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WORKING PARTY ON STRUCTURE AND PROPERTIES OF
COMMERCIAL POLYMERS*

**INFLUENCE OF ACRYLIC
PROCESSING AIDS ON THE
RHEOLOGY AND STRUCTURE OF
POLYVINYL CHLORIDE**

Prepared for publication by

F. N. COGSWELL

Petrochemicals and Plastics Division,
Imperial Chemical Industries, Welwyn Garden City, UK

*Membership of the Working Party engaged on this programme during 1979-81 was principally as follows:

Chairman: P. L. CLEGG (UK); *Secretary:* M. E. CARREGA (France); *Members:* G. AJROLDI (Italy); J. M. CANN (UK); J. C. CHAUFFOUREAUX (Belgium); F. N. COGSWELL (UK); D. CONSTANTIN (France); H. COSTER (Netherlands); M. FLEISSNER (FRG); A. GHIJSELS (Netherlands); G. GOLDBACH (FRG); P. B. KEATING (Belgium); A. S. LODGE (USA); J. MEISSNER (Switzerland); H. MÜNSTEDT (FRG); G. SCHOUKENS (Belgium); J. SEFERIS (USA); A. K. van der VEGT (Netherlands); A. J. de VRIES (France); J. L. S. WALES (Netherlands); H. H. WINTER (USA); J. YOUNG (Netherlands).

THE INFLUENCE OF ACRYLIC PROCESSING AIDS ON THE RHEOLOGY AND STRUCTURE OF PVC

Prepared for publication by F. N. Cogswell

Imperial Chemical Industries PLC, Petrochemicals & Plastics Division, P O Box No 6,
Bessemer Road, Welwyn Garden City, Hertfordshire AL7 1HD

Abstract - High molecular weight acrylic polymers enhance the processability of PVC especially by increasing their extensibility and suppressing various kinds of defect. Eight industrial laboratories have collaborated in this joint investigation of the mechanism by which acrylic processing aids have their effect. Amongst the variables investigated are:

level of processing aid
molecular weight of processing aid
molecular weight of polymer
compounding history.

The investigation included: studies of the gelation process; observations of constrained flow into, through, and out of, channels; and experiments with free surface deformations. Acrylic processing aids: increase the work done within the polymer during compounding; have little effect on "viscosity" in capillary flow; and greatly enhances extensibility without significantly altering the stress/strain response up to failure. The evidence available to the working party is consistent with the flow mechanism of PVC being a continual rupture and reformation process in the connective tissue between flow units which are approximately 100nm in size. It is concluded that the acrylic processing aid is dispersed onto the boundaries of these particles and that it acts there as a stress transfer agent distributing intense local stresses through larger volume of the material.

Incidental to this investigation are comparisons of several "non-standard" experimental techniques. Friction studies using an annular shear cell and a sliding plug gave qualitatively consistent results with respect to the major transitions in friction response as a function of temperature and pressure. Extensibility studies using direct rheological measurement, an instrumented haul off and constant force extension give quantitatively agreement for both deformation and rupture behaviour.

1 INTRODUCTION

This programme has its origins in two earlier investigations by this Working Party. The programme on "Sub-primary particles in PVC: identification and elucidation of their role during flow" (Pure and Applied Chemistry 52, 2031, 1980) concluded that the flow unit in PVC is an approximately spherical domain of about 100nm diameter, and that these domains are essentially undeformed during flow but that the connective tissue between them is highly deformable. The second parent was an exchange of information between members of the Working Party on the phenomenon of rupture in polymer melts in which it was noted that acrylic processing aids altered the rupture characteristics of PVC (unpublished papers of the Working Party). This research was undertaken in order to:

- i) further elucidate the flow mechanism of PVC by studying the changes which occur in its rheology and structure when that polymer is compounded with acrylic processing aids;
- ii) seek an understanding of the connective tissue between flow units;
- iii) understand the mechanism by which acrylic processing aids have their effect;
- iv) characterise the rupture properties of PVC at its processing temperature.

The active participants in this collaboration are identified in the text as follows:

BASF	Badische Aniline und Soda Fabrik AG, Ludwigshafen.
BP	BP Chemicals PLC, Penarth.
BW	Borg-Warner Chemicals, Amsterdam.
Huls	Chemische Werke Huls AG, Marl.
ICI	ICI PLC, Welwyn Garden City.
Shell	Koninklijke/Shell Laboratorium, Amsterdam.
Solvay	Solvay et Cie, Bruxelles.
TNO	Organisation for Industrial Research, Delft.

2 MATERIALS USED IN THIS STUDY

Most of the work reported here was carried out on powder samples prepared by Shell to the following recipe:

"Carina" 67-01 (unplasticised PVC having K value 67)	100 parts
L3651 (acoprecipitate of stabiliser and lubricant from AKZO)	2 parts
Omya 95T (calcium carbonate from Plusstauffer)	2 parts

containing 0, 1 and 3 parts of 'Diakon' APA-1, an acrylic processing aid having a molecular weight of 1,000,000. The powder samples were prepared by blending in a Papenmeier mixer.

Additional studies were made on a variety of other commercially available unplasticised, suspension polymerised PVCs and one sample of emulsion polymerised PVC including:

"Solvic" 261RA (K value 61)
"Breon" S110/11 (K value 66)
"Corvic" S57/116 (K value 57)
"Corvic" S62/109 (K value 62)
"Carina" 67-01 (K value 67)
"Corvic" E72/660 (K value 69, emulsion polymerised)

Full details of the formulations used are included in Appendix 1.

As well as 'Diakon' APA-1 ($\bar{M}_w = 1,000,000$) a second grade of commercially available processing aid was used namely 'Diakon' APA-3 ($\bar{M}_w = 3,000,000$) and an experimental sample was polymerised to give a molecular weight of 48,000,000.

3 RESULTS

For the purposes of this study we consider a processing operation to comprise three distinctive stages:

- i) gelation or fusion,
 - ii) constrained flow into, through, and out of, channels,
- and
- iii) free surface deformations.

In addition to studying the rheology relevant to these stages participants have also made some study of the morphology and mechanical properties of samples prepared in this programme.

3.1 Studies of the Gelation Process

3.1.1. Gelation in a Brabender Plasticorder

Three participants (BP, Solvay and ICI) used torque rheometry to study fusion processes. Solvay used the Brabender with a programmed heating rate of 4°C/min observing a peak torque in the range 180-185°C (Fig. 1).

BP also used a programmed heating rate of 40°C/min (Fig. 2) and also observed torque as a function of time when the sample was charged to the Brabender at 140°C. ICI charged the sample at 180°C and observed torque as a function of time (Fig. 3).

All participants observed a higher value of peak torque in the case of samples containing acrylic processing aid.

Parts of APA-1	Peak torque			
	Peak torque for control sample (0 APA-1)			
	Solvay at 4°C/min	BP at 4°C/min	BP at 140°C	ICI at 180°C
1	1.03		1.16	1.10
3	1.28	1.65	1.30	1.19

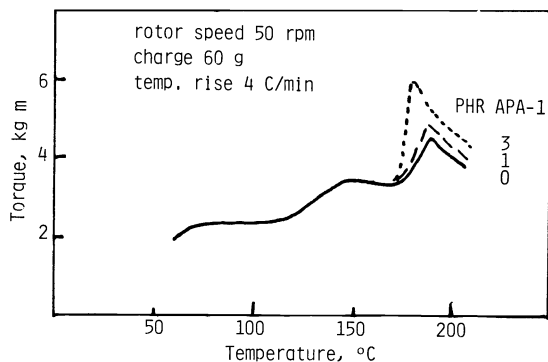


Fig. 1 (Results of Solvay)

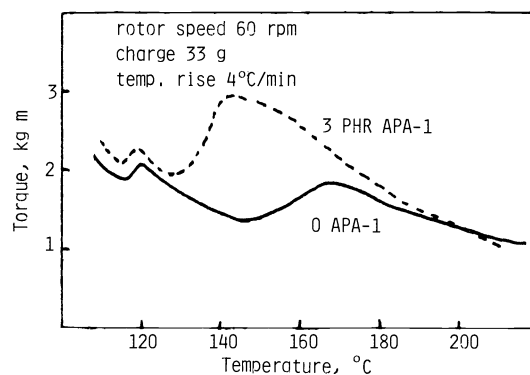


Fig. 2 (Results of BP)

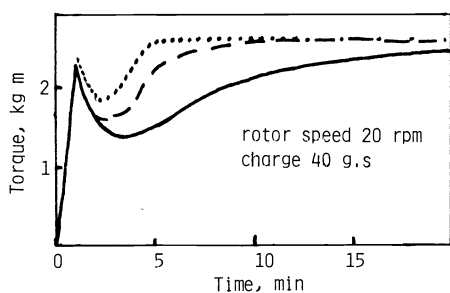


Fig. 3 (Results of ICI)

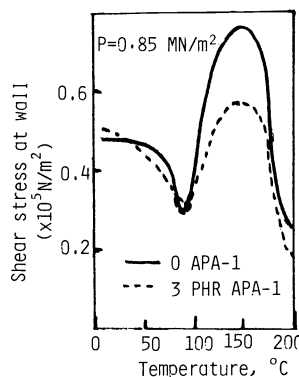


Fig. 4 (Results of TNO)

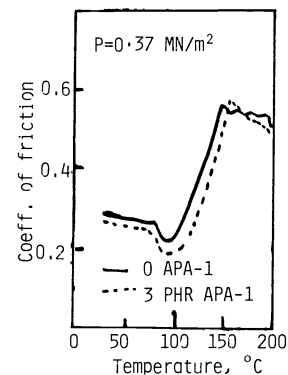


Fig. 5 (Results of ICI)

In passing we must note that despite superficially similar conditions being used (when the sample is charged to a hot vessel its temperature will rise from ambient to, or above, the set temperature) the shapes of the Brabender traces is dramatically different between different laboratories through, within the practice of each laboratory, very similar. These differences are, at least in part, associated with the use of different charges and rotation rates and emphasise that it is always preferred to carry out control experiments with this type of measurement.

The Brabender test involves work between the polymer and a metal surface and within the polymer itself. To clarify how much of the response is internal to the polymer three participants made studies of friction against a metal surface.

TNO used a sliding cylinder technique while ICI used a rotating annular shear cell (Plastics and Rubber Processing and Applications 1, 87-93, (1981)) to investigate friction behaviour up to fusion. Considering the differences in technique the results are encouragingly similar (Figs. 4 and 5).

Both participants find the same qualitative shape in the response of friction against temperature with a major transition in response at about 100°C (Fig. 6).

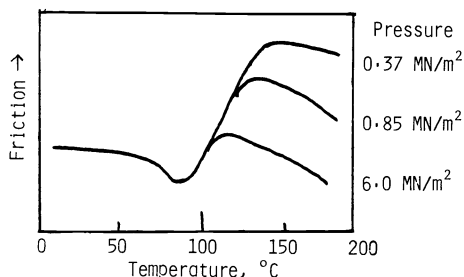


Fig. 6

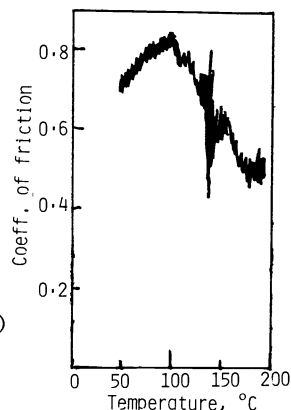


Fig. 7 (Results of ICI)

Both participants also observe that, if anything, the sample containing acrylic processing aid is of lower friction up to 150°C - a result which is particularly surprising in the light of the very high coefficient of friction of the processing aid itself (Fig. 7).

To check that the friction response was not an anomaly associated with the lubricant system in this particular formulation ICI also made studies of friction with a different formulation (Appendix 1) based on a 'Corvic' polymer: the results were qualitatively similar.

Solvay used a technique based on slit die rheometry using rough and smooth dies (J Rheol 23, 1, 1-24 (1979)). To maintain a given shear stress the material must be extruded at a nominal shear rate which is much greater in the case of the smooth die than in the case of the rough die,

Nominal Shear Rates (sec⁻¹) at 195°C

Shear stress	0 APA-1		1phr APA-1		3phr APA-1	
	Smooth	Rough	Smooth	Rough	Smooth	Rough
1 x 10 ⁵ N/m ²	40	9	33	9	29	9

but that this difference is least in the sample with the greatest amount of processing aid. These results may be interpreted to give a slip velocity as follows

Slip velocity for shear stress 1x10⁵N/m² at 195°C
(mm/s)

0phr APA-1	14
1phr APA-1	11
3phr APA-1	10

In respect of friction properties we thus see two regions: below 150°C where the processing aid slightly reduces friction; and above 190°C where it appears to slightly increase friction. The consensus view of the Working Party was that friction at the polymer/metal interface was not primarily responsible for major peaks observed in the Brabender tests, but that those features were associated with internal work in the sample.

3.1.3. Instrumented Extrusion

BP studied the flow behaviour in an instrumented 34mm counter rotating twin screw extruder: details of the conditions used are given in Appendix 2.

While all the powders have satisfactory flow properties BP noted an increase in powder bulk density for samples containing acrylic processing aid.

Parts APA-1	Powder Bulk Density (kg/m ³)
0	584
1	597
2.5	610
(SD)	(2)

suggesting that during the powder mixing process the hard particles of processing aid break up some of the more loose particles of PVC. This increase in bulk density gives a corresponding increase in output rate at a set screw speed. The addition of processing aid causes an increase in energy consumption and extrusion pressure consistent with the results of the Brabender study but in particular BP note very large increases in pressure in the feed zone of the extruder indicative of earlier, or more complete, gelation.

Melt Pressure (MN/m^2)

Parts APA-1	Feed Zone	Compression Zone	Die
0	0.65	3.8	10.1
1	0.85	4.3	10.4
2.5	1.0	5.0	11.3
% increase for 2.5 parts	54%	32%	12%

3.2 Constrained Flow

3.2.1. Small Amplitude Rotational Rheometry

Two participants (BW, Huls) made studies of oscillatory flow and of stress relaxation. The dynamic response of all samples was identical in the angular frequency range 10^{-1} to 3×10^2 radians/sec: typical data are shown in Fig. 8.

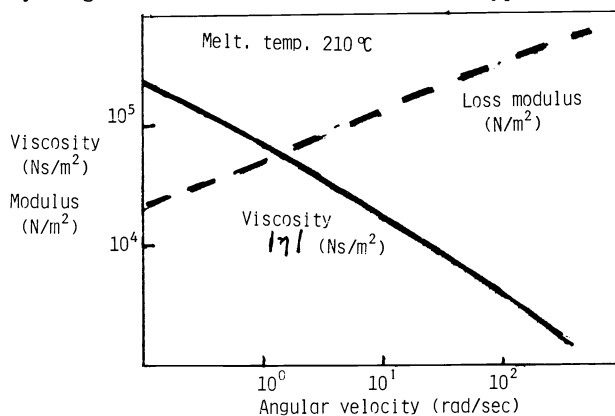


Fig. 8 (Results of Huls)

stress relaxation experiments (BW) also suggested similar response for all the samples.

3.2.2. Capillary Rheometry

Several participants (BASF, BW, Huls, ICI and Solvay) studied capillary flow in the temperature range 170°C - 210°C using both powder and differently compounded materials in both ram and screw driven extrusion rheometers. A typical comparison between the different laboratories is shown below:

$\frac{\text{Viscosity}}{\text{Viscosity of control sample}} \text{ at } 100 \text{ sec}^{-1}$

Participant	Temp °C	1phr APA-1	3phr APA-1
BASF	190	1.05	1.12
BW	290	0.99	1.05
Huls	180	1.09	1.18
Huls	210	0.98	1.11
ICI	190	1.02	1.14
Solvay	180	1.00	1.09
Mean (SD)		1.02 (0.04)	1.12 (0.04)

indicating a small increase in capillary viscosity with the addition of processing aid: typical data are shown in Fig. 9.

Solvay also studied the effects of die L/R ratio and entrance angle. They observed that at low L/R ratio and wide entrance angle and high flow rate the pressure drop for samples containing acrylic processing aid was very much higher than the control value.

Pressure Drop/Pressure for control sample

Die length/radius	1		15	
	1 phr APA-1	3 phr APA-1	1 phr APA-1	3 phr APA-1
Shear rate(Sec-1)				
48	0.92	1.00	0.96	1.07
480	0.96	1.18	0.95	0.98
4800	1.13	1.52	1.00	1.05

(die diameter 1mm entrance angle 180°C, Temp 180°C).

This result was supported by BP who carried out extrusion rheometry, with an orifice die at 130°C using a Sieglaff- McKelvey rheometer, on a series of materials removed from a Brabender fusion cycle at different temperatures - Fig. 10.

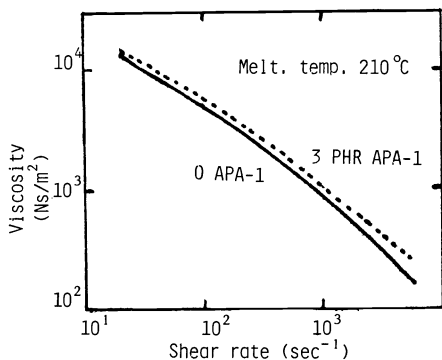


Fig. 9 (Results of Huls)

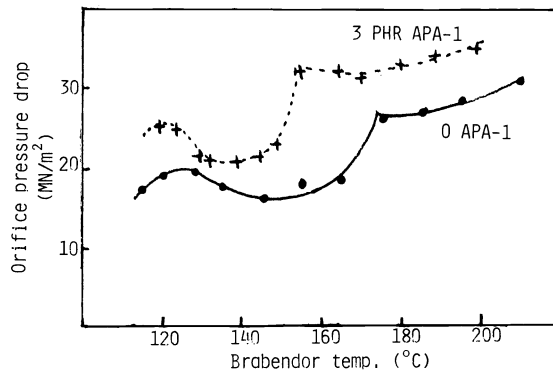
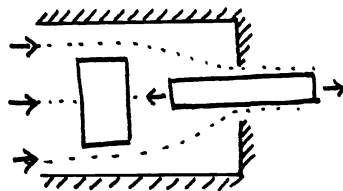


Fig. 10 (Results of BP)

The samples containing processing aid have consistently higher extrusion pressure. It is probable that the small effect on capillary flow, usually regarded as a simple shear flow, is associated with the observation of Solvay (3.1.2 above) that the processing aid reduces slip at high temperatures. Converging flow through short, or orifice dies, is usually regarded as containing a stretching flow component,



and the significant increase in resistance to flow in such geometries suggests a modification of the elongational flow behaviour of the material.

Three laboratories, (BW, ICI and Shell) commented on a significant reduction in the flow defect commonly known as "sharkskin" as a result of the addition of acrylic processing aid - Fig. 11,

Extruded at 190°C shear rate 36 sec⁻¹

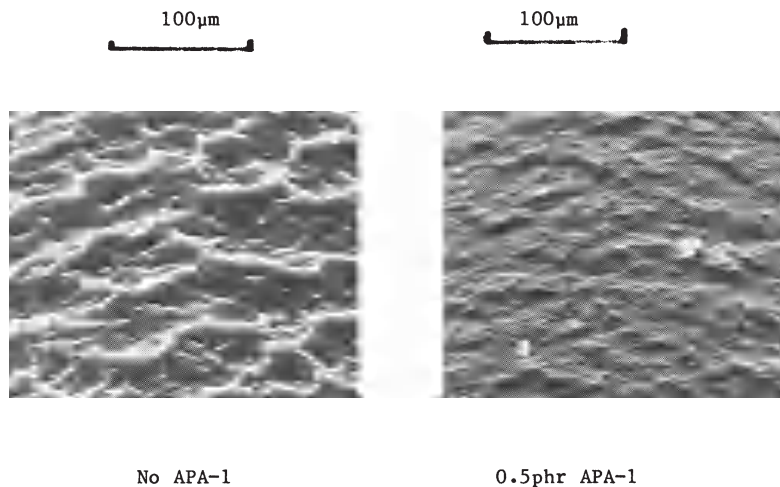


Fig. 11 (Results of Borg Warner)

"Sharkskin", has been associated with the extensibility of the melt at the die exit (J. Non-Newtonian Fluid Mechanics 2, 1, 37 (1977)).

3.3 Extensibility

Four participants carried out extensibility studies. Shell used a "Rheotens" device in combination with a screw extruder, BASF and BW used a "Rheotens" melt tensile tester in conjunction with a ram extruder, BASF also used constant strain rate extension using the "Munstedt Universal Extensional Rheometer" at 0.01sec⁻¹, and ICI used constant force extension on the extrudate from a ram extruder. Shell and ICI used the standard compound, BW and BASF used modified compounds to give enhanced thermal stability.

Up to 195°C all participants found qualitatively similar force/deformation response up to failure - Figs. 12, 13, 14.

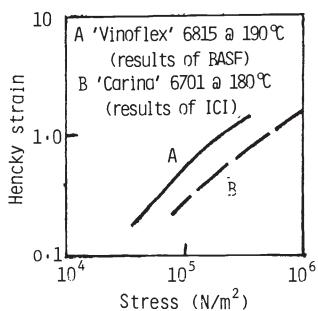


Fig. 12 Direct Measurement and Constant Force Extension

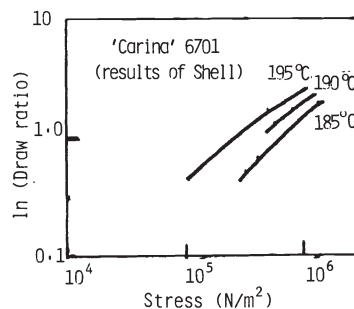


Fig. 13 "Rheotens" Experiment

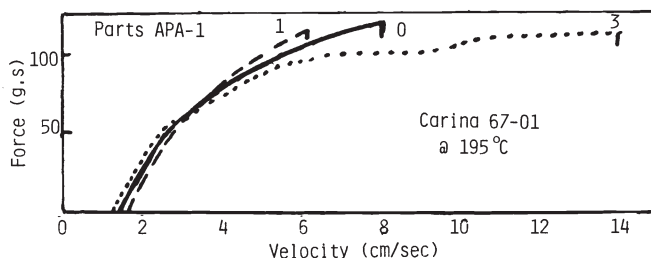


Fig. 14 (Results of Shell)

with acrylic processing aid having little effect on the deformation response but a large effect on final rupture. Quantitatively consistent results were obtained by Shell and ICI on the same polymer despite the differences in test technique and material prehistory.

Rupture of "Carina" 67-01

Parts APA-1	Tested by	Temp °C	at Break	
			Draw Ratio	Stress (N/m ²)
0	ICI	180	1.4	1.1 x 10 ⁵
	Shell	185	1.7	3.4
	Shell	190	4.9	8.1
	Shell	195	6.0	7.4
1	ICI	180	2.4	4.3
	Shell	185	2.0	4.1
	Shell	190	3.7	5.9
	Shell	195	6.0	6.1
3	ICI	180	6.4	12.1
	Shell	185	6.8	13.2
	Shell	190	7.9	12.0
	Shell	195	12.3	11.8

The results of BASF and BW were consistent with this pattern and are discussed in more detail later (3.3.4 below).

3.3.1 Molecular Weight of Processing Aid

ICI varied the molecular weight of the processing aid including an experimental sample of very high molecular weight: typical results are tabulated below.

Extensibility of 'Corvic' S61/109 at 170°C

Parts of Processing Aid	APA-1	APA-3	Expt Sample
	1,000,000	3,000,000	48,000,000
0	1.1	1.1	1.1
1	1.8	5.5	2.7
3	5.0	-	7.4
5	9.0	22	7.5
10	25	37	5.0

At low levels of processing aid the higher molecular weight commercial sample is clearly beneficial, however, that advantage is less marked at high loadings. The very high molecular weight sample shows reduced effectiveness and even a deterioration in extensibility at high loadings. The very high molecular weight sample was also observed to have increased opacity suggesting poor dispersion (see 3.5.2. below).

3.3.2 Particle Size of Processing Aid

ICI used sieved fractions of the processing aid, <53µm and >63µm, and compared the extensibility of such fractions of APA-1 at 1phr loading in 'Corvic' D62/109 at 170°C.

	Draw Ratio at Break	
Fines	2.2)
Whole sample	1.8)
Coarse Particles	2.5)
) standard deviation 0.2

There are no significant differences associated with particle size.

3.3.3 PVC Polymerisation Route

All the work discussed to date has been concerned with suspension polymerised PVC. The Working Party has recognised that there may be a qualitative difference in flow mechanism associated with different polymerisation routes (Pure and Applied Chemistry 52, 2031 (1980)) and in particular the different flow behaviour of suspension and emulsion polymerised samples. ICI compared the extensibility of suspension and emulsion polymerised samples and observed qualitatively similar response in both cases but quantitatively less enhancement in the emulsion grade - probably associated with the higher molecular weight of that polymer (3.3.4 below).

Comparison of Emulsion and Suspension Polymers at 170°C

phr APA-1	'Corvic' E72/660 Emulsion	'Corvic' S62/109 Suspension
0	1.1	1.1
1	1.6	1.8
3	3.0	5.0
5	5.0	9.0
10	6.0	25.0

3.3.4. Molecular Weight of PVC and the Additive Recipe

BASF, BW, ICI and Shell studied extensibility using a variety of polymers of different molecular weights and also different combinations of stabilisers and lubricants (Appendix 1). At a constant level of APA-1 extensibility increases with temperature and decreases with molecular weight - Figure 15, 16.

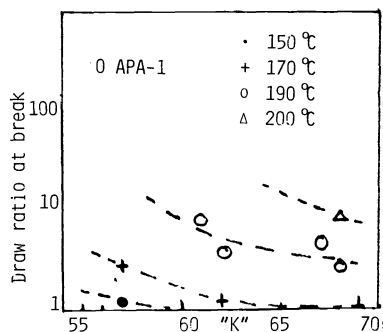


Fig. 15 (Results of Shell, BASF, BW, ICI)

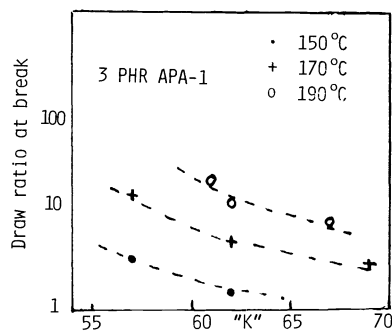


Fig. 16 (Results of Shell, BW, ICI)

More complete data is included in Appendix 3. No attempt has been made to rationalise the results in respect of the various additives and lubricants used in the compositions but it appears unlikely that these are a significant factor.

3.4 Mechanical Properties of Modified PVC

Two laboratories (BW or Huls) made dynamic measurements covering the temperature range - 200°C to + 100°C. Typical data are shown in Fig. 17.

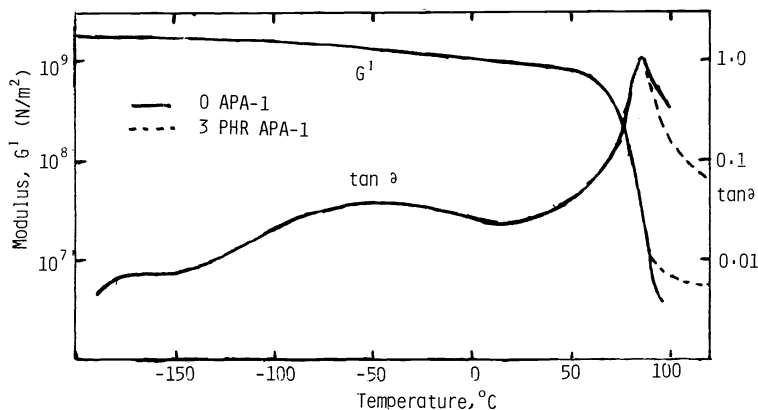


Fig. 17 (Results of Huls)

The results show little difference but suggest that above 80°C the modified sample may retain greater stiffness.

3.5 Morphology

3.5.1. X-ray Studies

On the basis of wide angle x-ray scattering Huls deduced a slight increase in crystallinity for the compound containing 3pts of APA-1 (7% compared to 5% for the control sample). In a previous report of this Working Party (Pure and Applied Chemistry 52, 2031, 1980) it was observed that high work input increased the coherence of the 100nm flow units and the increased crystallinity observed here resulting from the addition of APA-1 (giving higher work input during compounding) may be associated with that observation.

Small angle scattering (Huls) showed no difference as a the addition of processing aid.

3.5.2. Microscopy

Huls used electron microscopy to study the morphology blends observing no differences resulting from the processing aid.

ICI and BP used electron microscopy to study samples. Fracture surfaces show enhanced gelation in containing low levels of both APA-1 and APA-3 (Figure 18).

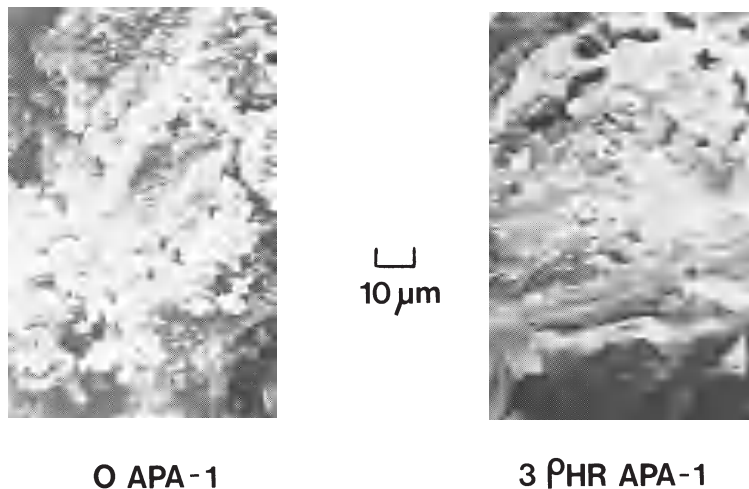


Fig. 18 (Results of BP) ex-Brabender at 155°C

It was not possible to detect the presence of the processing aid as a discrete phase in the fully compounded samples but there is some evidence of the processing aid distributed on the surface of the 100nm particles at low temperature. Sections of the gelled sample containing 10% of the experimental batch of very high molecular weight processing aid (Figure 19).

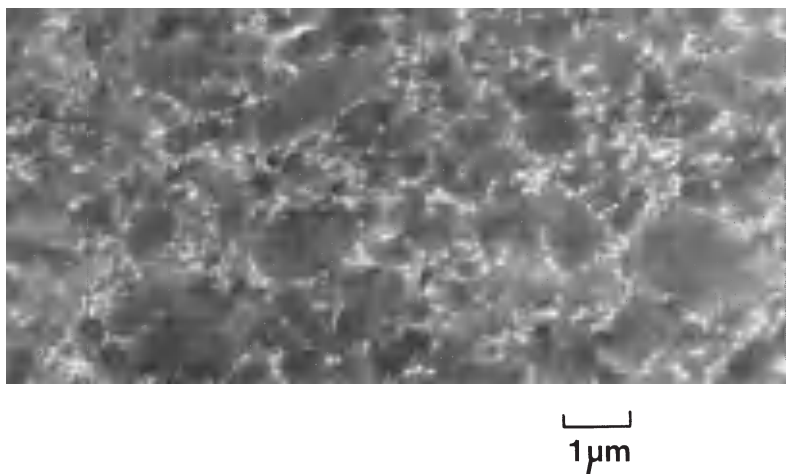


Fig. 19 (Results of ICI)

revealed a discrete phase, presumed to be of poorly dispersed processing aid, surrounding approximately spherical domains of PVC at all levels down to the 100 nm scale. In the case of the very high molecular weight sample we must recall (3.3.1. above) that this had reduced effectiveness compared to lower molecular weight materials and conclude that the processing aid is only effective when fully absorbed into the surfaces of the PVC particles.

4 DISCUSSION : THE FLOW MECHANISM IN PVC

The evidence available to this Working Party indicates that acrylic processing aids:

- i) have no significant effect on small amplitude dynamic measurements or on the stress/strain response up to rupture;
- ii) slightly increase the resistance to flow in extrusion, that difference being significantly greater during converging flows;
- iii) considerably increase the torque in mixing flows;
- iv) greatly increase the rupture stress in extension and suppress defects such as "sharkskin" which are associated with melt rupture;
- v) are dispersed in the PVC down to, or below, the 100nm scale.

These observations are consistent with the flow mechanism of PVC being a continual rupture and reformation process in the connective tissue between 100nm flow units.

For small deformations there is no effect of the processing aid. Large constrained deformations require rupture to take place: in the case of converging flow the higher rupture stress of the modified polymer leads to higher extrusion pressure; in capillary flow wall slip, or local flow in a layer near the wall, dominates the response. Mixing flows, such as occur in a Brabender, require a considerable amount of flow where the streamlines do not remain parallel, in addition they include rupture of the streamlines which requires greater energy in the case of the modified materials. Up to rupture, free surface deformations are stable and similar in modified or unmodified resins but rupture is considerably delayed in the case of samples with acrylic processing aid present.

5 DISCUSSION: POSSIBLE MECHANISMS FOR THE ACTION OF ACRYLIC PROCESSING AIDS

The Working Party considered several possible mechanisms by which the processing aid might have its effect. These divide into three groups:

- i) elimination of faults in the structure by filling holes or breaking down the particulate character;
- ii) by acting as a stress transfer medium through which a local stress between two flow units can be distributed through a larger volume (noting that a chain of molecular weight 10^6 would have a fully extended length of about $2.5\mu\text{m}$);
- iii) by synergism, such as promoting lubricant migration or by diffusing into the PVC and altering its melting characteristics.

Of these mechanisms the third argument has no direct evidence to support it. Indeed the fact that distinctly different recipes give similar results suggests that any synergism acting is not specific to particular ingredients. That molecular weight of the processing aid is an important fact suggests that the effect cannot be purely chemical in nature and the observation that high molecular weight is beneficial (providing that it is not so high that it cannot be dispersed) seems to exclude the possibility of molecular diffusion mechanisms.

Failure to detect the presence of the preferred grades of processing aid in the fully compounded products suggest that it is dispersed at a scale of less than 10nm and is not acting in a space filling role. However, the results of increased density of the powder compounds does suggest that the processing aid has a role in breaking down the granule structure leading to improved packing. That interaction is to be expected of a hard particle (T_g 100°C) with the softer PVC (T_g 80°C) but should not be affected by molecular weight and since the effect of molecular weight is clear this can only be a small part of the total picture.

The evidence available to the Working Party is consistent with the processing aid being dispersed onto the boundaries of the flow units at all scales down to 100nm, and acting there as a stress transfer medium dissipating local stresses through larger volumes of the PVC. It is in this capacity that the preferred higher molecular weight is important by increasing the volume through which a stress concentration can be dissipated.

APPENDIX 1

RESIN, STABILISER AND LUBRICANT FORMULATIONS

The basic fomulation common to all participants was

"Carina" 67/01	100
L3651	2
Omya 95T	2
Processing aid 'Diakon' APA-1	0 to 3

Materials based on "Corvic" polymers (ICI) and "Solvic" 261 RA (BW) were based on

Polymer	100 parts
"Irgastab" 17MOK	1.5
Wax PA 190	0.2
Wax E	0.8
"Loxio1" GH4	1.0
Processing aid	0 to 10

BP used

"Breon" S110/11	100 parts by weight
TBLS	1.0
DBLS	0.9
Winnofil 'S'	1.0
Stearic acid	0.3
Calcium stearage	0.4
GS 2411P wax	0.2

BASF used

"Vinoflex" S6815	100
Tin stabiliser	2
Low molecular weight additives	1.2

APPENDIX 2

CONDITIONS USED FOR INSTRUMENTED EXTRUDER STUDIES (BP)

A Leistritz LS.30.34. counter-rotating parallel twin screw extruder was used. The screw configuration consisted of the following elements:-

Feed zone
Compression zone (compression ratio 1.5)
Shear rings
Decompression and vent zone
Melt conveying (compression ratio 1.0 relative to feed zone)

Pressure transducers and thermocouples were mounted in the barrel wall near the lower cusps of the double barrel section. Pressure readings fluctuate cyclically, especially in the feed and compression zones, as the screws rotate. Mean values are quoted here.

An annular pipe die of 27mm diameter and 2mm gap, was used.

The screw speed was 30rpm throughout. The melt temperatures observed did not vary significantly and are given here in comparison with the set temperatures.

Zone	Set temperature °C	Melt temperature °C
1 : Hopper	cooled	
2	160	170
3	170	174
4 : Vent	170	
5	175	174
6	180	178
7 : Die adaptor	180	182
8 : Die	185	
9 : Die	190	

APPENDIX 3

DRAW RATIO AT BREAK

Polymer	K value	Tested by	Method*	Melt Temp °C	Draw ratio at break		
					No APA-1	1 Part APA-1	3 APA-1
"Corvic" S57/116	57	ICI	CF	150	1.1	1.1	3.3
				170	2.7	6.7	13
"Solvic" 261RA	61	BW	R	190	8.2	11	21
"Corvic" S62/109	62	ICI	CF	150	1.1	1.1	1.6
				170	1.1	1.8	5.0
				190	3.7	6.7	12
"Carina" 67/06	67	ICI Shell	CF R	180	1.4	2.4	6.4
				185	1.7	2.0	6.8
				190	4.9	3.7	7.9
				195	6.0	6.0	12.3
"Vinoflex" S6815	68	BASF	UER R	190	2.3		
				200	8.4	>10	>10
"Corvic" E72/110	69	ICI	CF	270	1.1	1.6	3.0

* Key for Method CF Constant Force
 R "Rheotens"
 UER "Universal Extensional Rheometer"

LIST OF MAIN CONTRIBUTORS

H Munstedt	BASF	J C Chauffoureaux)	
		C Dehennau)	Solvay
		G Shoukens)	
S T E Aldhouse)	BP		
S C Jones)			
H Coster	Borg Warner	J L S Wales	TNO
G Goldbach	Huls	M S Swerdlow)	
		R C Young)	ICI
J J S M Ente)			
A Ghijsels)	Shell		
J Raadsen)			