THE COLD EXTRUSION OF CHOCOLATE

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We report experimental observations on the plastic extrusion of a milk chocolate where the material starts in the solid state and is extruded isothermally below its normal melting point. Continuous extrusions were achieved and it is shown that it is possible to produce rods, tubes and injection moulded articles using this process. A time dependent, post processing plasticity of the extruded material was also observed for the material and this, together with the other information suggests that the extrusion process occurs as a consequence of isothermal shear induced crystal melting of the fat matrix that surrounds the sugar crystals within the chocolate.

Keywords: extrusion; chocolate; rheology; plastic flow

INTRODUCTION

Chocolate processing at present is carried out from the molten state. Following a melt conditioning process, known as tempering, the molten chocolate, which is usually at a temperature of about 27–32°C is then either poured onto a sweet centre or into a mould. Subsequent solidification and crystallisation then occurs as the molten chocolate cools (see for example Beckett, 1988). In the molten state the chocolate has been described as a Casson fluid (Chevalley, 1975 and 1988) where the shear stress τ is given by

$$\tau^{1/2} = \tau_o^{1/2} + k\dot{\gamma}^{1/2}$$

τ_o is the fluids yield stress and k a viscosity coefficient, $\dot{\gamma}$ is the applied shear rate. The yield stress τ_o depends on the type of chocolate used and is typically of order 10–200 Pa. The viscosity coefficient k^2 is of order 10–200 Pas which can give an apparent viscosity of order 121 Pas at a shear rate of 10^2 s^{-1}, if $\tau_o = 100$ Pa and $k^2 = 100$ Pas.

The composition of chocolate is shown schematically in Figure 1. Often, a volume fraction of between 45–55% sugar crystals are present and typically the mean mass particle size of the 'inert' sugar crystals is of order 20 μm, which is equivalent to 2 μm on a number basis. The surrounding matrix, in the case of plain chocolate, consists of cocoa butter which is either in the molten, semi crystalline or crystalline state. Plain chocolate also contains additional cocoa solid particles. Milk chocolate includes additional butter fat and milk solids, whilst white chocolate is the same, but does not include the cocoa solid (non fat) particles.

In this paper we introduce an alternative method to melt processing of chocolate. We believe the 'cold extrusion' process that is described in this paper is novel and has potential use as a commercial process route. Our objective is to process chocolate in a physical state where its general deformation behaviour is of a plastic nature rather than that of a viscous fluid. The high phase volume of sugar crystals in chocolate suggest that it might be processed in a rather similar way to ceramic and other food pastes (see for example, Benbow and Bridgwater, 1993), where the inorganic particles are surrounded by a plasticising viscous matrix. In addition, it has been shown that it is possible to extrude certain polymers below their normal melting range and by processing in this way, precision section extrusions can be obtained (Kanomoto *et al.*, 1987 and 1988, Anton *et al.*, 1987). From these examples a clear advantage in semi solid state extrusion can be identified in that the shape retention of the extrusion can be maintained immediately the material has left the extrusion die.

In this paper, we present experimental observations on the way initially solid chocolate can be extruded through a die. Most of our results were obtained using a capillary rheometer and further experiments were carried out using a hydraulically driven ram extruder and injection moulding machine.

CAPILLARY RHEOMETER EXPERIMENTS

Piston driven extrusion was carried out using a modified Davenport capillary rheometer which is shown schematically in Figure 2. Solid chocolate buttons of order 10 mm in diameter and 3 mm in thickness were compacted into the barrel of the rheometer and an isothermal extrusion was carried out at a constant piston speed. The diameter of the extruder barrel is 19.5 mm and the barrel is temperature controlled by means of electrical heaters surrounding the outside of the barrel. Extrusion took place through different dies having a range of *L*/*D* ratios and entry angles, as shown in Figure 2b. The piston speed could be controlled by means of a variable speed electric motor driving through a gearbox. The rheometer is capable of precision and constant piston speeds which in this paper are recorded as a volumetric displacement rate.

For any given extrusion, the upstream pressure was logged as a function of time and a schematic diagram of a

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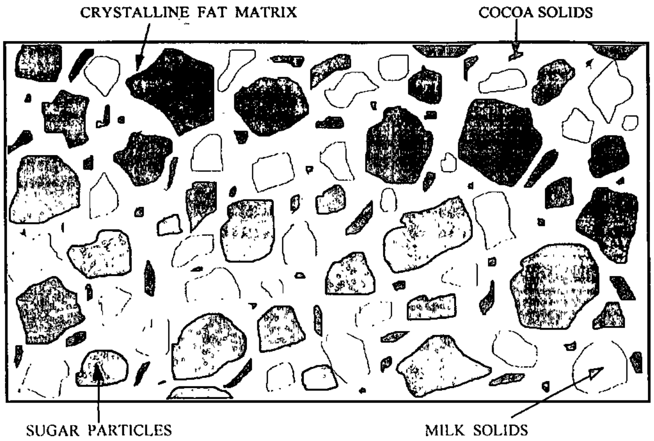

TYPICAL COMPOSITION

SUGAR CRYSTALS	:	50% WT
MILK SOLIDS	:	15% WT
COCOA SOLIDS	:	5% WT
TOTAL FAT	:	30% WT

Figure 1. Schematic diagram depicting the structure of chocolate.

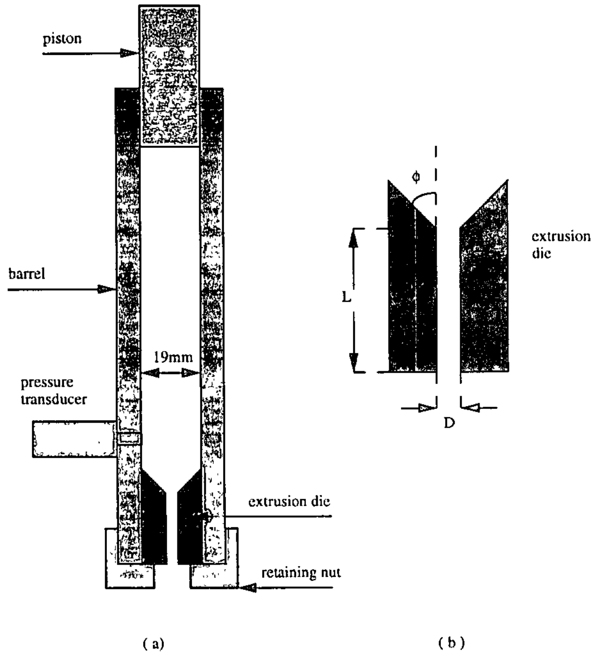

Figure 2. Schematic diagram of the Davenport Rheometer. (a) extrusion geometry; (b) die configurations.

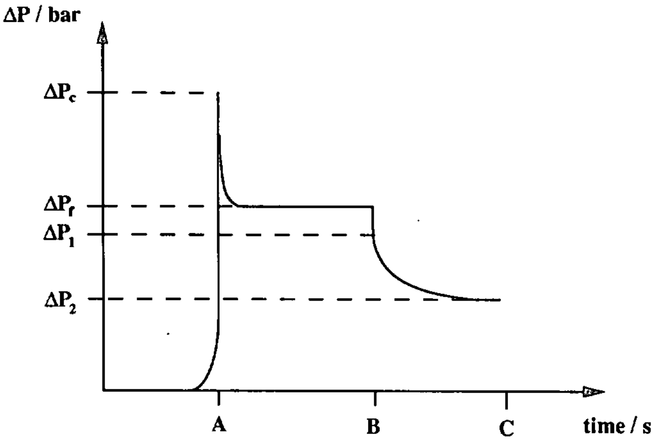

Figure 3. Schematic diagram of extrusion pressure profile plotted as a function of time.

typical pressure trace is shown in Figure 3. After initial compaction, the chocolate often went through a yielding process (ΔP_c in Figure 3. This could occur over a very short period of time (i.e. less than one second) and the data logging programme had to be modified in order to increase the frequency of measurements when large changes in the pressure profiles occurred. When flow of the material commenced a generally constant 'steady state' extrusion pressure ΔP_f was monitored and in this regime the measured pressure did not change significantly as the piston advanced down the barrel. This region is shown schematically between points A and B on Figure 3. On the cessation of the piston movement, (indicated by point B in Figure 3), the subsequent pressure trace could take a number of different forms depending mainly on the value of the extrusion temperature. We divide the pressure relaxation into three components. An 'instantaneous' viscous relaxation ΔP_f to ΔP_l. A longer time scale, viscoelastic relaxation ΔP_l to ΔP_2 and finally an essentially time independent residue plastic extrusion pressure ΔP_2. We define a viscous fraction VF for the pressure relaxation as being given by

$$VF = \frac{\Delta P_f - \Delta P_l}{\Delta P_f} \tag{1}$$

The value of VF then enables us to estimate the fraction of the stress that can be associated solely with a viscous component of flow.

We have conducted experiments using white, plain and milk chocolate samples and found that the basic qualitative effects to be reported are similar, however the extrusion pressure profiles can vary. In this paper we report data for a single variety of milk chocolate. This material has a 29% volume fraction of cocoa butter and butter fat and a 45% volume fraction of sugar crystals. Before solidification the starting material had been tempered. We have found that for any given set of extrusion conditions and chocolate, the pressure profiles are reproducible to within an accuracy of about 10%. The most sensitive parameter appears to be temperature and we estimate that the experiments carried out in this work were accurate to within 0.5°C.

The Effect of Temperature

For a given die geometry and material, the main process variables are temperature and volumetric flow rate. In order to describe the basic effect we have chosen three extrusion temperatures and followed the extrusion behaviour initially at one extrusion speed for a 4 mm diameter die of length 31 mm.

There is a distinct processing window using this system within which the cold extrusion of chocolate can take place and this is illustrated in Figure 4 where the extrusion profiles at three different temperatures are

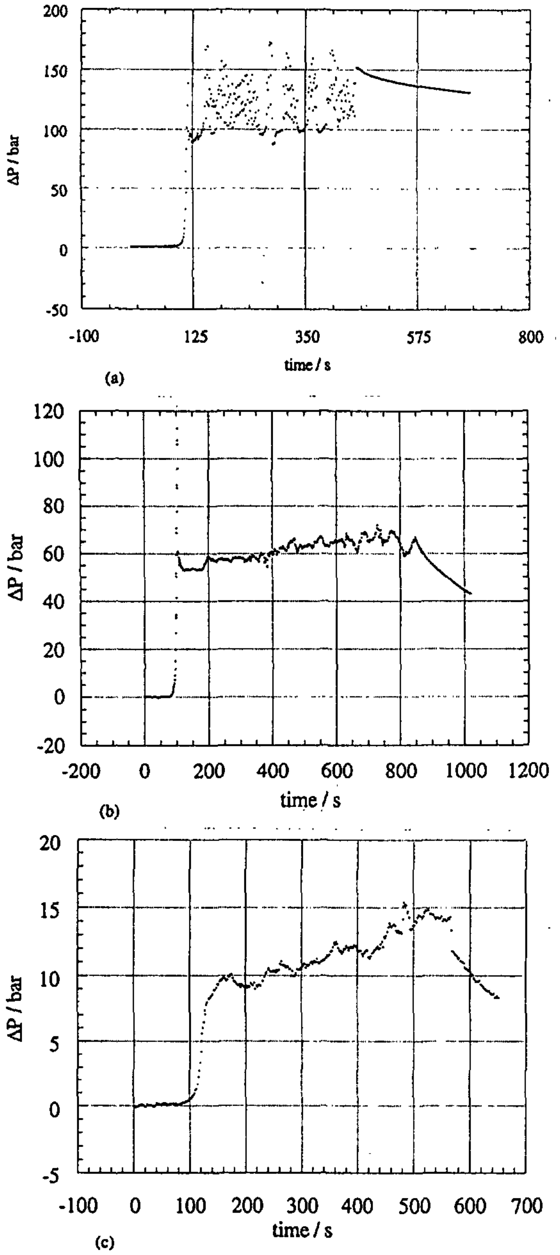

Figure 4. (a) Pressure profile at $T=18°C$, $Q=0.47 \times 10^{-7}$ m^3/s (ram speed = 1 cm/min), $L=31$ mm, $D=4$ mm, $\phi=45°C$. (b) As for (a) but $T=22°C$. (c) As for (a) but $T=26°C$.

shown. If the temperature is low (i.e. 18°C for the particular chocolate described here), extrusion occurs at high pressures and in an erratic manner as indicated in Figure 4a. Under these conditions it is not possible to produce a continuous uniform product and the material tends to emerge from the die in small pieces. A further reduction in temperature for this particular die will result in the 500 bar upper pressure limit of the rheometer being reached without any extrusion occurring.

If extrusion was carried out above 18°C, but below the melting range of the material which is of the order of 30°C, continuous uniform extrudates could be obtained. Figure 4b shows the extrusion profile obtained at a temperature of 22°C. Under these conditions a sharp yield pressure is reached followed by a region where smooth continuous extrusion is observed to occur. We are able to control the barrel temperature of the Davenport rheometer to within ±0.5°C and under the extrusion conditions described by Figure 4b we were unable to detect any change in the temperature of the chocolate extrudate. This was carried out by placing the tip of a thermocouple into the extrudate as it emerged from the die. It is from this observation that we have named the process, cold extrusion.

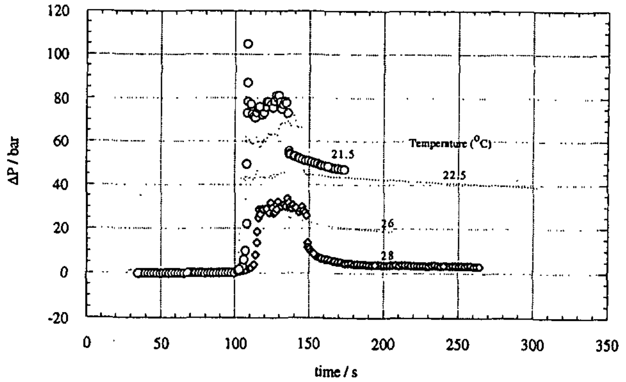

Figure 5. Pressure profiles showing dependence on T at $Q=7.1 \times 10^{-7}$ m^3/s (ram speed = 15 cm/min), $L=31$ mm, $D=4$ mm, $\phi=45°C$.

On the cessation of the extrusion process at a temperature of 22°C two components of pressure relaxation occur. Figure 4b shows a time dependent viscoelastic relaxation and a residue plastic pressure. The instantaneous viscous relaxation is barely detectable.

If the temperature is increased to close to the melting range of chocolate, as shown in Figure 4c, a more viscous like response is approached. At the temperature of 26°C the initial yield pressure is lost. The magnitude of the steady state extrusion pressure is reduced from the lower temperature value and viscous contribution to the pressure relaxation becomes apparent. Between temperatures of 20 and 28°C smooth continuous extrusions were obtained and the extrudate maintained its general shape after emerging from the die.

At above 30°C, the melting point of the chocolate, the viscosity of the material was low and it was not possible to produce a controlled extrusion with the die used for the above experiments. In addition, the extrudate emerged as a molten stream which could not be manipulated in the same way as the cold extruded samples.

Figure 5 shows the systematic variation of extrusion pressure with temperature and in this figure the loss of the yield stress together with a reduction in ΔP_f is apparent as the temperature increases. Table 1 gives the values of the flow pressure ΔP_f at each temperature, together with the viscous fraction VF association with

Table 1. Variation of Δp_f and VF with T. ($Q=7.1 \times 10^{-7}$ m^3/s; $L=31$ mm; $D=4$ mm; $\phi=45°$).

T (°C)	ΔP_f (bar)	VF
28	29	0.60
26	44	0.41
22.5	67	0.33
21.5	78	0.29

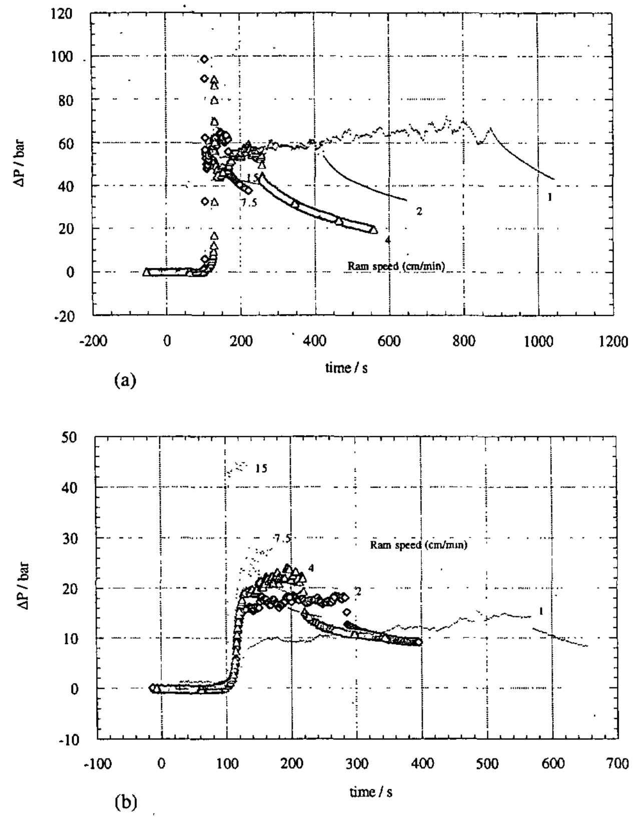

Figure 6. Pressure profiles showing dependence on Q at, (a) = 22°C and (b) = 26°C, $L = 31$ mm, $D = 4$ mm, $\phi = 45$°C.

pressure relaxation. As the temperature increases ΔP_f decreases and VF increases. Both factors indicate a more viscous response of the material at elevated temperatures.

The Effect of Extrusion Rate

Variation of ram speed had different effects at different temperatures. Figure 6a shows the effect of ram speed at the lower temperature of 22°C. Here it can be seen that the steady extrusion pressure ΔP_f is little influenced by the ram speed and in this respect the chocolate is behaving as a perfect plastic material. If, however, the temperature is increased the flow pressure does become dependent on extrusion rate. Figure 6b shows pressure profiles at $T = 26$°C and Figure 7 plots the value of ΔP_f as a function of flowrate. From the data a best fit power law fit can be obtained where

$$\Delta P_f = (K)\,Q^n \qquad (2)$$

where $K = 1.7 \times 10^9$ Pa $[\mathrm{m^3/s}]^{-0.4}$ and $n = 0.4$.

The Effect of Entry Conditions and L/D Ratio

A limited range of experiments were carried out with different entry geometries and L/D ratios and these results are tabulated in Tables 2 and 3. Table 2 shows that the variation of the entry cone angle ϕ had relatively little effect on the extrusion process and Table 3 shows that a significant amount of pressure drop occurs at the entry of the die in the region of the flow constriction. Increasing the length of the die does increase the

Table 2. Variation of ΔP_f and VF with ϕ. ($T = 24$°C; $Q = 3.5 \times 10^{-7}$ m³/s; $L = 10$ mm; $D = 4$ mm).

Entry angle, ϕ	ΔP_f (bar)	VF
15°	51	0.18
45°	55	0.30
90°	51	0.25

Table 3. Variation of ΔP_f of VF with L. ($T = 23$°C; $Q = 3.5 \times 10^{-7}$ m³/s; $D = 4$ mm; $\phi = 45$°).

L (mm)	ΔP_f (bar)	VF
0	53	0.17
10	62	0.23
20	88	0.23

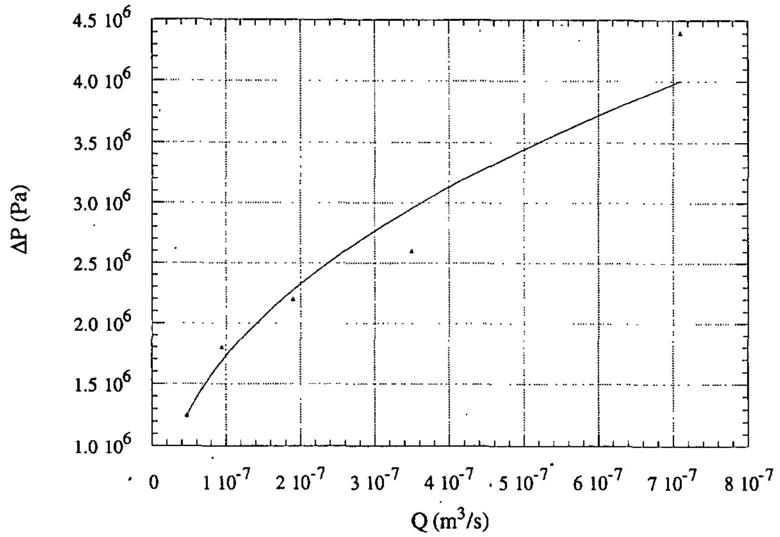

Figure 7. Power Law curve fitted to ΔP versus Q, at 26°C, $L = 31$ mm, $D = 4$ mm, $\phi = 45$°C.

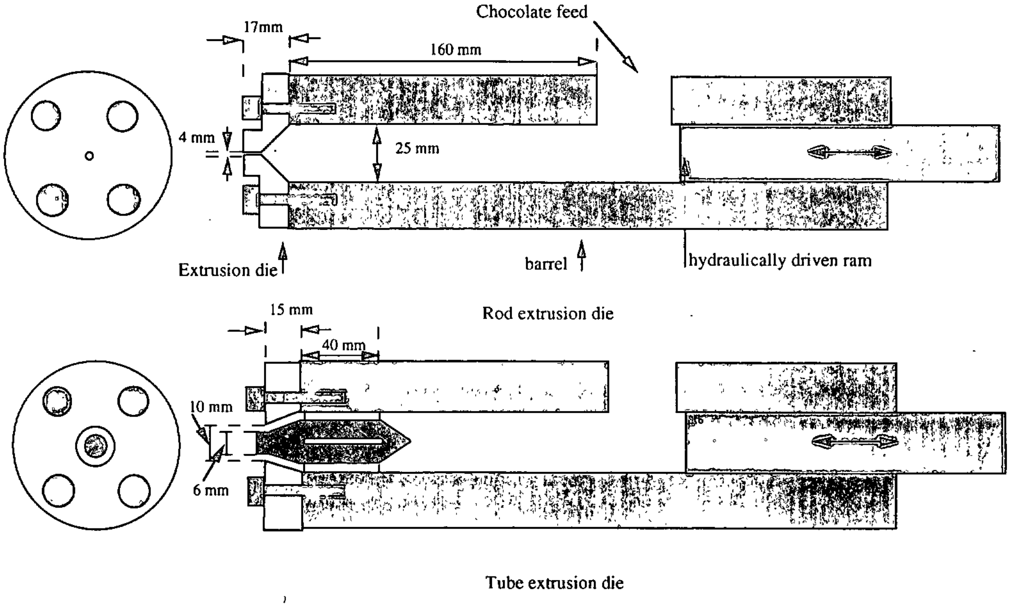

Figure 8. Schematic diagram of the 'Florin' ram extruder.

extrusion pressure but with, for example, a die of L/D ratio 5, 60% of the pressure drop is due to the entry.

Hydraulic Ram Extrusion and Post Extrusion Plasticity

Further cold extrusion experiments were carried out using a Florin hydraulically driven ram extruder and the geometry of the barrel and piston is shown schematically in Figure 8. The unit was designed some forty years ago as a simple plastics injection moulding machine and a significant feature of the machine in relation to current injection moulding units is that it does not contain a melting or metering extrusion screw.

In one experiment, semi continuous rods of 4 mm diameter were produced and in the other operating mode a centred 'torpedo' was placed in the barrel, thereby enabling the manufacture of hollow tubular sections. Experiments were carried out by dropping chocolate buttons into the feed section of the extruder followed by hydraulically advancing the ram. The ram pressure was set at 80 bar and extrusion was carried out at ambient conditions (23°C).The piston of the ram is controlled by a servo hydraulic pump and valve. Activation of the pump produces hydraulic pressure that drives the piston into the barrel. The machine was operated at room temperature and in a similar way to the Davenport rheometer experiments, no temperature change was detected in the extruded product.

In this class of experiment the deformation is essentially a controlled stress experiment where the pressure is set and the flow rate of the extrusion determined by the geometry and material properties of the chocolate. We found that smooth continuous lengths of chocolate could be produced for both the cylindrical and annular die. In the case of 4 mm rods, extrusion rates were of the order 0.3 m/s and again no detectable temperature rise in the extrudate could be measured. Figure 9 shows photographs of both the extruded rods and tube. The cross section of the extrudate is the same as the section of the extrusion die at its exit and consequently no die swell or contraction occurs.

A striking feature of chocolate cold extrusion is that immediately after extrusion and for some further delay time the chocolate can sustain significant plastic deformation. This is illustrated in Figure 9 where immediately after extrusion the 4 mm diameter extrusion had been twisted in the form of a wire. Depending on the nature of the chocolate, extrusion conditions and post extrusion storage temperature, the degree of plasticity of the chocolate will reduce and after a period of between 30 mins to several hours the chocolate will return to its more usual brittle form.

In an attempt to follow the kinetics of this property change, we measured the time evolution of the viscoelastic storage modulus of the material as a function of time. Immediately after cold extruding a 4 mm diameter rod of chocolate, a 50 mm length of the chocolate was fixed using torsion clamps to an RDS II

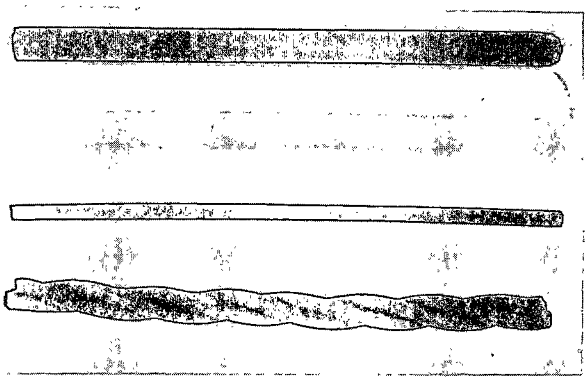

Figure 9. Photographs showing the cold extruded products. Milk and white chocolate tubes, 10 mm external diameter. A milk chocolate rod, 4 mm diameter. A two stranded twisted milk chocolate 'wire'.

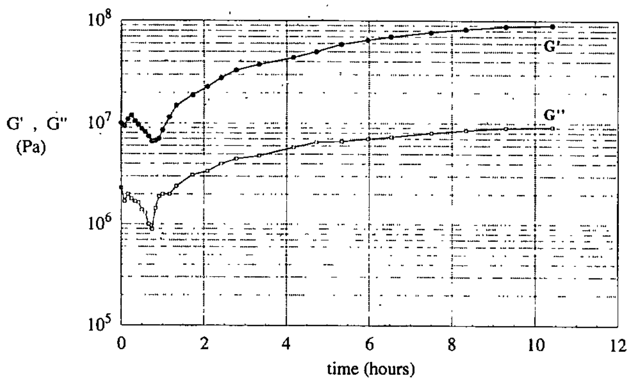

Figure 10. Storage G' and loss G'' modulus measurements of freshly extruded chocolate rod plotted as a function of time after extrusion (angular frequency $\omega = 10$ rad/s at 0.01% strain).

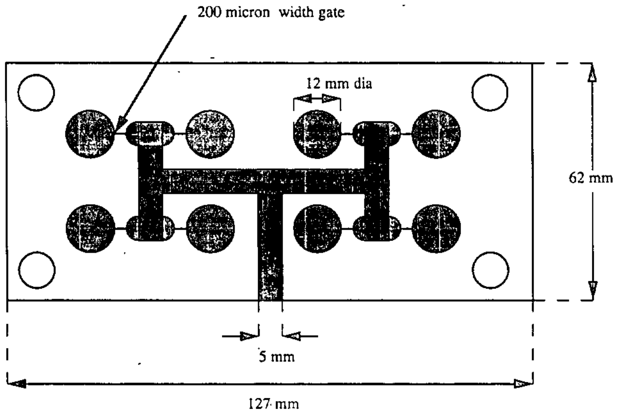

Figure 12. Schematic diagram of ball injection mould.

Rheometrics mechanical spectrometer. In Figure 10 we plot the change in storage modulus G' as a function of time. The oscillatory angular frequency was fixed at 10 radians/s and the maximum applied shear strain was 0.01%. From the data presented it can be seen that after an initial drop in modulus there is a subsequent progressive hardening of the material. In Figure 11 we show the full frequency response for a cold extruded rod. The figure shows that at small strain deformation the material is dominantly elastic at all the tested frequencies.

Finally, using the ram extrusion we have observed that in switching from one colour of chocolate to another, (i.e. white to milk) the colour transition in the extruded product occurs over a narrow length of material which might typically extend along a 15 cm length of the extrudate. This strongly suggests to us that within the extrusion process the material essentially deforms in plug flow, which in turn strongly suggests significant slip at the wall.

INJECTION MOULDING

The Florin ram extruder was originally designed as a small polymer injection moulding machine and consequently, it was possible to explore the potential of cold extrusion for the manufacture of chocolate moulded articles. The system was tested using a mould containing 12 mm diameter balls as shown schematically in Figure 12. Injection moulding of the chocolate was achieved with the simultaneous pressurisation of the injection ram and the clamping of the split mould using hydraulic pressure. Experiments were carried out with both the extruder and mould at room temperature.

We found the mould filling could occur within about 4 seconds and Figure 13 shows photographs of the partially filled and completely filled mould. An initially surprising aspect of these results is that the chocolate could be extruded through gates into the mould of 200 μm in diameter.

On the completion of the moulding operation, the moulds could be split and the injection moulded balls removed from the mould using either air suction or a knife. The surface finish of the balls was variable ranging from a matt to a glossy appearance. Little or no moulding flash was present and from the experiments carried out there is full potential for using isothermal injection moulding techniques to form chocolate objects of various shapes.

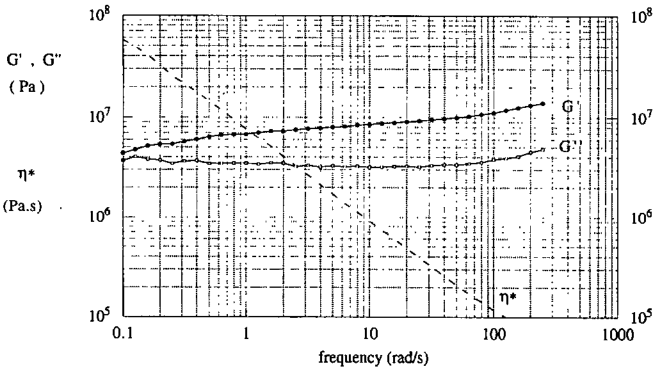

Figure 11. Frequency sweep of freshly extruded chocolate. $T = 26°C$ at 0.1% strain.

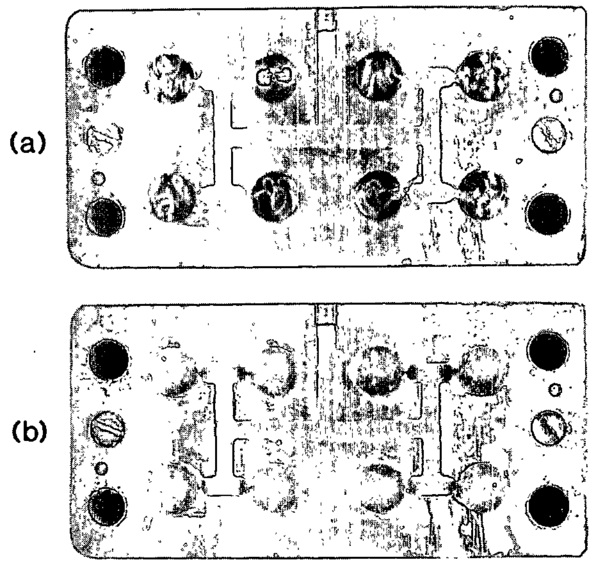

Figure 13. Photographs of separated injection mould. (a) Partial filled mould; (b) Completely filled mould.

The Mechanism of Cold Extrusion

In the temperature range of the order of 20–24°C, the extrusion of chocolate has many features associated with plastic flow. The extrusion pressure is essentially independent of extrusion rate and most of the extrusion pressure drop occurs in the entry region of the die. It is our belief that once the yield pressure within the material has been reached, the deformation is capable of partially melting some of the fat phase that surrounds the sugar crystals. This partial melting then causes the matrix to act as a viscous lubricating agent thereby allowing the plastic deformation of the material to occur.

We were initially surprised to observe that the extrusion process was isothermal, however, a simple energy balance for the process shows that this is plausible.

If we consider a zero L/D die, the work done W/kg of chocolate extruded during the extrusion is given by

$$W = \frac{F\,d}{m} \tag{3}$$

where F is the applied force, d is the distance travelled and m the mass of chocolate extruded. If it is assumed that F is constant during extrusion, then

$$F = \Delta P_f A$$

$$W = \Delta P_f \frac{A\,d}{m}, \tag{4}$$

where ΔP_f is the extrusion pressure drop and A the cross section of barrel. However $(A\,d)$ is the volume of chocolate displaced hence

$$\frac{A\,d}{m} = \frac{1}{\rho} \tag{5}$$

where ρ is the density of the chocolate. The density of chocolate is approximately $\rho = 1200$ kg/m^3 and for a typical extrusion $\Delta P = 55$ bar, hence, W is of order 5000 J/kg.

The specific heat capacity of chocolate is approximately $Cp = 1200$ J/Kg°C. Hence, if the work done by the ram were used to heat the chocolate, a rise in temperature of the order of 4.2°C would be seen. An increase of this magnitude is not observed. We believe it more likely that the work goes into partial melting of the milk and cocoa fats. The latent heat λ of melting for triglyceride fats is of the order of 150×10^3 J/Kg and these fats contain about 30% by weight of the chocolate mass. If we assume a volume fraction ϕ of the fats are melted, then a thermal balance yields

$$\phi = \frac{\Delta P_f}{0.3\lambda\rho} \tag{6}$$

For our conditions of operation we estimate ϕ to be of order 10% which appears to be an acceptable number in relation to the potential rheological change required. In addition, the calculation shows that isothermal melting can easily account for the energy dissipation. The above calculation assumes that no heat is lost to the body of the extruder. It is our belief that because of the low thermal conductivity of chocolate this term will be low and we hope to establish this point in a later publication.

The experimentally observed post extrusion plasticity of the chocolate also supports the idea of local melting. Presumably, the time dependent hardening of the extruded chocolate is a consequence of the material reverting to its original or other crystalline form. Chocolate has a number of crystallographic forms (Beckett, 1988) and at this stage without X-ray information we are unable to establish the details of this transition. We conclude that the recovery of the material to its original stiffness is in some way related to the recovery of the structure in the chocolate which was disrupted by the cold extrusion process.

As the temperature increases towards the melting point of chocolate, the extrusion process takes on a more viscous nature, the clear yield behaviour is lost and extrusion pressures become dependent on extrusion rate. The contributions of plastic and viscous flow to the deformation can be very clearly seen from pressure relaxation profiles. At low temperatures, the plastic pressure contribution dominates and at high temperature the viscous contribution is greatest. Clearly, at any given temperature the constitutive response of the material will range from plastic extrusion, similar to ceramic pastes, to the full melt behaviour that has been characterised in steady simple shear as following a Casson response.

The boundary conditions for the extrusion appear to be an important part of the process. Slip conditions at the wall can greatly modify the extrusion behaviour of materials and our observations strongly suggest partial or total slip at the wall.

DISCUSSION AND CONCLUSIONS

Cold extrusion of chocolate appears to offer a viable alternative processing route to existing methods (Mackley, 1992). Both the final texture and taste of the extruded products appear similar to conventional products made by melt casting. It is possible to extrude with very high precision and at high rates both rods and tubes. In addition, precision injection moulding is possible. The extrusion process occurs without any detectable rise in the temperature of the extrudate and we conclude that the work being put into the material is dissipated as partial isothermal melting of the fat component rather than any adiabatic heating.

The strategic advantage of the cold extrusion is shape control after the forming process. If processing is carried out in the melt as is usual at present, the molten liquid chocolate stream cannot be manipulated as has been described in this paper with cold extrusion. The rheology and boundary conditions for the cold extruded chocolate are, of course, crucial for successful operation and we have established that within a temperature window of about 10°C continuous extrusion is possible. Different chocolate compositions will have different rheological responses and clearly the detailed dependence of composition variation will need to be explored.

In terms of modelling the process, a generalised constitutive equation will need to be developed that can represent visco-plastic fluid deformation. Analytic expressions exist for purely plastic deformation in entry flow (Chakrabarty, 1987) and numerical solvers now exist that could in principle cope with constitutive equations and boundary conditions appropriate for this problem (Bobart and Crochet, 1992).

The post extrusion plasticity of the material offers a number of interesting possibilities that are not available in conventional chocolate processing. For example, multi-filaments of chocolate can be cold extruded and then subsequently plaited before complete hardening has occurred. The ability to extrude tubes of chocolate also strongly suggests that co-extrusion of different components could be readily achieved and many process routes currently used, for example, in the polymer processing industry could now be applied to chocolate processing. With these points in mind we believe cold extrusion of chocolate offers genuine benefit in a number of areas over current melt processing technology.

NOMENCLATURE

τ	shear stress, N/m^2
τ_0	yield stress, N/m^2
k	viscosity coefficient, $N\ s^{0.5}/m^2$
$\dot{\gamma}$	shear rate, s^{-1}
$VF = \frac{\Delta P_f - \Delta P_l}{\Delta P_f}$	viscous fraction
ΔP_f	steady state pressure drop during flow, N/m^2
ΔP_l	pressure immediately after flow cessation, N/m^2
K	power law coefficient, $N\ s^{n=1}/m^2$
Q	flowrate, m^3/s
n	power law index
W	work done/kg, J/kg
F	force, N
d	distance, m
A	cross sectional area of die, m^2
m	mass of chocolate extruded, Kg
ρ	density, kg/m^3
λ	latent heat, J/kg
ϕ	volume fraction of fat

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ADDRESS

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