THE USE OF BRABENDER PLASTICORDER TO STUDY THE FUSION BEHAVIOUR OF CALCIUM CARBONATE FILLED IMPACT-MODIFIED PVC-U

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Abstract *The properties of poly(vinyl chloride) can be modified through the incorporation of suitable additives such as fillers. Calcium carbonate is the most popular type of filler for PVC because of their excellent combination of low cost, high brightness, and the ability to be used at high loadings. Fusion has a profound influence on mechanical, physical and chemical properties. Due to these factors, various methods for assessment of fusion have been developed. In this project, Brabender Plasticorder mixing chamber was used to study the fusion behaviour of calcium carbonate filled impact modified PVC-U compounds. In this research, the effect of calcium carbonate particles size on the fusion properties of impact modified PVC-U was conducted. The other objective is to determine the effect of surface modification on the fusion properties. The result showed that the fusion time for the 0.8 μm particles size calcium carbonate filled impact-modified PVC-U had the shortest fusion time and highest torque. The fusion time increased with increasing calcium carbonate particle size. The coated calcium carbonate fillers increased the fusion time but decreased the melt viscosity compared to the uncoated calcium carbonate fillers. The use of the coupling agent increased the fusion time and increased the melt viscosity.*

Keywords: PVC; Brabender Plasticorder; fusion; calcium carbonate

1. Introduction

The properties of poly(vinyl chloride) (PVC) can be modified through the incorporation of suitable additives (1-4). Filler is a type of additive added to PVC in order to reduce cost or improve mechanical and physical properties. Calcium carbonate is the most popular type of filler for PVC because of their excellent combination of low cost, high brightness, and the ability to be used at high loadings (2, 5, 6).

Fusion can be defined as the process whereby the PVC agglomerates, primary particles, domain and mircodomains are attached together during processing. Previous studies have shown that fusion has a profound influence on mechanical, physical and chemical properties. In order to obtain optimum mechanical properties, an appropriate level of fusion is needed. Benjamin (7) found that the long-term properties of pipes are very dependent on fusion level, like pipe. The time to ductile/brittle transition increased with increased in fusion level. Meanwhile, Marshall (2) reported that well-fused pipe could withstand quite significant flows, while poorly fused samples can fail in a brittle manner from very small flows.

Due to the profound compounds influence of the degree of fusion of PVC on mechanical, physical and chemical properties, various methods for assessment of fusion have been developed. In this project, Brabender Plasticorder mixing chamber is used to study the fusion behaviour of PVC compounds. The Brabender Plasticorder is a torque measuring rheometer to which it can be interchangeably attached a number of different measuring heads. Because of its flexibility in shear rates and reasonably good temperature control, the plasticorder is uniquely suited to investigate the fusion behaviour of PVC. The use of Brabender Plasticorder also has proven itself an invaluable tool in the attempt to predict processability performance before committing large amounts of time and materials. Method of assessing the state of PVC fusion using a Brabender Plasticorder, has been developed by a number of workers (9-12). The most common and widely used is measuring the torque as a function of processing time for constant chamber temperature and rotor speed.

Although the addition of calcium carbonate fillers to impact-modified PVC-U (unplasticised PVC) formulation for structural applications is widely used, the research into their effect on the fusion behavior is still limited. Most papers and reviews reported on the effect of lubricants, processing aids and stabilizers towards PVC-U compound (13-15). In this research, the effects of calcium carbonate fillers in impact-modified PVC-U formulation will be studied. The objectives of this research are as follows:

(a) To investigate the effect of calcium carbonate particles size on the fusion properties
(b) To determine the effect of surface modification on the fusion properties.

2. Materials and Methods

The PVC used in this study is a suspension resin with solution viscosity K-value 66 supplied by IRM. The formulations are based upon a tin stabilised commercial window profile with a slight modification. The additives used are shown in Table 1.

Table 1 Blend Formulations

Ingredient	Parts per hundred (phr)	Suppliers
PVC	100	Industrial Resin Malaysia
Impact modifier - core-shell type acrylic modifier	6	Rohm and Haas
Fillers - ground calcium carbonate (GCC)	20	Malaysian Calcium Carbonate Sdn. Bhd.
Fillers - precipitated calcium carbonate (PCC)	20	Schaefer Honaik Sd. Bhd.
Fillers – coated calcium carbonate (CCC)	20	Sun Minerals
Coupling agent – LICA 38	0.2	Kenrich Petrochemicals
External lubricant - stearic acid	1.5	Ciba-Geigy
Internal lubricant - Calcium stearate	1.0	Ciba-Geigy
Heat stabiliser – Atofina 890F)	2.0	Rohm and Haas
Processing aids –polymer acrylic	2.0	Rohm and Haas

Numbers indicates parts per hundred resin (phr) in blend.

The amount and types of fillers in the blend formulations are shown in Table 2. Blend S1 is a control sample without any fillers.

Table 2 Filler Types and Content in Formulation

	S1	S2	S3	S4	S5	S6	S7	S8
0.8μm PCC	0	20	0	0	0	0	0	0
1 μm GCC	0	0	20	0	0	0	0	0
3μm GCC	0	0	0	20	0	0	0	0

6μm GCC	0	0	0	0	20	0	0	0
10μm GCC	0	0	0	0	0	20	0	0
1μm CCC	0	0	0	0	0	0	20	0
1 μm GCC + coupling agent	0	0	0	0	0	0	0	20

Numbers indicates parts per hundred resin (phr) in blend.

A high speed laboratory mixer was used to blend PVC and the additive after which the PVC dry powder blends (around 56 gram) was placed in the internal mixer. To ensure a good exposure of the dry powder blend to air during mixing, the cavity was not filled completely. All the samples were run at a mixer temperature of 185 °C with a rotor speed of 30 rpm. A 5 kg loading chute was used to introduce the powder blend into the mixer as quickly as possible (within 20 seconds) for best reproducibility and comparability of test result. This fusion behaviour was studied by observing the changes in curve torque.

3. Results And Discussion

A processibility study was conducted to investigate the fusion behavior of filled impact-modified PVC-U using Brabender Plasticorder with fitted mixing head. The present investigation is an attempt to analyze and compare the effect of the particles size of calcium carbonate fillers on the fusion behavior of impact-modified PVC-U. The effects of surface coating and coupling agent were also studied.

The effects of calcium carbonate particles size on fusion time and fusion torque of impact-modified PVC are illustrated in Figure 1. Fusion time is when the primarily particles disappear due to the fusion of the particles (9). Comparisons were made between PCC (0.8 μm) and GCC (1 μm, 3 μm, 6 μm and 10 μm) filled impact-modified PVC-U samples. The content of calcium carbonate was fixed at 20 phr. The result showed that the fusion time for the 0.8 μm PCC filled impact-modified PVC-U was shortest followed by GCC 1 μm, 3 μm, 6 μm and 10 μm. The result also showed that the fusion torque values decreased with increasing calcium carbonate particle size. Generally, the shorter the fusion time the higher is the fusion torque.

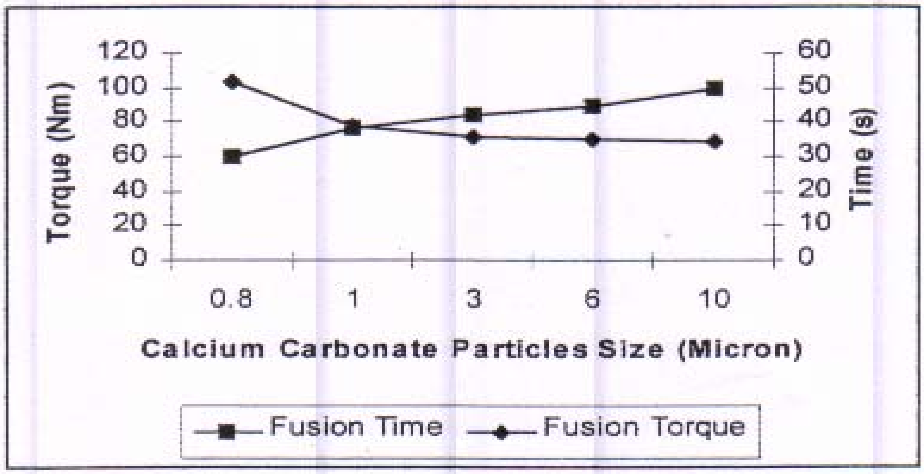

Figure 1 Effect of Calcium Carbonate Particles Size on Fusion Time and Fusion Torque of Impact-Modified PVC-U

The manufacturer's data sheet confirmed that the melt viscosity for PVC compound increases with decreasing calcium carbonate particles size (16). A number of reviews also stated that fusion process is more likely to happen in a viscous material, in which the shear between particles is high enough (17-19).

Finer particles size of calcium carbonate provides more particles per unit volume and therefore smoother surfaces and more uniform properties compared to coarse particles. Thus the finer particles size of calcium carbonate is able to form more powerful and viscous material so that the primarily particles of PVC fused together easily. PCC, which have more uniform particles morphology, narrow particle size distribution and finer particles size compared to all GCC samples would have the tendency to fuse faster and easier. Since the finer particles size calcium carbonate filled sample produced higher melt viscosity, more force is needed for the compound to fuse together. The result suggested that 1μm calcium carbonate was the most optimum choice among the uncoated calcium carbonate for the processing of rigid PVC because its fusion time was the lowest and the melt viscosity was moderate.

The values of the end torque decreased with increasing particles size as shown in Figure 2. Many researchers have proved that the end torque value is proportional to melt viscosity of the sample (11, 20). Reduction of the end torque means reduce in melt viscosity which means less force is required to continue mixing and homogenizing the fused stock.

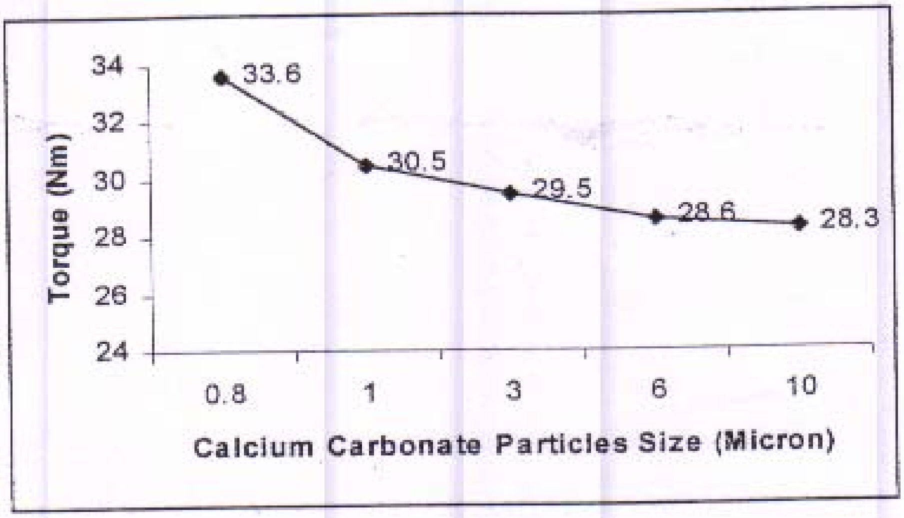

Figure 2 Effect of Calcium Carbonate Particles Size on End Torque of Impact-Modified PVC-U

The larger particles size calcium carbonate have low surface area and require least energy to disperse (21). The driving forces are minimal and the aggregates formed are generally weak. Conversely, very high surface area types are the most difficult because penetration of the dispersing liquid into the very narrow pores of the agglomerates requires a very efficient dispersant and considerable mechanical work (22).

Figure 3 shows the effect of surface treated calcium carbonate on fusion time and fusion torque of impact-modified PVC-U. A comparison was made between sample S1, S3, S7 and S8. S1 was the control sample with no fillers added. S3 and S7 were uncoated and coated GCC respectively, while S8 was uncoated GCC with 0.2 phr coupling agent. The impact modifier content and particles size of the filled sample were fixed at 6 phr and 1 μm respectively. As shown in Figure 3, sample S1 had the shortest fusion time value followed by sample S3, S17 and S18. However, sample S1 had the highest fusion torque compared to the others samples. There have been few reports on the effects of calcium carbonate fillers on melt processing (21, 22, 23). The studies showed that surface-treated calcium carbonates significantly reduced peak torque and melt viscosity versus untreated calcium carbonate in typical calendered vinyl compound.

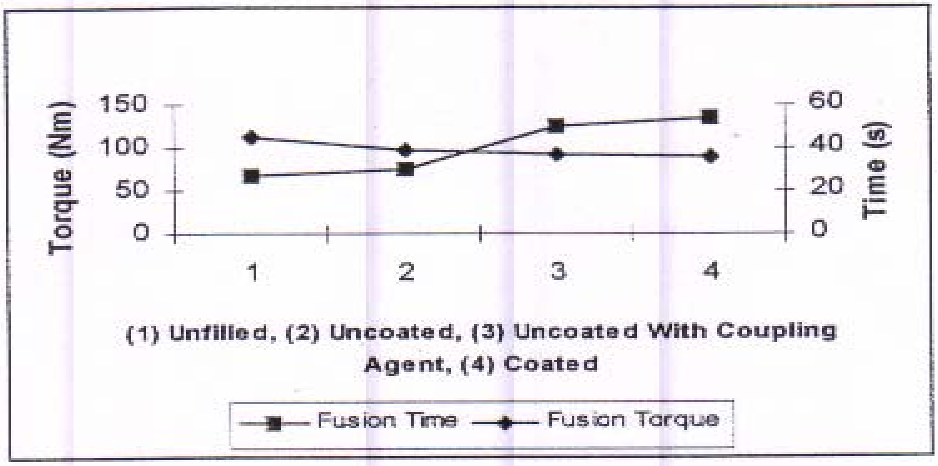

Figure 3 Effect of Surface Treated Calcium Carbonate on Fusion Time and Fusion Torque of Impact-Modified PVC-U

In another report, Tapper (6) stated that the addition of calcium carbonate increases melt viscosity, but the increase can be reduced by using surface treatment such as 1% stearic acid on filler in rigid PVC. Certain titanates are claimed to be more effective than stearic acid at filler phr higher above 60 based on equilibrium torque determined with a torque rheometer.

The information above seems consistent with our present study. As mentioned earlier, shear is one of the major factor that controls the fusion of PVC. Higher shear between primarily particles resulted in a better fusion of the primarily particles. The material will become more viscous and consequently, will raise up the fusion torque. The present results indicate that unfilled samples (S1) have shorter fusion time with higher fusion torque compared to filled sample (S3). By adding coupling agent or coated the GCC stearic acid, the fusion torque dropped with much longer fusion time. The most probable reason for this is better dispersion and adhesion of surface treated GCC in the PVC matrix.

Figure 4 indicates that, addition of 20 phr GCC increased the end torque value, However the values decreased when the GCC was surfacing treated with coupling agent or coated with stearic acid. The result shows that uncoated GCC sample (S3) has the highest melt viscosity, followed by unfilled sample (S1), uncoated with coupling agent (S8) and lastly coated GCC (S7).

The presence of a monomolecular layer of a surface coating such as fatty acid results in soft agglomerates that are easier to break down by low-level mechanical energy (21). So, surface treated calcium carbonate tend to cause the reduction in torque.

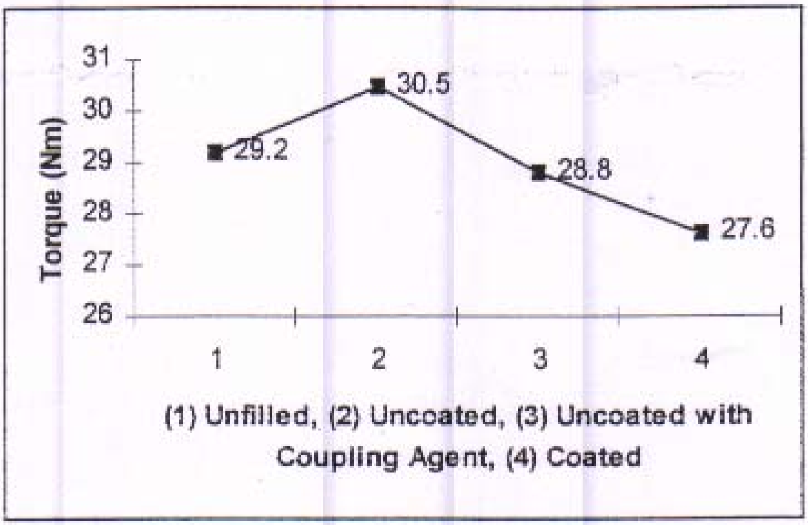

Figure 4 Effect of Surface Treated Calcium Carbonate on End Torque of Impact-Modified PVC-U

4. Conclusions

(1) 0.8μm particles size calcium carbonate filled impact-modified PVC-U had the shortest fusion time and highest torque. The fusion time increased with increasing calcium carbonate particles size.

(2) The melt viscosity decreased with increasing calcium carbonate particles size.

(3) The coated calcium carbonate had higher fusion time but lower melt viscosity compared to the uncoated ones.

(4) 1μm calcium carbonate was the most optimum choice among the uncoated calcium carbonate for the processing of rigid PVC because its fusion time was the lowest and the melt viscosity was moderate

(5) The application of coupling agent decreased the fusion time but increased the melt viscosity.

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