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RHEOLOGICAL CHARACTERISATION OF AGE THICKENING IN MILK CONCENTRATES

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SUMMARY

This project investigates the time-dependent rheological behaviour of fresh and reconstituted milk concentrates.

New experimental protocols, including sampling and measurement techniques, as well as equipment calibration and data analysis procedures were developed for both the industrial surveys and controlled rheology experiments.

The controlled rheology experiments were mainly carried out on reconstituted milk concentrates to minimise the variation in composition of fresh milk. A new recombination rig was built which could minmise the age thickening process by mixing at 35°C and recirculating at 40,000 s^{-1} to break down the structure completely. This is the essence of this project, where age thickening is studied from a starting point of a fully broken down structure in contrast to past research. Using this method, the replicate milk concentrate samples had reproducible rheological behaviour, with a maximum reproducible error of 10%.

Age thickening involves two stages, a slow initial increase in apparent viscosity with storage time, followed by a sudden sharp rise which marks the onset of gelation.

The age thickening behaviour of milk concentrates is dependent on the processing variables prior to rheological measurement. These include solids content, shear rate and temperature during recombination, shear rate and residence time in the plate heat exchanger, and most importantly the raw material. The viscosity at the gelling point is an important characteristic of the age thickening process, and seems to depend mainly on the powder used, rather than the process treatments applied.

Industrial surveys exhibited similar trends, even under varying conditions that could not be completely controlled.

It is proposed that two types of age thickening phenomena can be distinguished: type I occurs below the temperature at minimum viscosity (65°C in this case), where weak

interactions take place between the casein micelles; type II occurs above the temperature at minimum viscosity, where additional stronger covalent bonds are formed, primarily due to the denaturation of whey proteins.

No mathematical model for the time-dependent rheology was developed. However, some important issues that must be taken into account during modelling were discussed.

The results showed that the age thickening process is more complex than had previously been envisaged. The knowledge of the interactions between the operating conditions, rheology of fresh concentrates and powder properties should be invaluable in the improvement of plant efficiency and quality control.

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LIST OF SYMBOLS

Roman letters		Unit
A	Empirical constant A in the Weltman model	Dimensionless
B	Empirical constant B in the Weltman model	Dimensionless
f_{ss}	Shear stress conversion factor	Dimensionless
f_{sr}	Shear rate conversion factor	Dimensionless
K	Consistency coefficient	$Pa.s^n$
L	Length of bob	m
m	slope of the logarithmic plot of $\ln N$ versus $\ln \tau_b$	$rpm.Pa^{-1}$
M	Torque	Nm
n	Behaviour index	Dimensionless
n''	Slope of a logarithmic plot of torque versus rotational speed	$Nm.rpm^{-1}$
N	Spindle speed	rpm
R_b	Radius of bob	m
R_c	Radius of cup	m
t	Time	s or min or hour

Greek Symbol		**Unit**
ε	Ratio of Rb/R_c	Dimensionless
ϕ	Volume fraction	Dimensionless
ϕ_{max}	Maximum volume fraction of the dispersed phase	Dimensionless
τ	Shear stress	Pa
τ_B	Baxter interaction parameter	Pa
τ_b	Shear stress at the surface of the spindle	Pa
τ_y	Yield stress	Pa
τ_e	Elastic stress	Pa
τ_t	Total stress	Pa
τ_v	Viscous stress	Pa
μ	Viscosity	Pa.s
μ_{app}	Apparent viscosity	Pa.s
μ_s	Viscosity of the solvent	Pa.s
$[\mu]$	Intrinsic viscosity	Pa.s
$\mu_{normalised}$	Normalised viscosity	Dimensionless
$\mu_{high\ shear}$	Steady viscosity attained at high shear rates	Pa.s
μ_{∞}	Viscosity at infinite shear rate	Pa.s
$\mu_{\infty ad}$	Adjusted extrapolated viscosity at infinite shear rate	Pa.s
γ_e	Elastic strain associated with the flow structure	Dimensionless
$\dot{\gamma}$	Shear rate	s^{-1}
$\dot{\gamma}_b$	Shear rate at the surface of the spindle	s^{-1}
COV	Coefficient of variation	Dimensionless

LIST OF ABBREVIATIONS

β-lg	β-lactoglobulin
CCP	Colloidal calcium phosphate
CIP	Cleaning-in-place
COV	Coefficient of variation
DFGM	Damaged fat globule membrane
HCT	Heat coagulation time
HLA	Hysteresis loop area
NEM	N-ethylmaleimide
NFGM	Native fat globule membrane
PHE	Plate heat exchanger
PSD	Particle size distribution
RAT	Rate of age thickening
RSCM	Recombined sweetened condensed milk
SCM	Sweetened condensed milk
SDS-PAGE	Sodium dodecyl sulphate polyacrylamide gel electrophoresis
SNF	Solids non-fat
TASMP	Te Awamutu skim milk powder
TAWMP	Te Awamutu whole milk powder
TEM	Transmission electron microscopy

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