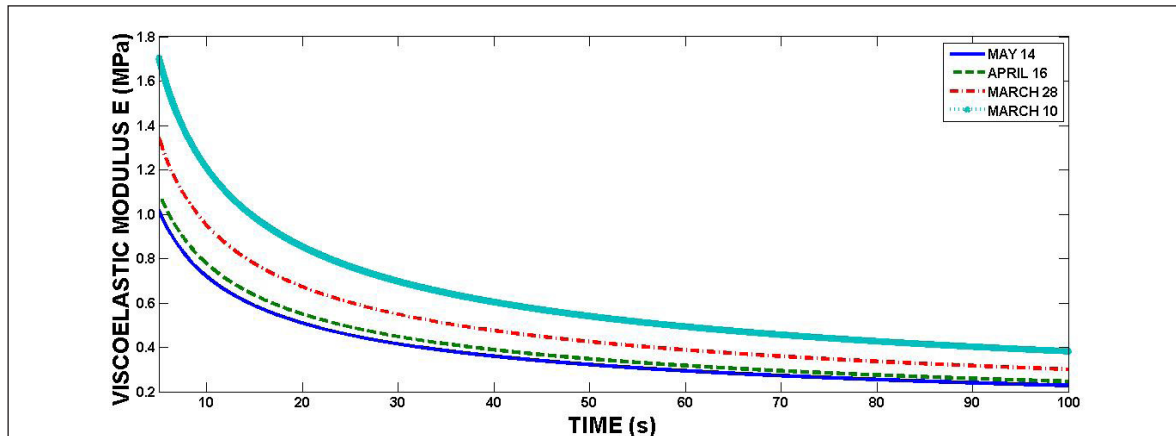
$$L^{-1}[P(s)] = \frac{1}{2\pi i} \int_{c-i\infty}^{c+i\infty} P(s) e^{st} ds.$$

The evaluation of this transform is very easy using of the MATLAB software. If we applied this procedure to the experimental data obtained in the previous chapter one must use the substitution  $h = v_o t$ , where  $v_o$  is the cross-head speed. The Laplace transform of the fiction given by the Eq. (1) is

$$P(s) = \frac{6av_o^3}{s^4} + \frac{2bv_o^2}{s^3} + \frac{cv_o}{s^2} + \frac{d}{s}.$$

In order to evaluate the fiction given by the Eq. (3) one must perform the inverse transformation of the function  $P(s)s^{\frac{3}{2}}$ . The use of the MATLAB software leads to

$$L^{-1} \left[ s^{\frac{3}{2}} P(s) \right] = 6av_o^3 L^{-1} \left( s^{-\frac{5}{2}} \right) + 2bv_o^2 L^{-1} \left( s^{-\frac{3}{2}} \right) + \frac{cv_o}{\sqrt{\pi t}} + dL^{-1} \left( \frac{1}{s^{\frac{1}{2}}} \right).$$



10: The influence of the time of the ripening on the viscoelastic moduli. Edam cheese – 30%.

It means this transform has no analytical form. It must be evaluated only numerically. In the Fig. 10 the time histories of the stress relaxation (viscoelastic) moduli are displayed.

The same dependence has been obtained for the cheese with 45% of the fat. It has been shown that these moduli are independent on the loading rate. It means these moduli represent viscoelastic properties.

## CONCLUSIONS

The paper contains results of the comprehensive study on the indentation test of the Edam cheese during its ripening. Instead of the ripening time the influence of the fat content and the loading rates has been also examined. The obtained results

suggest that the indentation force increases with the loading rates. Its value decreases with the fat content. The decrease in this force with the time of the ripening has been also reported.

The experimental data have been interpreted in terms of the theory of the indentation of the viscoelastic materials. It has been shown that this approach enables to obtain a general expression for the evaluation of the relaxation modulus which describes the viscoelastic properties. These moduli reflect the influence of the time of the ripening. They are also independent on the loading rate. It means these moduli can be used for the evaluation of the degrees of the cheese maturity. The indentation test thus represents the proper tool for the non-destructive testing of the cheeses.

## SOUHRN

Viskozita eidamského sýru během jeho zrání

Práce je zaměřena na studium deformačního chování sýrů (Edam 30 a 45 % tvs) pomocí nedestruktivních zkoušek, kdy je do bloku sýru vlačována kulička o průměru 10 mm. Během tohoto testu byly zaznamenány průběhy sil v závislosti na hloubce penetrace. Ukazuje se, že tato závislost může být popsána polynomem. Ukázalo se, že síla potřebná k dosažení určité hloubky penetrace roste s rychlostí zatěžování, která byla konstantní. Celkem bylo použito pět různých velikostí této rychlosti (1, 5, 10, 20 a 100 mm/min). Velikost této síly klesá s dobou zrání a je nižší pro sýr s vyšším obsahem tuku. Získané výsledky byly interpretovány pomocí teorie, kdy místo síly a hloubky penetrace používáme reprezentativních hodnot napětí a deformace. Byla adaptována teorie penetrace vyvinutá pro případ ryze elastické deformace na model viskoelastického chování, a to pomocí Boltzmannova integrálu, který zahrnuje vliv historie zatěžování. Byl navržen obecný vztah, který umožňuje získat tzv. relaxační modul, který nahrazuje modul pružnosti  $E$  a je závislý na čase. Ukázalo se, že tento modul klesá s časem a nezávisí na rychlosti zatěžování. Jeho průběh umožňuje popsat stupeň zrání sýru. Jeho hodnota ve všech časech klesá s dobou zrání sýru. Znalost této veličiny současně umožňuje hodnotit chování sýrů při jiných způsobech zatěžování (deformace vlastní tíhou, namáhání při dopravě, výpočet sil potřebných pro krájení apod.). Daná metodika je tak velmi vhodná pro stanovení této veličiny.

Edam, viskozita, zrání, zatěžování, penetrace

## SUMMARY

This work is focused on deformation behaviour of cheese (Edam with 30 and 45 % of fat in matter) by the non-destructive methods by the indentation of the ball (10 mm in diameter) by the constant speed into blocks. The corresponding force-displacement responses were fitted with an analytical solution to obtain the time-dependent constants and the instantaneous force-displacement response. The indentation tests were performed at different speeds (1, 5, 10, 20 and 100 mm/min). The measurement has been performed for the cheeses of different stages of their maturity. The indentation force decreases with cheese fat content.

The dependence of the indentation force on the penetration depth has been evaluated. This dependence can be fitted by a polynomial. It increases with the loading rate. Its value also decreases with the time of the cheese ripening. The concept of representative stress and strain fields at indentation contact enables one to derive straightforward the elastic constitutive equation. Furthermore, this approach leads to time dependent linear viscoelastic constitutive relations replacing the elastic modulus with the relaxation modulus in the elastic relations, and the integrating the resultant relations using Boltzmann's hereditary integral. The viscoelastic constitutive equation thus derived in general formulae was then utilized to describe time-dependent deformation and flow at the constant speed of the ball penetration into the tested cheeses. It has been approved that the indentation technique is very convenient for the evaluation of the cheese viscoelasticity.

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