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Shear work induced changes in the rheology of model Mozzarella cheeses

A thesis presented in partial fulfilment of the requirements for the degree

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Prateek Sharma

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With the blessings of

Grandparents

Late Smt. Dharmo Devi and **Late Sh. Mool Chand Sharma**, &

Parents

Smt. Leelwati Sharma and **Sh. Suresh Chandra Sharma**

I dedicate this thesis to

My beloved wife **Bharti Sharma**,

Son Master **Pratham Sharma**, &

the departed souls of my dear ones.

- Prateek Sharma

Abstract

Mozzarella cheese is a pasta filata type of cheese. Its manufacture includes a kneading – stretching step that creates a fibrous protein network and distributes fat-serum channels to attain desirable melt functionality on a pizza. During processing and manufacturing of pasta-filata cheese, large deformations take place. For appropriate characterization of a food material, rheological evaluation should be conducted in similar operating conditions, length scales and time scales to those taking place in the actual process. Development of the typical fibrous pasta-filata structure of mozzarella cheese depends on composition and process variables. Critical process variables in the development of cheese structure are time, temperature and shear. In this study we studied the effect of shear work on rheology, structure and melt functionality of model Mozzarella cheese.

Three types of model cheeses (full-fat, non-fat and full-fat with added tri-sodium citrate) were prepared by working cheese components together at 70 ^{o}C in a twin screw Blentech cooker. Varied amounts of shear work input (2.8-185 kJ/kg) were given to the cheese samples using 50, 150 and 250 rpm screw speeds. Samples were subjected to a range of rheological tests, confocal laser scanning microscopy, fat particle size measurements (DLS) and melt functionality evaluation.

While measuring steady shear viscosity of Mozzarella-type cheeses in a rotational rheometer at 70^{o}C, three main difficulties were encountered; wall slip, structural failure during measurement and viscoelastic time dependent effects. A flow curve method was successfully devised to measure steady shear rheology by using serrated plates as surface modification to avoid wall slip, giving enough measurement duration at low shear rate to avoid viscoelastic effects and selecting limited shear steps to cause minimum structural changes. These techniques enabled successful measurement of steady shear viscosity of molten Mozzarella-type cheeses at 70^{o}C at shear rates up to 250 s^{-1}.

Strong work thickening was observed for full fat Mozzarella cheese from steady shear rheology, oscillatory rheology, creep, elongational viscosity and tensile testing data. Steady shear rheology and melt functionality were found to be strongly dependent on total shear work input. An exponential increase in consistency coefficient (K from power law model) was observed with increasing amounts of accumulated shear work, indicating work thickening behaviour. An exponential work thickening equation is proposed to describe this behaviour. Excessively worked cheese samples exhibited liquid exudation, poor melting and poor stretch. Nonfat cheese exhibited similar but smaller changes after excessive shear work input.

At lower shear work inputs (<30 kJ/kg), cheese behaved like a viscoelastic liquid exhibiting typical entangled polymer melt behaviour with moderate frequency dependence and at excessive shear work levels (>70 kJ/kg) it behaved like a viscoelastic solid with low frequency dependence. A definite critical point for structural and viscoelastic transition was identified at a medium shear work level (~ 58 kJ/kg at 150 rpm). Similar viscoelastic property changes occurred in non-fat cheese suggesting that major changes were taking place in the protein matrix during working.

Confocal microstructures plus macroscopic observations showed systematic changes in structure with increased shear work inputs with unmixed buttery liquid observed at <5 kJ/kg, typical Mozzarella type microstructures (elongated fat-serum channels) at 6-15 kJ/kg and homogeneously distributed, small size fat droplets at >58 kJ/kg. At very high shear work inputs, > 75 kJ/kg, striations or anisotropy in the microstructures had disappeared and small micro-cracks were evident. Volume-weighted mean fat particle size decreased with shear work input and particle size distributions also changed. To account for the short and long term relaxation response behaviour, a 4-element Burger's model was found adequate for fitting the creep data of model cheese at 70 ^{o}C but a 6-element model was required at 20 ^{o}C. As shear work input increased, retarded compliance decreased and zero shear viscosity increased indicating the more elastic behavior of the cheeses with higher shear work input.

Fracture stress and strain for longitudinal samples from elongated full fat cheese did not vary significantly with shear work input up to 26.3 kJ/kg then decreased dramatically at 58.2 kJ/kg. Longitudinal samples with shear work input <30 kJ/kg, demonstrated significant strain hardening. At shear work inputs <30 kJ/kg strong anisotropy was observed in both fracture stress and strain. After a shear work input of 58.2 kJ/kg anisotropy and strain hardening were absent. Perpendicular samples did not show strain hardening at any level of shear work input.

A good correlation was found between the steady shear, oscillatory shear and transient rheological properties and the melting properties of the cheeses. The order for the rheological properties in terms of their sensitivity towards both shear work input and melt functionality is ηapp > G' > elongational viscosity > consistency coefficient, K. It was concluded that the dominant contributor to the changes in rheology, structure and melt properties with increased shear work was shear induced structural changes to the protein matrix. An increase in calcium induced protein-protein interactions after high shear work at 70 ^{o}C.

In summary, this thesis provides useful insights to shear work induced changes in material properties. It proposes useful linkages between the manufacturing process and the application of model Mozzarella cheese using appropriate rheological methods. Since the linkages were validated for only one composition and in only one processing environment, it is proposed that they should be tested in other conditions. In order to build a more complete picture, a molecular level study is proposed for future work to elucidate chemical changes during working and find appropriate linkages with physical and functional characteristics.

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List of Symbols

γ (t)	Shear strain at time t
$\dot{\gamma}_{A,}$	Apparent shear rate, s^{-1}
$\dot{\gamma}$	Shear rate, s^{-1}
γ_{end}	Shear strain at the end of the recovery phase
γ_r	Relative recovery, %
δ	Phase angle shift, degrees
ε_b	Biaxial strain
ε_f	Tensile fracture strain
ε_H	Hencky tensile strain
$\varepsilon_0, \gamma_{max}$	Maximum strain attained during creep phase
$\dot{\varepsilon}_b$	Biaxial strain rate, s^{-1}
$\dot{\epsilon}$	Strain rate in extension, s^{-1}
η	Shear viscosity, Pa.s
η^*	Complex viscosity, Pa.s
η^*_B	Biaxial extensional viscosity, Pa.s
η_0	Zero shear viscosity or viscosity before shear thickening, Pa.s
η_E	Tensile or extensional viscosity, Pa.s
η_T	Coefficient of viscous traction or extensional viscosity , Pa.s
η_∞	Infinite shear viscosity, Pa.s
η_{app}	Apparent viscosity, Pa.s
η_1 & η_2	Shear viscosity in viscoelastic region, Pa.s
η_b , η_B	Biaxial transient elongational viscosity, Pa.s
η_v	Newtonian viscosity, Pa.s
λ	Stress relaxation time or retardation time, s; Stretching of a fluid element
λ_1 & λ_2	retardation times of two retarded elements = $\frac{\eta_1}{G_1}$ and $\frac{\eta_2}{G_2}$, s
σ_b	Biaxial extensional stress, Pa
σ_f	Tensile fracture stress, Pa
τ	Shear stress, Pa
τ_0	Yield shear stress, Applied shear stress, Pa

τ_w	Wall shear stress, Pa
τ_1, τ_2 and T_{ret}	Retardation time, s
Λ	Lyapunov exponent of the flow
ω	Angular frequency, rad.s^{-1}
a_T	Shift factor in TTS
ΔP	Pressure drop, Pa
a,b	Work thickening constants
A_0	Initial cross sectional area of the specimen, m^2
B(t)	Time dependent compliance in bulk creep, Pa^{-1}
C	Constants in Table 2.1
D(t)	Time dependent compliance in tension or compression creep, Pa^{-1}
E(t)	Time dependent modulus in tension, Pa
E_a	Activation energy, J.$mole^{-1}$
F(t)	squeezing force at time t, N
F, F_N	Squeezing force, N
F_0	Baseline force in Blentech, N
G(t)	Time dependent modulus in shear, Pa
G*	Complex modulus, Pa
G’	Storage/elastic modulus, Pa
G’’	Viscous/loss/shear modulus, Pa
G_0	Initial modulus in stress relaxation, Pa
G'_{70}	Storage modulus at 70 ^oC
G_1& G_2	Viscoelastic moduli of two retarded elements, Pa
H(t)	Height at time t, m
H_0	Height at zero time, m
J_0	Instantaneous shear compliance, Pa^{-1}
J(t)	Time dependent compliance in shear creep, Pa^{-1}
J_1 and J_2	Retarded compliance, Pa^{-1}
K	Consistency coefficient, Pa.s^n
K(t)	Time dependent modulus in bulk compression, Pa
K_E, K_s, K_m	Constants in Table 2.1
$k_{elastic}$	Power index for frequency dependence of G’
K_{SH}	Strength coefficient , Pa

$k_{viscous}$	Power index for frequency dependence of G”
L	Capillary length, Distance of the force sensor from the rotating shaft axis, m
l_n	Length at time t, m
l_o	Initial length, m
m	Mass, kg
M (t)	Torque at time t, N.m
n	Flow behaviour index, degree of frequency dependence
n_{SH}	Strain hardening index (SHI)
Q	flow rate in the capillary, $m^3.s^{-1}$
R	Capillary radius, m; Radius of circular disc cheese sample used in LSF, m; Universal gas constant, $J.mole^{-1}.K^{-1}$
t	Time, s
T	Temperature, K
$T_{o,}$ T_{ref}	Reference temperature, K
T_R	Trouton ratio, dimensionless
V_z	Constant deformation rate, s^{-1}
W_s	Shear work, kJ/kg

List of Abbreviations

ASH	Apparent strain hardening
CSLM	Confocal scanning laser microscopy
DLS	Dynamic light scattering
FRDC	Fonterra Research and Development Centre
HBC	Hydrodynamic boundary condition
HDPE	High Density Polyethylene
LAOS	Large amplitude oscillatory shear
LLDPE	Linear low density polyethylene
LSF	Lubricated squeezing flow
LT	Loss tangent
LVDT	Linear variable differential transformer
LVE	Linear visco-elastic limit/range
MMC	Model Mozzarella cheese
MS	Melt score
RP	Rheological property
SAOS	Small amplitude oscillatory shear measurement
SER	Sentmanat Extensional Rheometer
SHI	Strain hardening Index
SHR	Strain hardening ratio
SIS	Shear induced structuring
SME	Specific mechanical energy, J/kg
SPR	Sliding plate rheometer
TSC	Tri-sodium citrate
TTS	Time-temperature superposition
VI	Viscoelasticity index
WLF	William-Landel-Ferry equation

List of Publications from PhD work

1. **Sharma, P.**, Munro, P.A., Dessev, T.T. & Wiles, P.G. (2016). Shear work induced changes in the viscoelastic properties of model Mozzarella cheese. International Dairy Journal, 56, 108-118.
2. **Sharma P**, Munro, PA, Dessev, T, Wiles, P, and Buwalda, RJ. (2016). Effect of shear work input on steady shear rheology and melt functionality of model Mozzarella cheeses. *Food Hydrocolloids*, 54, 266-277.
3. **Sharma P**, Dessev, T, Munro, PA, Gillies, G, Wiles, P, Golding, M, James, B & Janssen, P. (2015). Measurement techniques for steady shear viscosity of Mozzarella-type cheeses at high shear rates and high temperature. *International Dairy Journal*, 47, 102-108.
4. Bast R, **Sharma P**, Easton HKB, Dessev TT, Lad M & Munro PA (2015) Tensile testing to quantitate the anisotropy and strain hardening of mozzarella cheese. *International Dairy Journal*, 44, 6-14 (Appendix 1).
5. **Sharma P**., Munro, P.A., Gillies, G., Dessev, T. T., & Wiles, P. G. (2016). Creep behaviour and structure changes in model Mozzarella cheese during working, *Journal of Dairy Science*. (under review).

6. **Sharma P**., Munro, P.A., Dessev, T. T., Wiles, P. G. & Foegeding, E.A. (2016). Tensile fracture properties and strain hardening of sheared model Mozzarella cheese. *Journal of Dairy Science*. (submitted to Fonterra for permission to publish).

List of Conference Presentations from PhD work

1. **Sharma P**., Munro, P.A., Dessev, T., and Wiles, P (2016). Shear induced changes in rheology and structure of a model Mozzarella-type cheese. *2nd Food Structure and Functionality Forum Symposium – from Molecules to Functionality*, Singapore, 28 Feb.-2 March (Oral).
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4. **Sharma P**., Munro, P.A., Dessev, T., Wiles, P., (2015). Effect of accumulated shear work on the rheology, structure and melt functionality of a mozzarella-type cheese. 7th *International Symposium of Food Rheology and Structure*, ETH, Zurich, Switzerland, 7-11 June (Oral).
5. **Sharma P**., Dessev, T., Munro, P.A., Gillies, G., Wiles, P., Golding, M., James, B. and Janssen, P. (2014). Techniques to successfully measure steady shear viscosity of Mozzarella cheese. *IFT Annual Meeting and Food Expo*, New Orleans, Louisiana, USA, 21-25 June (Oral and Poster) (Appendix 3).
6. Munro, P.A., Bast, R., Dessev, T., and **Sharma, P.** (2013). Food structure design for optimum functionality. *NZ Conference of Chemical and Materials Engineering (NZCCME),* Auckland, New Zealand, 25–26 November (Oral).