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# Investigation of Precipitated Calcium Carbonate as a Processing Aid and Impact Modifier in Poly(vinyl chloride)

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#### **Abstract**

The effect of ultra-fine precipitated calcium carbonate (PCC), with a primary particle size of 50nm, on the fusion behaviour and mechanical properties of Ca/Zn stabilized poly(vinyl chloride) PVC has been investigated. The optimum concentration of PCC was determined and, in addition, experimental design was used to examine possible synergies between PCC and two important additives in a PVC-U formulation: namely, the processing aid and impact modifier. Dry blends were extruded and tested by Differential Scanning Calorimetry (DSC), tensile testing and Charpy impact testing. The results confirm that ultra-fine PCC can give rise to large improvements in single notch impact strengths. Control values of 8 kJ/m<sup>-2</sup> were increased to 79 kJ/m<sup>-2</sup> through the addition of ultra-fine PCC. There was also found to be a significant increase in fusion levels but no effect on the tensile strength of the samples. It is concluded that PCC behaves as both a processing aid and impact modifier in PVC-U formulations, and the reasons for this are discussed.

# 1. INTRODUCTION

Synthetic calcium carbonate, known as precipitated calcium carbonate (PCC), has the advantages of a very fine particle size, narrow particle size distribution and high purity. Typical primary particle sizes of ultra-fine grades are in the range of 50 - 70 nm and these are agglomerated into larger particles of 0.7 - 2.0 µm diameter. Products are usually coated with 1 – 2% of stearic acid. The surface coating introduces hydrophobicity to the surface of the PCC particles, reduces surface energy and promotes mixing with polymers. The fine PCC particles, which are comparable in size with primary particles of PVC, have the capability to generate inter-particle friction in PVC formulations, thereby increasing shear and promoting fusion [1]. Hence, PCC is added as a functional filler to PVC formulations, where it can act as a processing aid.

Various studies have investigated the effect of ultra-fine PCC on impact properties of poly(vinyl chloride) (PVC) [1 - 9]. Substantial improvements in impact strength have been reported depending on the PVC formulation and processing method used, as well as the PCC particle size and coating applied.

Fernando and Thomas [2] have reported that when the mean particle size of calcium carbonate is reduced below 1 micron, then there is a significant increase in the notched impact strength of filled PVC-U compounds, and the smaller the particle size the greater the effect. Increasing the mean particle size above 1 micron caused a reduction in notched impact strength compared with the unfilled control. Bryant and Wiebking [3] have also reported that sub-micron calcium carbonate enhances impact performance of rigid PVC, whereas coarser filler particles (2 and 3 µm) detracted from the notched impact strength.

A number of studies [5-8] focus on the importance of coating and good dispersion of ultra-fine or nano-CaCO<sub>3</sub> in rigid PVC formulations. Particle agglomeration reduces impact performance and so it is important to optimise the addition level, coating and mixing regime to obtain significantly enhanced mechanical properties.

In the present study, the optimum concentration of PCC is investigated in terms of its effect on impact properties. Also, experimental design has been used to investigate possible synergy between PCC and the impact modifier and processing aid in a Ca/Zn stabilised PVC-U window profile formulation. The extent to which PCC can be considered as an impact modifier and processing aid in PVC formulations is discussed.

#### 2. EXPERIMENTAL

## 2.1 MATERIALS

# 2.1.1 Precipitated Calcium Carbonate

Precipitated calcium carbonate was supplied by Solvay Advanced Functional Minerals. The material had a mean particle size of 55 nm, a rhombohedral particle shape and a specific surface area (BET) of 23 m<sup>2</sup>/g. The weight % of stearic acid coating, as measured by CHN analysis, was found to be 2.9. A scanning electron micrograph of the PCC is shown in Figure 1.

#### 2.1.2 Formulations

For studying the optimum addition level of the PCC, dry blends were prepared according to the formulations shown in Table 1.

In the experimental design, the basic formulation was used as shown in Table 1, but levels of processing aid, impact modifier and PCC were all varied. Each of these variables was tested at 3 levels: impact modifier at levels of 4, 5.5 and 7 phr; processing aid at levels of 0, 0.5 and 1 phr; and PCC at levels of 5, 7.5 and 10 phr. A central composite design was chosen, which is a face-centred cube with a mid-point. This involved 15 different formulations with two replicates, as shown in Table 2. Replicates were included to allow estimates of the precision of the experimental measurements. The experiments were carried out in a random order.

# 2.2 PROCESSING

Dry blends were prepared according to the formulations shown above. The PVC resin and other additives were blended simultaneously in a high speed Fielder mixer at 120°C at 2000 rpm and then cooled to room temperature in the cooling chamber.

The fusion behaviour of the PVC dry blends was studied using a Haake Rheocord 90 torque rheometer. For each test 60g of dry blend was charged into the chamber and mixing was carried out at a rotor speed of 60rpm and a temperature of 190 °C.

The Krauss Maffei KMD 25KK-L twin-screw extruder fitted with conical counter-rotating screws was used to extrude the dry blends into profiles. A strip die was used with cross-section 4mm x 30mm. Extrusion was carried out using the set conditions shown in Table 3.

#### 2.3 PROPERTY MEASUREMENTS

#### 2.3.1 Fusion Measurements

The level of fusion developed on processing a PVC formulation is well known to have a profound effect on the mechanical properties of the product [10 - 12]. Early studies on the fusion of PVC were carried out by Gilbert [13], Summers [14] and Allsop [15]. During processing, under the influence of both heat and shear, there is partial melting of the primary PVC crystallites and molecular diffusion. During cooling, recrystallisation occurs and a network structure is formed linked together by tie molecules and secondary crystallites, so that the original primary particle boundaries no longer exist.

Differential Scanning Calorimetry (DSC) is a well established technique for assessing PVC fusion [13, 16]. A typical DSC plot from a sample of processed rigid PVC is shown in Figure 2. The two endothermic peaks correspond to the secondary crystallinity (peak A) and the residual primary crystallinity (peak B). The extent of the fusion process can be determined either by measuring the enthalpy of peak A ( $\Delta H_A$ ), or from the relationship shown in Equation 1 below.

$$\% Fusion = \left(\frac{\Delta H_A}{\Delta H_A + \Delta H_B}\right) x 100\% \qquad (1)$$

In this study both methods have been used. Another very useful feature of DSC is that the true processing temperature can be determined, as shown in Figure 2.

Specimens of 10-12 mg were taken from the core of each extrudate and DSC thermograms were recorded using a DuPont 2010 Thermal Analyser. Specimens were heated from 40°C to 240°C at 20°C per minute.

# 2.3.2 Impact Testing

Impact testing was performed according to British Standard 7413 using a pendulum Charpy Impact tester. Specimens were prepared with a milling machine and notched with a single tooth cutter. For each formulation 12 specimens were tested.

# 2.3.3 Tensile Testing

Tensile testing was carried out using a Hounsfield tensile testing machine according to British Standard 2782 at a strain rate of 25mm/min.

#### 3. RESULTS

# 3.1 EFFECT OF PCC CONCENTRATION

#### 3.1.1 Fusion Measurements

Table 4 shows the results of time to gelation as measured in the Haake Rheocord torque rheometer. It can be seen that the fusion time decreased as the content of PCC was increased from 0 phr to 10 phr, whereas increasing the level from 10 to 15 phr gave no further reduction in fusion time. No reproducible result could be obtained with samples containing 20phr of PCC.

Differential scanning calorimetry (DSC) measurements on samples taken from the PVC profiles extruded on Krauss Maffei KMD 25KK-L also confirm that ultra-fine PCC promotes fusion. Results from these tests are given in Table 5.

It is seen from Table 5 that as the content of PCC is increased incrementally from 0 to 15 phr, there is an increase in actual processing temperature, enthalpy of Peak A (melting of secondary crystallites) and % fusion level but these values decline as more PCC is added. It can be assumed that the formulations with 10 and 15 phr PCC content produce the optimum interparticle friction and hence the highest PVC fusion.

# 3.1.2 Mechanical Properties

The effect of PCC content on the Charpy impact strength of the extruded profiles is plotted in Figure 3. These results clearly demonstrate a dramatic increase in impact strength with PCC contents of 10 and 15 phr. The drop in impact strength at the 20 phr content was found to be due to filler agglomeration.

The impact failure mode of the extruded profiles with 10 phr and 15 phr PCC loading was ductile while the profiles containing 0, 5 and 20 phr showed brittle fracture. Figure 4 shows a Scanning Electron micrograph of a fracture surface from a sample containing 10phr PCC. The morphology seen here consists of fibrils of highly strained, cold drawn PVC, indicative of ductile failure.

The values of tensile strength and elongation-to-break of the extruded profiles with different PCC loadings are shown in Table 6. Increasing the PCC loading from 0-15 phr gave no significant change in the tensile strength results. Further addition up to 20 phr PCC has shown a slight decrease in tensile strength, probably due to agglomeration of the filler. Results of elongation-to-break measurements confirm that ductility increases with addition of PCC at least up to a content of 10 phr,

which it was concluded was the optimum addition level in terms of enhancement of mechanical properties.

#### 3. 2 EXPERIMENTAL DESIGN

# 3.2.1 Fusion Measurements

Differential scanning calorimetry (DSC) results obtained for the experimental design formulations are shown in Table 7.

Multiple regression analysis was carried out on these results using the ECHIP statistical analysis software. There was found to be a statistically significant fit to the model for the % fusion data but no significant fit for the enthalpy data. The analysis showed that the most significant effect for increasing the % fusion was increasing the PCC content (from 5 to 10 phr). This had a greater effect than increasing the processing aid level (from 0 to 1phr). This is illustrated in the 3D contour plot of Figure 5. There was found to be no effect of impact modifier level.

# 3.2.2 Mechanical Properties

Mechanical property data from the experimental design tests are given in Table 8. Multiple regression analysis was carried out on these data to assess the effect of the formulation variables and combinations of these variables on the mechanical properties of the extruded PVC profiles. In the case of the Charpy impact data, it can be seen that there is a discontinuity in the data for formulation no. 8, where the fracture mode has switched from brittle to ductile failure. As discussed above, the fracture surface shows fibrillation, indicative of a ductile failure mechanism, and hence a very high impact energy is measured. This discontinuity in the data made it difficult to get a good fit for the regression analysis. Nevertheless, the results indicate that increasing the PCC content (from 5 to 10 phr) gave an equivalent improvement in the impact strength compared with increasing the impact modifier from 4 to 7 phr. There was also found to be a strong positive interaction between these two variables. These effects are shown in the 3D contour plot of Figure 6. Increasing the processing aid content also improved the impact strength but to a lesser extent.

There was found to be no statistically significant effect of the formulation variables on tensile strength.

# 4. DISCUSSION

The results of the two studies show consistent findings. Firstly, it is clear that PCC promotes the fusion of PVC, and in this respect acts as a processing aid. This is both when fusion rate is

measured in the Haake Torque Rheometer and when degree of fusion is measured using DSC. The reason that ultra-fine PCC can act as a processing aid is that its particle size is below that of the primary particles of PVC (0.2–1.0µm) and hence it is capable of generating inter-particle friction with PVC particles, increasing shear, improving additive dispersion and promoting fusion. However, conventional processing aids (usually high molecular weight acrylic copolymers) give rise to other attributes that have not been shown to be the case for PCC. In particular, high molecular weight, long chain acrylics become entangled with PVC chains during processing and thus improve the extensibility and strength of the melt. There is no evidence of PCC giving rise to improved hot melt strength from these studies.

Secondly, there is clear evidence from the studies that ultra-fine PCC improves the toughness of rigid PVC formulations when used in conjunction with impact modifier. Also that some impact modifier can be replaced with ultra-fine PCC and very good impact results still be achieved. For example, reducing the impact modifier level from 7phr to 4phr in the presence of 10 phr of PCC (experiment 7) gave a Charpy impact strength of 27 kJm<sup>-2</sup>, which is well above the pass for BS 7413 of 12 kJm<sup>-2</sup>.

The toughening mechanism of fine filler particles in poly(propylene) has been reported in a number of publications [17-19] and it seems that a similar mechanism applies to ultra-fine PCC particles in PVC. The PCC particles cannot deform when the PVC matrix is strained and so act as stress concentrators. The triaxial stress generated around the filler particles gives rise to debonding between the matrix and filler particles. Microvoids are formed and the stress state in the polymer between the microvoids changes from triaxial to biaxial or uniaxial and shear yielding takes place. Hence the debonded polymer is pulled into strands giving rise to a fibrillated fracture surface. Ductile yielding and fibrillation lead to the absorption of large amounts of local strain energy and hence the high values of impact strength measured.

# **CONCLUSIONS**

This study has confirmed that ultra-fine precipitated calcium carbonate promotes the fusion of PVC, and in this respect acts as a processing aid. Results from tests in the Haake torque rheometer on a Ca/Zn stabilised rigid PVC formulation showed that the fusion time decreased as the content of PCC was increased from 0 phr to 10 phr. Also results from DSC experiments on extruded PVC profiles confirmed that ultra-fine PCC promotes fusion. As the content of PCC was increased from 0

to 15 phr, there was an increase in processing temperature,  $\Delta H_A$  (melting of secondary crystallites) and % fusion level. Similarly DSC results from the experimental design programme showed that increasing the PCC content (from 5 to 10 phr) had a greater effect on fusion level than increasing the processing aid level (from 0 to 1phr).

There was found to be no effect of PCC on the tensile strength of extruded PVC profiles. However, toughness was significantly improved when PCC was used in conjunction with impact modifier. There was found to be a dramatic increase in impact strength with PCC contents of 10 and 15 phr. The impact failure mode of the extruded profiles with 10 phr and 15 phr PCC loading was ductile while the profiles containing 0, 5 and 20 phr showed brittle fracture. Results from the experimental design programme confirmed the synergy between ultra-fine PCC and impact modifier. It is proposed that PCC particles act as stress concentrators giving rise to local shear yielding of the PVC matrix.

# **ACKNOWLEDGEMENTS**

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Table 1. Formulation for Studying the Optimum Level of PCC

Ingredient	Concentration – Parts per hundred parts of polymer (phr)
Poly(vinyl chloride) – K66	100
Acrylic Impact Modifier	7
Ca/Zn Thermal Stabiliser	4.5
Acrylic Processing Aid	1.0
Titanium Dioxide Pigment	4
Precipitated Calcium Carbonate	0, 5, 10, 15, 20

Table 2. Levels of Variables in the Experimental Design

Experiment	Impact modifier	Processing aid	Filler level
number	level (phr)	level (phr)	(phr)
1	4	0	5
2	4	1	5
3	7	1	5
4	7	0	5
5	7	0	10
6	4	0	10
7	4	1	10
8	7	1	10
9	7	0.5	7.5
10	5.5	0.5	5
11	5.5	1	7.5
12	5.5	0	7.5
13	5.5	0.5	10
14	4	0.5	7.5
15	5.5	0.5	7.5
16	5.5	0.5	7.5
17	5.5	0.5	7.5

Table 3. Extrusion Conditions on the Krauss Maffei KMD 25KK-L

Die	Adapter	Zone 2	Zone 1	Screw	Screw
Temperature	Temperature	Temperature	Temperature	Temperature	Speed
200°C	200°C	190°C	180°C	150°C	20
					rpm

Table 4 Effect of PCC Content on Fusion Time

Filler Content	Gelation Time	
(phr)	(seconds)	
0	75.8 ± 4.1	
5	59.8 ± 0.4	
10	36.1 ± 0.2	
15	36.2 ± 0.8	

Table 5. Results from Differential Scanning Calorimetry (DSC) Tests

	Processing	∆H <sub>A</sub> - Enthalpy	
PCC	Temperature	of Peak A	%
Content (phr)	(°C)	(J/g)	Fusion
0	189.3 ± 0.3	$2.9 \pm 0.2$	81.8 ± 1.0
5	190.6 ± 1.0	$3.0 \pm 0.2$	83.2 ± 1.5
10	193.3 ± 0.2	4.3 ± 0.2	87.9 ± 1.1
15	193.7 ± 0.4	4.5 ± 0.4	91.8 ± 1.6
20	192.1 ± 0.4	4.0 ± 0.1	88.6 ± 1.8

Table 6. Tensile Testing Results for Extruded Profiles with Different PCC Loadings

PCC content (phr)	Tensile Strength (MPa)	Elongation at break (%)
0	42.7 ± 0.6	30.2 ± 2.0
5	43.4 ± 1.0	71.2 ± 14.7
10	$42.5 \pm 0.5$	96.9 ± 22.7
15	41.9 ± 0.4	50.9 ± 14.4
20	39.3 ± 0.6	19.5 ± 4.4

Table 7. Results from Differential Scanning Calorimetry (DSC) Tests

Experiment	Processing	Enthalpy of	Fusion
Number	Temperature (°C)	peak A (J/g)	(%)
1	190.8 ± 1.9	$4.0 \pm 0.4$	84.2 ± 1.7
2	192.9 ± 0.2	$3.1 \pm 0.3$	86.6 ± 0.9
3	190.6 ± 1.0	$3.0 \pm 0.2$	83.2 ± 1.5
4	195.7 ± 2.5	$3.1 \pm 0.5$	82.7 ± 1.7
5	191.6 ± 0.9	$3.3 \pm 0.2$	84.9 ± 1.4
6	190.6 ± 1.1	$2.9 \pm 0.2$	85.9 ± 2.1
7	191.9 ± 0.6	$3.5 \pm 0.4$	85.6 ± 1.1
8	193.3 ± 0.2	4.7 ± 0.2	89.3 ± 1.3
9	193.4 ± 0.7	$3.5 \pm 0.2$	88.5 ± 2.4
10	189.9 ± 0.8	$3.7 \pm 0.4$	84.6 ± 0.8
11	192.5 ± 1.1	$3.7 \pm 0.2$	86.1 ± 2.1
12	190.9 ± 1.6	$2.9 \pm 0.2$	83.0 ± 2.5
13	192.6 ± 0.4	2.6 ± 0.1	88.3 ± 1.2
14	191.0 ± 1.2	3.3 ± 0.1	85.1 ± 0.9
15	192.5 ± 0.5	3.5 ± 0.1	88.4 ± 1.7
16	191.7 ± 0.9	3.7 ± 0.1	87.7 ± 0.7
17	191.5 ± 1.3	$3.6 \pm 0.2$	85.5 ± 1.4

Table 8. Results of Mechanical Property Tests

Experiment	Impact Strength	Tensile Strength	Elongation at
Number	(kJ/m <sup>2</sup> )	(MPa)	break (%)
1	7.6 ± 1.5	45.9 ± 0.6	22.5 ± 5.9
2	16.3 ± 1.1	44.7 ± 0.3	57.6 ± 24.8
3	25.0 ± 1.8	45.4 ± 0.9	71.9 ± 20.5
4	18.5 ± 2.5	45.5 ± 2.4	104.3 ± 20.6
5	$33.3 \pm 1.4$	42.4 ± 1.3	40.5 ± 8.8
6	23.0 ± 1.6	44.3 ± 0.5	31.9 ± 3.6
7	27.3 ± 1.8	46.0 ± 0.3	62.6 ± 32.5
8	79.0 ± 1.9	42.5 ± 0.5	96.9 ± 22.9
9	29.4 ± 1.9	42.7 ± 0.5	45.4 ± 9.3
10	21.6 ± 1.9	45.8 ± 0.7	41.9 ± 8.9
11	25.9 ± 1.9	45.4 ± 0.4	68.5 ± 32.6
12	25.6 ± 1.7	42.8 ± 1.3	50.6 ± 14.5
13	27.3 ± 1.9	43.9 ± 1.3	50.7 ± 24.6
14	17.4 ± 1.5	46.9 ± 0.6	60.4 ± 15.8
15	24.6 ± 1.8	44.6 ± 1.6	40.2 ± 6.1
16	25.4 ± 1.6	47.5 ± 0.6	54.5 ± 26.5
17	24.7 ± 2.0	45.6 ± 0.5	68.8 ± 18.6

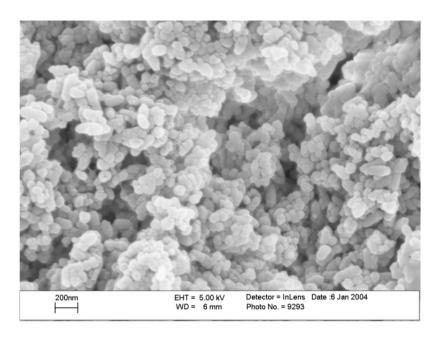


Figure 1. Scanning Electron Micrograph showing PCC Morphology

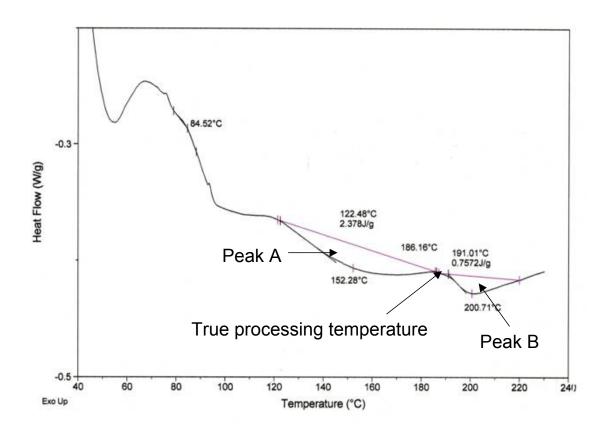


Figure 2. Typical DSC Plot of Processed Rigid PVC.

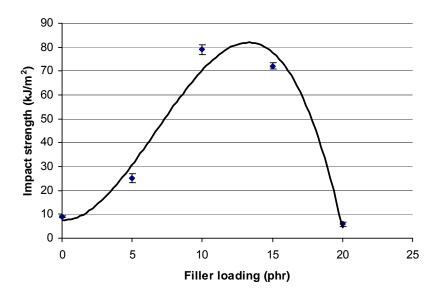


Figure 3. Effect of PCC Loading on the Impact Strength of Extruded Profiles

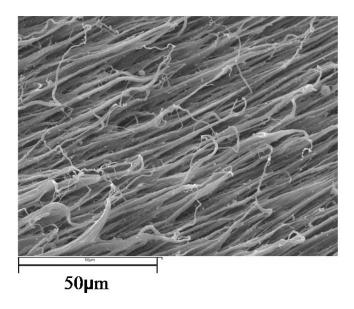


Figure 4. SEM Micrograph of Fracture Surface of Sample containing 10phr of PCC.

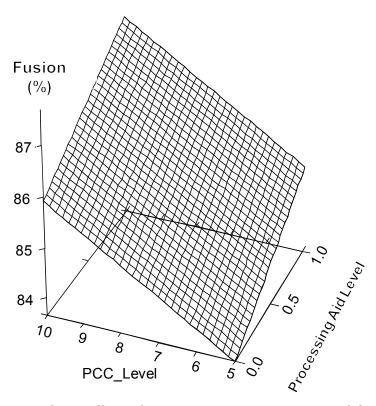


Figure 5. 3D Plot of the Effect of Processing Aid Level and PCC Level on % Fusion

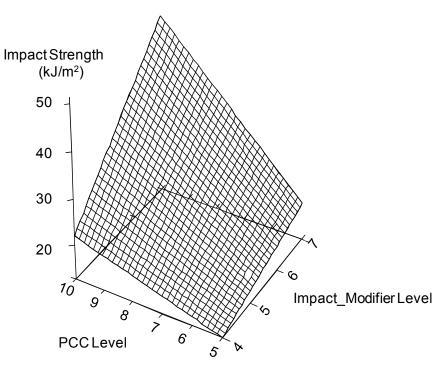


Figure 6. 3D Plot of the Effect of Impact Modifier and PCC on Impact Strength

# **Figure Captions**

- Figure 1. Scanning Electron Micrograph showing PCC Morphology
- Figure 2. Typical DSC Plot of Processed Rigid PVC.
- Figure 3. Effect of PCC Loading on the Impact Strength of Extruded Profiles
- Figure 4. SEM Micrograph of Fracture Surface of Sample containing 10phr of PCC.
- Figure 5. 3D Plot of the Effect of Processing Aid Level and PCC Level on % Fusion
- Figure 6. 3D Plot of the Effect of Impact Modifier and PCC on Impact Strength