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ON THE SELECTED RHEOLOGICAL PROPERTIES OF COMMERCIAL KETCHUPS

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Abstract

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The rheological properties of four different ketchups were determined. The viscosity, shear rate and shear stress were measured by use of Anton Paar DV-3 P Digital Viscometer and received data can be successfully characterized by several models, primarily by Hershel – Bulkley model. The ketchup was found to be a typical time-dependent thixotropic fluid. Shear stress and viscosity decrease with time of shear. Decrease during first 300 s is more significant than later decrease. The influence of spindle speed, shear rate potentially, on a sample viscosity was observed. The viscosity was rapidly changing during low speeds. The data were compared with Carreau model. Many other dependeces (low and high shear rates, difference between mixed and rested material etc.) were observed and modelled with satisfying results. Dependence of shear stress on shear rate under two temperatures was also examined. The tendency of increasing shear stress with increasing shear rate is evident. The samples were examined under temperature 22 °C and temperature 7 °C.

tomato ketchup, viscosity, strain rate, modelling

Tomato (*Lycopersicon esculentum*) is the world's second largest vegetable crop. Tomatoes may be consumed fresh or, because of its perishable nature, processed to give tomato juice, puree or paste. Tomato paste may also be an ingredient in other derivatives, such as tomato ketchup or sauces (Hayes et al., 1998). Tomato paste, the source material to produce a ketchup, is a dispersion of solid particles (pulp) in a aqueous media (serum) resulting from the concentration of tomato pulp, after the removal of skin and seeds containing at least 24% (w/w) of natural soluble solids. The raw material is manufactured by process that includes unit operations such as heat-treatment and pulping/fishing. As has been previously reported the rheological behaviour of tomato products is clearly dependent on processing variables. Thus, for example, these processing conditions have an indirect effect on the apparent viscosity and flow parameters, by influencing the concentration of water insoluble solids (WIS), the physicochemical characteristics of the WIS particles and serum viscosity (Valencia et al., 2002; Xu et al., 1986; Tanglertpaitbul and Rao, 1987; Gould, 1992; Rao and Cooley, 1992). The relevance of rheological properties in food process engineering is discussed in details in Velez (2002).

The importance of tomato ketchup as well as other food materials rheology has been emphasized in the last years and literature reflects such interest (Donel and Butler, 2002; Genovese and Lozano, 2001; Hayes et al., 1998; Sobolík et al., 2001; Wang and Kokini, 1994; etc.). The fundamental mechanical properties of ketchup are important in determining both the handling properties and the quality of finished products containing ketchup. The methods which have been developed for evaluating and characterizing rheological properties of fluids are quite numerous and somewhat varied. A number of authors have characterized the time-dependent viscosity of food products (Tiu and Boger, 1974; De Kee et al., 1983; Ramaswamy and Basak, 1991; Benezech and Maingonnat, 1993; Alonso et al., 1995; Chan et al., 1996). The relation between rheology and food texture is discussed in details.

This work is aimed on determining of basic rheological properties of tomato ketchup, such as viscosity, shear stress and various combinations of dependences of these values with time of shear, shear rate and time of relaxation.

MATERIAL AND METHODS

Anton Paar DV-3 P Digital Viscometer was used to measure dynamic viscosity, shear stress (τ), and shear rate ($\dot{\gamma}$). The DV-3 P is a rotational viscometer, based on measuring the torque of a spindle rotating in the sample at a given speed.

Shear stress is expressed in [g/(cm.s^2)], shear rate in [s^{-1}] and viscosity in [mPa.s] speed of spindle in revolutions per minute [rpm].

To measure rheological properties a special accesory, small sample adapter with TR9 spindle was used. The small sample adapter permits more accurate measurements than a standard spindles. The measuring range of the viscometer can be extended to lower values. Due to the parallel cylinder geometry shear rate and shear stress can be determined. Only a very small quantity of sample is required. In case of our set with TR9 spindles this volume represents 10.5 ml.

Four commonly commercially sold (on a Czech market) ketchups produced by different manufacturers were used. Samples of ketchup were marked with symbols K1 to K4. In the period of analysis the samples were stored at room temperature. All the ketchups contain similar ingredients; the list of ingredients follows later in this section. Detailed description of ingredients, their proportional representation as well as processing procedure were not included on the ketchup glass container, thus ketchups were taken as a complex individual materials. Although the ingredients are similar, even the basic sensorial comparison suggests very different rheological properties of given samples. The most visible difference is a sample fluidity.

Label list of ketchup ingredients follows:

K1: water, tomato puree, sugar, vinegar, modified starch E 1422, salt, spicy additive
K2: tomato puree, water, sugar, vinegar, modified starch E 1422, salt, citric acid E 330, spicy extract
K3: tomato puree, sugar, wine vinegar, salt, herbs, seasoning
K4: tomato puree, water, sugar, vinegar, modified starches E 1422 and E330, salt, spicy extract.

To standardize the handling procedure of the sample and as a way to impose the same rheological history on the structure, the sample was pre-sheared at 50 s^{-1} for 60 s allowed to rest for 90 s for thixotropic relaxation, and then the desired rheological test was performed. All the ketchups were first of all examined at room temperature 22 °C. Viscosity and shear stress were determined under constant shear rate and monitored with time of shear. Values of viscosity fluctuated with different ketchups. One ketchup – K4 – was chosen to make a deeper investigation.

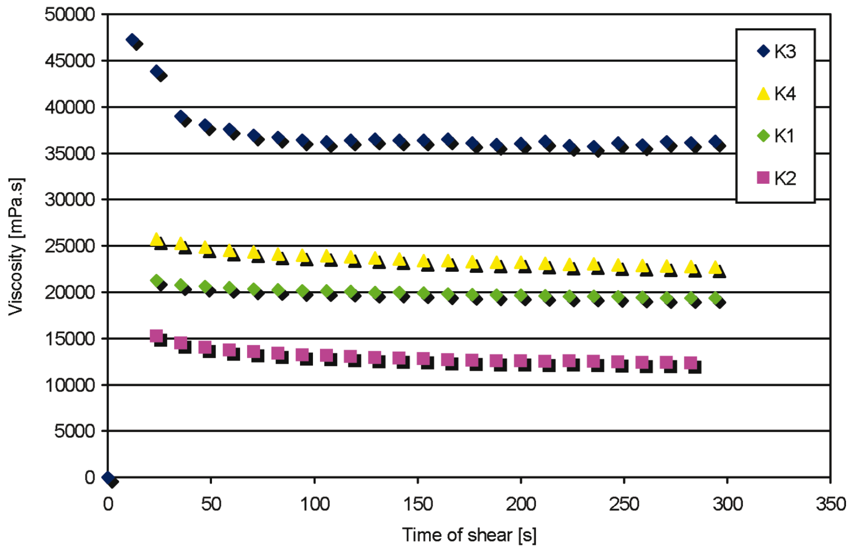
Next experiment describes time-dependent behaviour of K4 ketchup during rather long time of shear (3000 s) and decreasing values of viscosity and shear stress at constant shear rate. Ideally, time-dependent materials are considered to be inelastic with a viscosity function which depends on time. The response of the substance to stress is instantaneous and time-dependent behaviour is due to changes in the structure of the material itself. In contrast, time effects found in viscoelastic materials arise because of the response of stress to applied strain is not instantaneous and not associated with a structural change in a material (Steffe, 1996).

Following experiment was performed for determination of viscosity with changing spindle rate (0.1–200 rpm). Received data were fitted into Carreau model. The conversion of spindle speed in revolutions per minute [rpm] into shear rate in [s^{-1}] is following: spindle speed [rpm] / 2.9411 = shear rate [s^{-1}].

Next experiment is focused on evaluation of rheological behaviour in dependence on changing temperature. Dependence of shear stress on shear rate under two temperatures have been monitored.

RESULTS AND DISCUSSION

Fig. 1 shows viscosity as a function of shearing time at constant shear rate 1.7 s^{-1}, 5 rpm potentially. All four kinds of ketchup were examined. Time of shear was 300 s and this experiment time seems to be sufficient to determine basic quantity differences between single ketchups. The highest value of viscosity was exhibited by ketchup K3. After 100 s of shearing this quantity had a value of 36215 mPa.s. If we consider this value as 100%, the second ketchup – K4 – demonstrated at same time of shearing viscosity 23915 mPa.s, which means approximately 66% of maximum value. The statistical expression has this time an orientational meaning only and functions for general comparison of various materials. The third sample – K1 – shows the viscosity of 20155 mPa.s, which is 55.7% of maximum value. The lowest value of viscosity after given shear time was obtained for K2 ketchup and the value 13140 mPa.s represents 36.3%.



1: *Viscosity of different ketchups at constant shear rate 1.7 s^{-1}*

The viscosity values of all samples exhibited slight decrease with shear. Since the shear rate was relatively low, the decrease is not result of sample heating, but could be explained as a typical thixotropic behavior. Generally, the variances in viscosity values are partly caused by different composition and incurred differencies in dry matter content and other constituents.

Received data were fitted into different models, namely Casson model (Steffe, 1996), power law model (Steffe, 1996) and Herschel – Bulkley model (Steffe, 1996). The value of consistency coefficient K was constituted on 29.10 Pa.s^n and flow behaviour index n on 0.136. Especially Hershel – Bulkley model is very popular tool to describe rheological behaviour of food materials. An important characteristic of Herschel – Bulkley and Bingham plastic materials is the presence of the yield stress (σ_0) which represents a finite stress required to achieve flow. Below the yield stress a material exhibit a solid like characteristics. It stores energy at small strains and does not level out under the influence of gravity to form a flat surface. This characteristic is very important in process design and quality assessment for different food materials. Literature sources differ in values of σ_0 for ketchup also in dependence on measuring method. Several values and methods are contained in Tab. I.

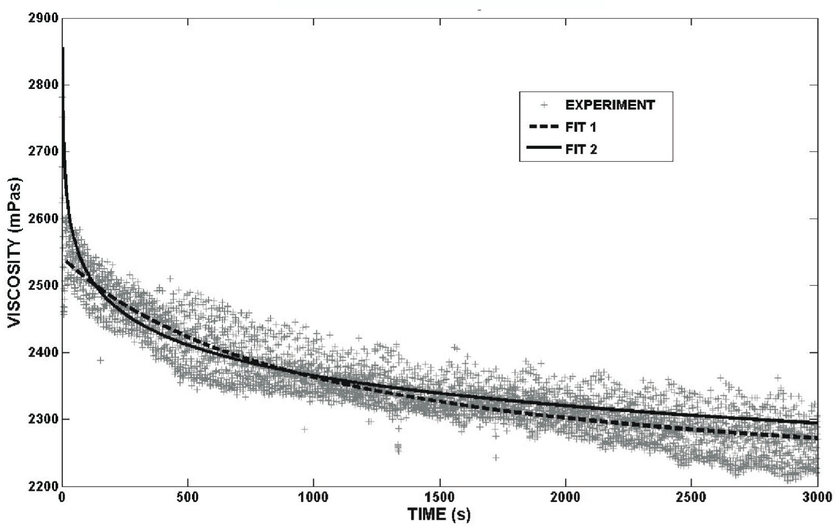
I: *Different measuring methods and values of σ_0*

σ_0 [Pa]	Measuring method	Literature source
15	Stress to initiate flow	De Kee et al. 1980
22.8	Extrapolation	Ofoli et al. 1987
18–30	Squeezing flow	Campanella et al. 1987
26–30	Vane method	Missaire et al. 1990

The Casson model was used with traditional exponent 0.5 although some authors (Rao 1999) received a good results for similar materials such as heated dispersion of tapioka starch with exponent 0.25. The power law model does not take account of yield stress, which is very importatnt rheological parameter. Herschel – Bulkley model is the most often recommended model to describe a flow curves of ketchup. All three models assigned a very good agreement

with measured data. The Hershel – Bulkley model with its highest degree of fit ($R^2 > 0.945$) seems to be the best method.

The dependence of viscosity on shear time is more obvious during longer testing time. Such relation is displayed in Fig. 2. Each "cross" in the graphic represents one measuring.



2: *Viscosity as a function of shear time*

$$\eta = \eta_\infty + \frac{\eta_0 - \eta_\infty}{1 + kt}, \quad (1)$$

$\eta_\infty = 2180$ mPas, $\eta_0 = 2542.3$ mPa, $k = 0.0009813$ s^{-1}, $R^2 = 0.8461$,

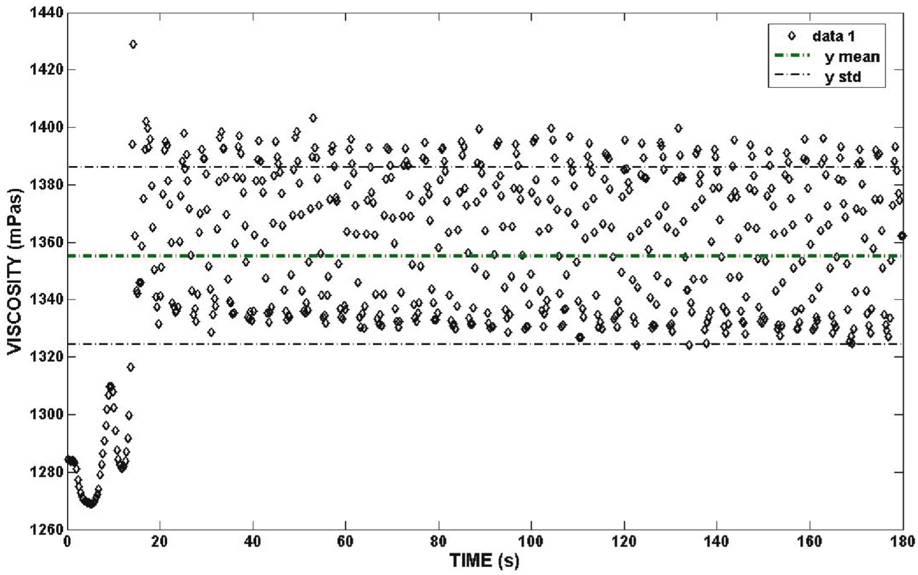
$$\eta = at^b + c, \quad (2)$$

$a = 3300$ mPas, $b = -0.02385$ $c = -444.4^1$, $R^2 = 0.8097$.

MATLAB software curve fitting application was used to process measured data and fit the curves.

The duration of shear was 3000 s this time, which is sufficient time to clearly determine the evidential tendency of viscosity value to decrease. Since the shear rate during this experiment was higher (34 s^{-1}), the part of decrease can be accounted to sample heating up. The ketchup sample was left to stand and the material structure was not affected by previous mixing. The viscosity was decreasing during a whole time of shear. The decrease is more intensive within first 300 s, where 1 s of shear represents decrease of $6.66 \cdot 10^{-3}$ % of viscosity and $1.53 \cdot 10^{-2}$ % of shear stress, while further shear is characterized by less intensive decrease of both viscosity and shear stress. Viscosity decreases, on average, for $2.66 \cdot 10^{-3}$ % per second and shear stress decreases for $2.55 \cdot 10^{-3}$ % per second.

Fig. 3 shows the history of K4 well mixed ketchup under constant 68 s^{-1} shear rate. The ketchup shows clear signs of strong oscillation with time.



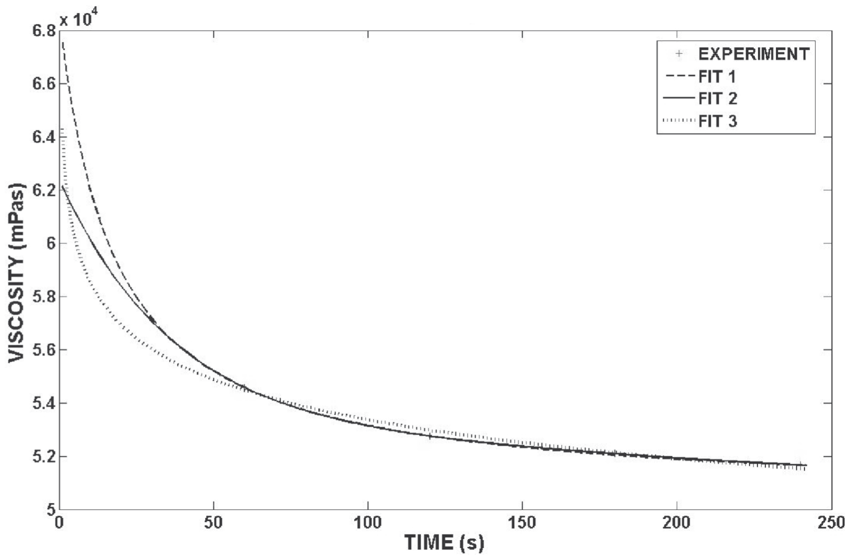
3: *Oscillating viscosity at 68 s⁻¹*

$$\eta = a_0 + \sum_{n=1}^{7}(a_n \cos(nwt) + b_n \sin(nwt)) \quad (3)$$

$a_0 = 1.118e4$, $a_1 = 2111$, $a_2 = –391.7$, $a_3 = –801.7$, $a_4 = –896.4$, $a_5 = –432.2$, $a_6 = –90$, $a_7 = 7.856$

$b_1 = –1880$, $b_2 = –228$, $b_3 = –1389$, $b_4 = –318.4$, $b_5 = 159.2$, $b_6 = 147.7$, $b_7 = 41.72$, $R^2 = 0.9917$.

Also the behaviour under very low shear rate values were observed. The example of this extensive investigation is in Fig. 4. where time/viscosity dependence for 1 rpm ($0.34^{–1}$) is displayed.



4: *Experimental and fiited data for shear rate 0.34 $s^{–1}$*

$$\eta = \eta_\infty + \frac{\eta_0 - \eta_\infty}{1 + kt} \quad (4)$$

η_∞ = 5.04 E4 mPas, η_0 = 6.863 E4 mPa, k = 0.05643 s^{-1}, R^2 = 0.9461

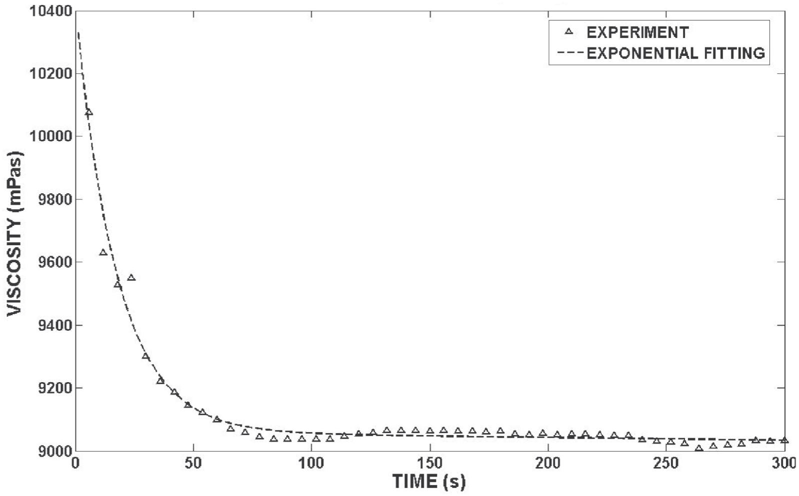
$$\eta = at^b + c \quad (5)$$

a = 5.979 e4 mPas, b = –0.0438, c = 4492 mPas, R^2 = 0.9838

$$\eta = a \exp(bt) + c \exp(dt) \quad (6)$$

a = 9230 mPas, b = –0.0273, c = 5.317E4 mPas, d = –0.001217, R^2 = 1.0

The behaviour of mixed and rested ketchup is obviously different and responses to the nature of material. Example of rested cold (7 °C) ketchup at 10 rpm (3.4 s^{-1}) is shown in Fig. 5.



5: *Rested cold (7°C) K4 ketchup at 3.4 s^{-1}*

$$\eta = \eta_\infty + \frac{\eta_0 - \eta_\infty}{1 + kt} \quad (7)$$

η_∞ = 9075mPas, η_0 9598.3mPas, k = 0.2493 s^{-1}, R^2 = 0.7612

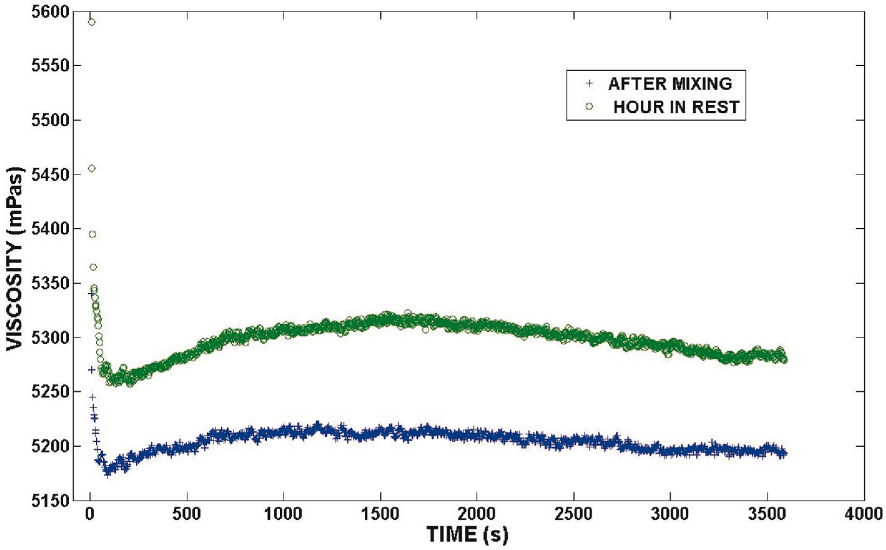
$$\eta = a \exp(bt) + c \exp(dt) \quad (8)$$

a = 1354, b = –0.05623, c = 9060, d = –9.538E-6, R^2 = 0.9723

$$\eta = \sum_{n=1}^{k} a_n \exp\left\{\left(-\frac{t - b_n}{c_n}\right)^2\right\} \quad (9)$$

a_1 = 2.89E4, b_1 = –134.7, c_1 = 76.21, a_2 = 9781, b_2 = 3.328E6, c_2 = 1.19 E7, R^2 = 0.9723.

Mentioned difference between well mixed and rested ketchup is demonstrated in Fig. 6. where time/viscosity dependance of K4 ketchup under 6.8 s^{-1} is diplayed. Two curves (mixed and 1 hour in rest) are presented and detailed specifications are listed in Tab II and Tab III.



6: *Mixed and rested K 4 ketchup at 6.8 s^{-1}*

Mixed material:
Gaussian distribution

$$\eta = \sum_{n=1}^{k} a_n \exp\left\{\left(-\frac{t-b_n}{c_n}\right)^2\right\} \quad (10)$$

$R^2 = 0.9021$

II: *Detailed specifications of mixed material curve*

a_1	29720
b_1	–164.1
c_1	73.22
a_2	7772
b_2	9193
c_2	2791
a_3	2479
b_3	4931
c_3	3731
a_4	3848
b_4	–845.1
c_4	5232
a_5	8082
b_5	1321000
c_5	912200

Material after 1 hour rest:

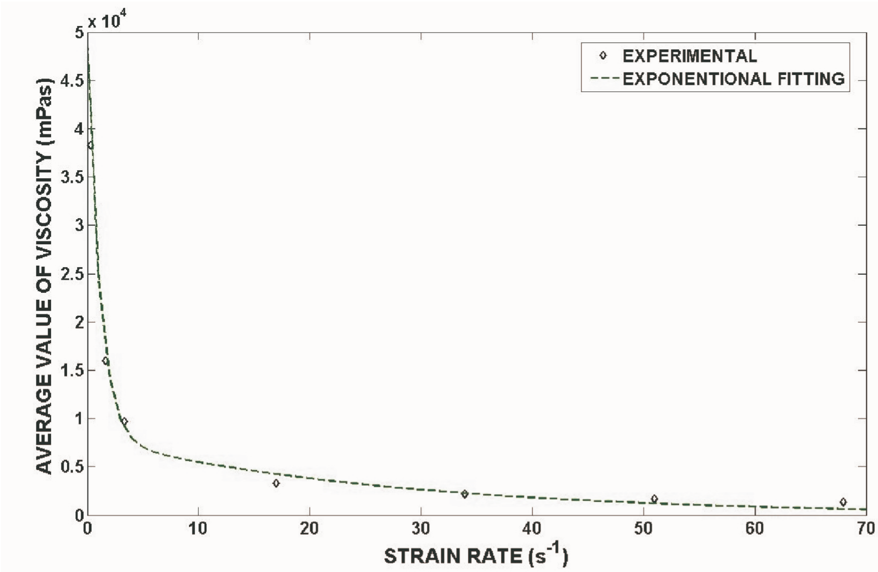
$$\eta = \sum_{n=1}^{k} a_n \exp\left\{\left(-\frac{t-b_n}{c_n}\right)^2\right\} \quad (11)$$

$R^2 = 0.9719$

III: *Detailed specifications of one hour resting material curve*

a_1	24400
b_1	–24.58
c_1	13.96
a_2	5288
b_2	8487
c_2	4793
a_3	2812
b_3	–2426
c_3	3343
a_4	12290
b_4	–359.3
c_4	164
a_5	3989
b_5	1764
c_5	4226

Next interest was focused on rate dependance (0.34 – 68 s^{-1}) of average value of viscosity. Such dependance is shown in Fig. 7 and concerns well mixed ketchup. Fitted curve according to later mentioned equation shows $R^2 = 0.9959$ correlation with measured data. The Y axis shows an average value of 5 measuring. Tab. IV. contains the experimental results and values of standard deviation of this test.



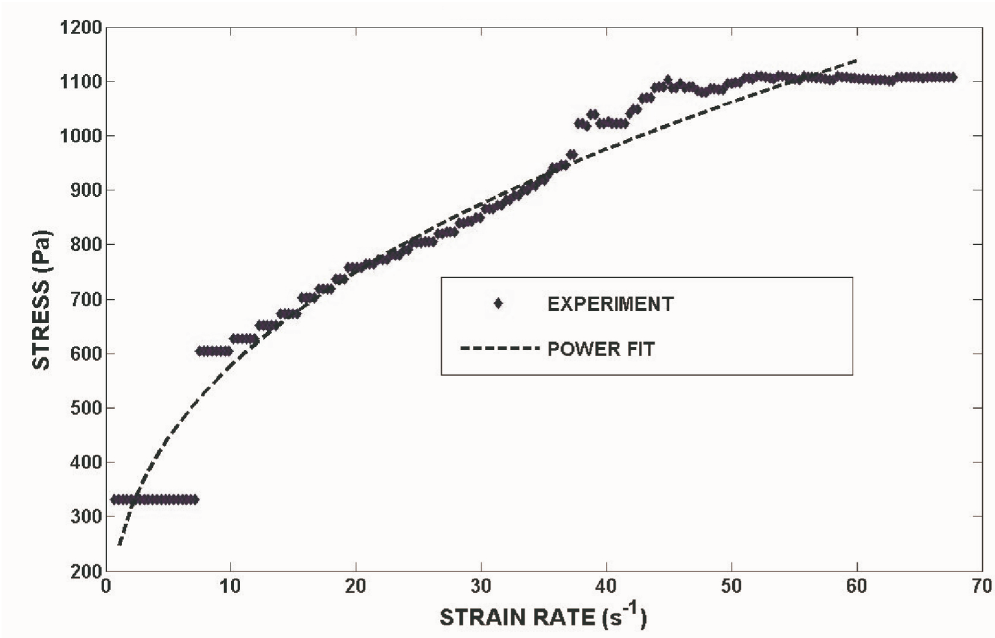
7: *K4 ketchup well mixed*

$$\eta = a \exp(b\dot{\gamma}) + c \exp(d\dot{\gamma}) \quad (12)$$
$a = 4.128e4$, $b = -0.9032$, $c = 7921$, $d = -0.03674$, $R^2 = 0.9959$.

V: *Experimental results and values of standard deviation*

rpm	STRAIN RATE (s^{-1})	Average viscosity (mPas)	Standard deviation (mPas)
1	0.34	38250	45.32
5	1.7	15960	88.37
10	3.4	9671	69.62
50	17	3332	97.21
100	34	2190	59.99
150	51	1651	40.37
200	68	1351	30.79

The dependance of shear stress on strain rate is an item of Fig.8. where well mixed K4 ketchup under wide range of strain rates was examined.

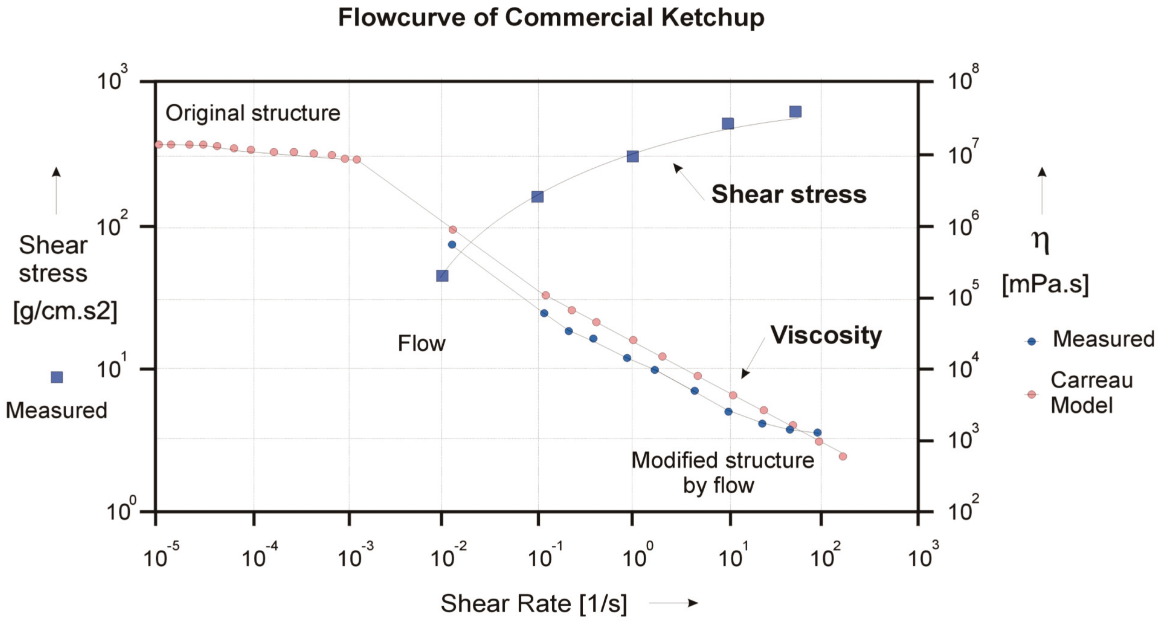


8: *K4 ketchup well mixed*

$\tau = K\dot{\gamma}^{n}$ (13)
$K = 241.2$, $n = 0.3778$, $R^2=0.9605$

$\eta = 289.5 - 0.2197\dot{\gamma}^2 + 26.9\,\dot{\gamma}$ (14)
$R^2 = 0.9722$

In order of confirming our measurements and computations we have compared our outcomes with models found in literature (Gaspar 2002). We have chosen the reological response of the sample to increasing levels of shear as it is shown in Fig. 9. The viscosity curve (according to Carreau model) clearly shows a Newtonian region that defines the consistency of a continuously changing structure. The viscous response could be accurately characterized by a Carreau mathematical model to reduce the information to a few significant variables (η_0, η_∞, p, a), where η_0 represents the initial viscosity of the original structure of the sample (also known as a zero shear viscosity), η_∞ represents the terminal viscosity (also known as a limiting viscosity), p presents the flow index and is associated with the pseudoplastic region and a represents a material consistency parameter. At specific conditions of temperature and pressure those variables, along with the yield stress σ_y and the critical maximum deformation γ_c, can be used for quality control, formulation, and product development, and other industrial purposes. For materials like ketchup, the Carreau mathematical model should be modified to include the yield stress property of material.



9: *Rheological behaviour for commercial ketchup at ambient temperature conditions. Viscosity vs. shear rate data fitted with a Carreau model*

Carreau Model $$\eta = \frac{\eta_0 - \eta_\infty}{[1 + [a\dot{\gamma}]^2]^2} + \eta_\infty \qquad (15)$$

Our measurements thus well correspond to previsously cited invesigators' deductions and findings and fit well into selected model.

Another problem to be examined is the influence of temperature. Rheological properties of food materials under different temperature conditions are studied in(Gaspar 2002, Steffe 1996). Food products are often processed at temperatures above 100 °C in sealed chambers to preclude boiling and drying (ketchup is processed at 125 °C), stored at sub-ambient temperature (5 °C) in sealed containers to extend a shelf life and stored in households at room temperature (25 °C), in sealed containers, and applied at room temperature out of the containers.

Concerning our results, the dependence of shear stress on shear rate under two temperatures was observed. The tendency of increasing shear stress with increasing shear rate was evident. The samples were examined under room temperature 22 °C and temperature 7 °C. The value of shear stress of colder sample was, on average, 2.5% higher.

At the moment our measuring devise is not equipped with thermoregulator, which would be powerful enough to provide data for correct monitoring of this dependence. To describe dependence of apparent viscosity vs temperature, the Arrhenius model seems to be appropriate (Juszczak et al., 2004).

CONCLUSIONS

Rheology testing is a very powerful tool to describe the structural nature and properties of food products. A different commercial ketchups exhibit slightly different rheological properties. Four examined ketchups varied in a value of viscosity and shear stress as it is documented and as it was described. Different models were applied to flow curves data. The best fit was exhibited for Hershel – Bulkley model with $R^2>0.945$ degree of fit. The viscosity and shear stress decrease with time of shear and tomato ketchup was found to be a time dependent thixotropic fluid. The decrease is more intensive during first 300 s.

The viscous response to increasing shear rate could be accurately characterized by a Carreau mathematical model. The experiment was performed in a range 0.1–200 rpm (0.034–70 s^{-1}) and received data fit very well into model.

Other tests such as long time shear, high and low rate shear or mixed and rested material shear were performed and modelled by use of MATLAB software. The results were satisfying and correlations ranged between $R^2 = 0.8–0.999$.

Increasing temperature causes reduction of viscosity. Higher temperature provides more internal energy, which increases the molecular mobility, consequently changing the viscosity and shear stress. Measuring under two temperatures (7 °C and 22 °C) were performed.

SOUHRN

O vybraných reologických vlastnostech komerčně distribuovaných kečupů

Práce je zaměřena na zkoumání reologických vlastností komerčně distribuovaných kečupů. Byly testovány čtyři druhy běžně dostupných kečupů a označeny jako K1–K4. Viskozita a smykové napětí byly stanoveny pomocí digitálního viskozimetru Anton Paar DV-3 P. Všechny čtyři kečupy vykazovaly částečně odlišné hodnoty viskozity a smykového napětí při stejných podmínkách zatěžování a stejné rychlosti deformace (rychlosti otáčení vřetene). Experimentální data byla srovnána s různými modely. Jako nejvhodnější se jeví model Hershel – Bulkley, kde hodnota R^2 byla vyšší než 0,945. Dále byly sledovány reologické vlastnosti během dlouhodobého zatěžování (3000 s). Kečup se choval jako typická tixotropní kapalina. Smykové napětí a viskozita klesaly s dobou zatěžování. Pokles během prvních 300 s byl výraznější než pokles během dalšího trvání experimentu. Byl též získán vliv rychlosti deformace 0.034–70 s^{-1} (počet otáček vřetene 0,1–200 rpm) na viskozitu vzorku a smykové napětí. Data velmi dobře korespondují s Carreauovým modelem.

Byla provedena řada dalších experimentů, kromě dříve zmíněných, kdy bylo sledováno chování kečupu při velmi nízkých a relativně vysokých rychlostech zatěžování, zkoumání vlastností rozmíchaných a odstátých kečupů atp. Výsledky byly vyhodnoceny pomocí programu MATLAB a výsledné závislosti prokládány vypočtenými křivkami. Korelace vypočtených dat byly velmi uspokojivé a dosahovaly ve většině případů hodnot nad R^2 = 0,95.

Dále byla získána závislost viskozity na rychlosti deformace při dvou různých teplotách (7 °C a 22 °C). Hodnota smykového napětí vzorku s nižší teplotou byla v průměru o 2,5 % vyšší oproti vzorku s vyšší teplotou.

rajčatový kečup, viskozita, rychlost deformace, modelování

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