# Turkish Online Journal of Qualitative Inquiry (TOJQI) Volume 12, Issue 7, July 2021: 9790 – 9803

#### Research Article

### Analysis of Material Viscosity Variations: Mix Processing PC1/PC2 Composites

Jamal Al Sadi<sup>a,</sup> Aysar Hawari<sup>b</sup>

a,b Department of Engineering, (Jadara University), Irbid, Jordan

### **Abstract**

The present study investigates the effect of the mixing and dispersion process of polycarbonate materials (PCs). The composite was prepared with different melt flow indexes of polycarbonate material. The steps of the (%PC1/%PC2) blends were (100%/0%), (30%/70%), and (0%/100%) (Weight percent). The blends were imparted with the mixing materials with pigment and without pigments. The weight percentage of the two-polycarbonate resins for (PC1/PC2) were PC1 content (30wt%-pph) of MFI (25gm/10mins) and PC2 content (70 wt. %-pph) of MFI (6.5gm/10mins). The minute formulation of PC/PC2 blends, temperature effect on viscosity and pigment dispersion were investigated. This work simulated the Carreau model to fit the viscosity curves at 230, 255, 280°C and for the Master curve at reference 255°C . The color of PC composites was measured according to a typical spectrophotometer. Increasing the temperature and weight percentage of PC1 with (higher melt flow index) showed a significant effect in lower viscosity value and decreased color matching values (dE). The formulation and processes showed an excellent effect on controlling the viscosity, and microtomed plastic sections performed characterizing to different thicknesses and temperatures. The optimal number of particles was increased at higher temperatures and thickness. The average pigment size measured approximately 0.1-0.2 µm. Microscope Observation of Morphology observations showed an excellent dispersion colour matching at lower viscosity.

**Keywords:** blends, Minute formulation, Viscosity, MFI, Master Viscosity Models, Microtome - characterization

#### Introduction

Plastics manufacture is an important industrial sector in the world. However, producing the right plastic color with minimal rejection is a challenge for plastic manufacturers. Producing plastic with a marketable color requires adding one or more resins; however, achieving the correct colour in the first attempt is a challenge. The color properties of the polymer are directly affected by formulation for various ratios of PC resins. The actual MFI or the viscosity for a specific ratio may vary due to formulation such as (PCs weight percent blends, pigments, and additives), and processing parameters like temperature, feed rate, and screw speed.

Various researchers have carried out a few studies regarding the effects of processing parameters by the dynamic mixing in a screw extrusion during the compounding of polymers [1, 2]. If the viscosity of the blend is too low or too high, the induced shear will be inadequate for optimum dispersion. However, viscosity is also an essential indicator of the stability of a pigment concentrate. If it changes during storage, the pigments are usually inadequately stabilized. In order to control the mentioned points, a system of additives and resins can be formulated to modify viscosity and improve thermal stability to have better dispersion [3]. Researchers have studied the importance and effects of adding pigment to the base resin. It has been shown that incorporating additives into polymeric materials during production often unpredictably affects rheological and optical properties [4-6]. However,

rheological properties are an essential link between the processing steps and the final performance of the product [7-8].

Polymer blending is a vital field of polymer science and has been reviewed by many scientists. The PC/PBT blends are transparent in the melt state, and most transparent in the solid-state are partially miscible blends, as was shown by Sanchez et al. [8]. In their work, Liang and Gupta (2000) studied the rheological properties of a recycled PC blended with virgin PC; they stated that separated PC could be added to pure PC up to the 15% level without significantly varying the properties of pure PC [9]. Khan et al. developed in their study characterizing alloys of ABS and PC through blending these two materials [16]. Lee S. et al. studied the rheological behavior of PC/Polyester blends and their phase behaviour [10]. However, they found that the blends do not follow the rule of mixtures, which is common in all studies. The particle sizes, fillers (pigments and additives), and processing parameters like temperature and pressure affect the rheology and the colour of the pigmented polymer. The significance of the processing variables was correlated with the rheological result [11]. Difficulties with the dispersion of pigments or obtaining a uniform blend can be overcome by reducing the resin viscosity and extending the mixing time [12]. Rheology as a concept is relevant for all color mismatch, which can arise, due to problems with formulations, interactions between materials and processing conditions, such as high operating temperatures or high processing shear rates, and Poor pigment dispersion Variations in the raw material properties can also be part of the problem. A system of additives and resins can be formulated to modify viscosity, increase mechanical prosperities, improve thermal stability, or improve wear performance [13].

Many researchers have studied the importance and effects of adding the pigment to the base resin, especially since it has been shown that incorporating additives into polymeric materials during production often affects rheological, mechanical, and optical properties unpredictably [14, 15,16]. Rheology as a conception applies to materials (solid, semi-solid and fluid) like polymers and their composites. In addition, rheological properties are an essential link between the processing steps and the final performance of the product [17, 18].

This study included additional literature reviews, investigation for the rheological characterization of the materials, Melt flow adjustment (MFA). Haake mixer and viscosity measurements [19, 20, 21, 22, 23]. More recent studies have indicated that the effective processing conditions were dependent on the materials used or blending different materials (resins, additives, pigments, and fillers) to improve the rheological characteristics [24, 25, 26, 27].

The effect of particle sizes, parameters like temperature, pressure, and amount of additive can be determined and used for improving processing conditions [28, 29]. The significant result of the processing variables was correlated with the rheological result. The viscosity was shown to affect the dispersion operation of the pigment in the plastic.

The pellets from each production run were moulded to get the sample chips, which were then used to measure colour coordinates on Color-Eye- 7000A spectrophotometer of X-Rite. Any deviation of output colour from the desired target can be reported as DL\*, Da\*, Db\* - the Euclidean distance of color deviation in the color space, which associates three coordinates L\*, a\*, and b\* to a color referred as its tristimulus data. L\* values represent the lightness of a color ranging from 0 (black) to 100 (white), whereas a\* and b\* represent redness-greenness and yellowness-blueness, respectively. The instrument settings were as follows: color space – CIE Lab; measurement mode; observer angle – 10° and D65 illuminant (light source) [30]. The deviation in color DE from a target is dimensionless and can be calculated using the Pythagorean Theorem equation (1) as follows:

$$DE = \sqrt{(L1 - L2)^2 + (a1 - a2)^2 + (b1 - b2)^2}$$
 (1)

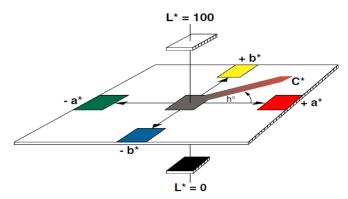


Figure 1. Color in the CIE 1976 (L\*a\*b\*) color space [31]

A colorist may be asked to match a color with a known reference color or compare two colors. Delta E\* is a single number indicating the difference in color between two readings and is based on the L\*, a\*, and b\* color space system. This is the geometric distance, delta E\*, between two points in the three-dimensional space. But delta E\* is not linear throughout color space and is not a good measure of color perceptions. Therefore, more complicated computational methods have been developed to predict color differences between two samples. [31]

This type of mixing is a high-shear process that breaks up large particles and disperses them as smaller particles throughout the melt. It is also referred to as intensive mixing. Dispersive mixing is a process used to blend two or more polymer resin systems and disperse pigments and liquid additives. [32]. Elements and screw geometries are used to accomplish each type of mixing. Mixing effectiveness or the level of mixing is dependent upon screw speed, percentage of fills in the screw elements and geometry, temperature, and shear rate. Shear rate and screw geometry also affect and influence the resin viscosity. [33]. Extruders help to achieve a constant and uniform melt temperature and pressure, and thus, a homogeneous product. These factors all relate to polymer rheology. The following simulates the effect of the addition of colorant on compounding plastic as were rheologically tested, which is reflected in lowering viscosity and decreasing the absorbance value. By taking the above results into account, it makes sense that the addition of colorant will minimize the viscosity and absorbance mechanisms here for degradation and yellowing will be decreased as was seen for samples subjected to an earlier study [34-35].

In this study Lexan\* PC resins were subjected to various blends formulations steps with additives and pigments (WA) and without additives (WOA). Functional groups and characteristic absorptions results were identified. Characterization results were correlated to the specific processing conditions of two different melt flow indexes polycarbonates with and without additives. This work is designed to identify the rheological properties of polycarbonate blends without (WOA) and with the addition of pigment and additives (WA) while changing the blend proportions in discrete equal intervals. The steps of the (%PC1/%PC2) blends were (100%/0%), (30%/70%), and (0%/100%) (Weight percent). The focus was extended to the polycarbonate grade (30-70%) to compare the impact of the dispersion and rheological characteristic. The minute formulation of PC/PC2 blends, temperature effect on viscosity and pigment dispersion were investigated. This work simulated the Carreau model to fit the viscosity curves at 230, 255, 280°C and for the Master curve at reference 255°C. Increasing the temperature and weight, the percentage of PC1 with (higher melt flow index) showed a significant effect in lower viscosity value and decreased color matching values (dE). Formulation and processing parameters showed an excellent effect on controlling the viscosity. Microscope Observation of Morphology observations showed an excellent dispersion color matching at lower viscosity.

### **Results & Discussions**

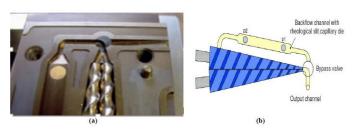
## 2.1 Compounding Extruders

The compounding process was carried out using two different extruders:

**Extruder 1.** The first extruder was an intermeshing co-rotating twin-screw extruder (TSE) manufactured by Coperion Germany(**Figure 2**). The pellets were then moulded via injection-moulding into three rectangular color chips (3x2x0.1"). For further optical microscopic tests and characterization measurements, the specimen was then dried at lab room temperature. For simplicity, the compounding process that involved this extruder is **abbreviated 'SB'** in this document.



**Figure 2.**Co-Rotating twin-screw extruder(SB)



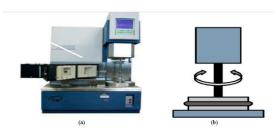
**Figure 3.** (a) Haake Minilab system and (b) schematic of the design (ML) [36]

**Extruder 2.** Thermo Haake Minilab II twin-screw micro compounder manufactured by Thermo Fisher scientific (**Figure 3**). For simplicity, the compounding process employing this extruder is abbreviated 'ML' here forth.

### 1.2 Rheological Equipment

**Table 1.** The composition of compounding material (g3)

SN	Ingredients	Material Name	PPH
1	R1	Bisphenol A (BPA)	30
2	R2	Bisphenol A (BPA)	70
3	F 1	Weather-resistant(L)	0.035
4	F 2	Stabilizer (Liquid)	0.065
5	F 3	Light Stabilizer	0.2
6	White	White Pigment	0.278
7	Black	Black Pigment	0.036
8	Red	Red Pigment	0.175
9	Yellow	Yellow Pigment	0.071



**Figure 4.** Ares-G2 rotational rheometer (a) ,(b parallel plate (TA)[37]

Rheology can be quantified through the use of rheometers and melt flow indexers.

**Rotational rheometer.** The rheometer used was Ares-G2 from TA Instruments(**Figure 4a**). We used the parallel plate geometry(**Figure 4b**). dynamic viscosities measurements were performed in a parallel-plate fixture with a diameter of 25 mm and a gap size of 1.0 mm. The testing sample was

sheared between the two plates. The measurement of the viscosity was the result of the ratio of the applied stress and the applied deformation rate

*Melt flow indexer.* A melt flow measurement device manufactured by Tinius Olsen, Model MP600M, was used to investigate the rheological characteristics of different polycarbonate formulations.

### 1.3 Experimental Procedure and Materials

This research investigated two grades of PC resins referred to as R1 and R2 in the thesis, each having a different melt flow index (MFI). One had an MFI of 25 and the other had an MFI of 6.5g/10min, respectively, here forth. The resins are manufactured by General Electric (GE) and traded under the name of Lexan. Four different color pigments, black, white, red and yellow, were used. Three additives were also used, called F1, F2, and F3 in this work. One was a stabilizer, one a light stabilizer, and the third offers weather-resistant properties.

Materials were extruded in an intermeshing, 25.5 mm, Coperion twin co-rotating screw extruder(SB). The total weight of the color additives (pigment and additive) was 0.86%. The two PC resins, R1 and R2, were used in a ratio of 30 and 70 wt. %, respectively. **Table 1** shows the formulation used. The additives and pigments were mixed with the resins at a 100:0.86 ratio and were batch blended by a super floater. These pellets were then molded using the injection-moulding machine to produce rectangular color chips (3x2x0.1" in dimensions). Utilizing a spectrophotometer, color measurements were carried out at three different spots in each specimen (coupon), to obtain the tristimulus values (L\*,a\*,b\*). The target values for L\*, a\*, b\* were defined as 68.5, 1.43, and 15.7, respectively. The color differences were then measured as dL\*, da\*, db\*, and dE\*.

To study the effects of the blending of two resins, a Thermo Haake Minilab II twin-screw micro compounder (ML) was used to prepare PC formulations with both resins individually as well as by varying the composition of the resins in steps of ten for eleven blends. The concentration ratios between the two polycarbonate resins used in %R1/%R2 were 100%/0%), 90%/10%, 80%/20%... and 0%/100% (shown in **Figure 5**. The eleven batches were prepared both with and without pigments and additives (WA&WOA) to characterize the viscosity. **Figure 5** presents a schematic that summarizes the steps in the compounding process

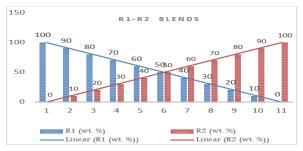


Figure 5.Composition of R1-R2 (polycarbonate resins) blends

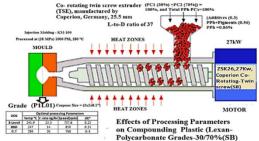


Figure 6. Schematic diagrams of process methods of plastics

### 1.4 The Compounding of Colored Plastic

The primary compounding processes used in the industry are either continuous mixing or batch mixing, after which a single screw or twin-screw extruder processes the material. The output material strands are cooled and then cut into small pellets by a pelletizer. These pellets are sold to manufacturers to be used as raw materials for their products. The eleven batches were prepared both with and without pigments and additives (WA&WOA) to characterize the viscosity. In addition, strain shear rate (SSR), dynamic strain sweep (DSS), and dynamic frequency sweep (DFS) were used to study the material's rheological properties and simulate viscosity models. **Figure 6.** Presents a schematic that summarizes the steps in the compounding process.

### 1.5. Processing conditions with temperature variation

The following tables show the experimental processing conditions. The general trends (GT) experimental design is shown in **Table 2** [38].

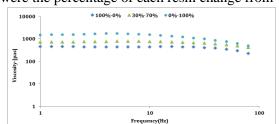
RPM	BZ1 (°C)	BZ2 (°C)	BZ3 (°C)	BZ4 (°C)	BZ5 (°C)	BZ6 (°C)	BZ7 (°C)	BZ8 (°C)	BZ9 (°C)	<b>DZ1</b> (°C)	Feed Rate (kg/hr)	DE
750	70	195	230	230	230	230	230	230	230	230	25	1.10
750	70	195	255	255	255	255	255	255	255	255	25	0.34
750	70	195	280	280	280	280	280	280	280	280	25	0.30

Table 2. Processing conditions with temperature variation [38]

Three parameters, including temperature, speed, and feed rate, were varied individually at three different levels while maintaining all other parameters fixed. This was referred to as general trends (GT). The selected processing temperatures were 230°C, 255°C and 280°C, with a speed and flow rate fixed at the middle values (750 rpm and 25 kg/hr, respectively). **Error! Reference source not found.** describes the design of the experiment of effect of temperature on colour output (dE\*). A sharp decrease in the color difference, dE\*, is evident when increasing temperature from 230°C to 240°C, beyond which near-constant colour values are observed up to 280°C.

# 1.6 Effect of Viscosity on PC (0%, 30% and 100%) blends

The first study to be undertaken was to investigate the effects of blending ratios on the rheological properties of two polycarbonate resins (R1, R2) comprising the selected PC grade. To do this, blends were prepared in which the resin ratios were controlled in steps of 10%. This resulted in three batches were the percentage of each resin change from



10000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

1000

Figure 7: Viscosity versus Steps of variations in PCs blends

Figure 8. Complex viscosity for R1 30% blends

**Figures 7.** is shown the effects of the following variations of rheological behaviors:

The Variation in the processing material; caused variation in viscosity; also, it is an influential factor in the rheological characterization. It is shown that the viscosity is affected at a particular minute variation of PC. The most

Influential factors on viscosity are the formulation (Wt. %) of polycarbonates; When resin R1 has a higher MFI and higher weight content of Polycarbonate, it will always reduce the viscosity.

Ultimately, when decreasing R1 to 30%, the viscosity increased. **Figure 8** indicate that as the temperature was increased, the composites showed a decrease in the complex viscosities,  $|\eta^*|$  of the PC compounds, and shear thinning occurred

In this study, the same methodology and the same technique was used in order to determine the degree of dispersion of PC compounds at temperatures of 230, 255, and 280°C. *Error! Reference source not found.*. illustrates the effect of temperature on color difference (dE\*) at temperatures of 230, 255, and 280°C. It reveals a reduced value of colour difference when increasing the temperature at a fixed feed rate and speed. However, the most favourable color difference value (dE\*) was obtained at the higher temperatures, namely 255°C and 280°C, with dE\*=0.3.

This phenomenon at a higher frequency and temperature decreases viscosity, decrease dia of pigment, decrease color value and increases pigment-wetting, as described in **Figure 9**. Wetting is an important aspect for the reason that shear forces produced during extrusion have to be transferred onto the pigments in order to deagglomerate particles, which reduces average pigment size and increases the number of particles (frequency) dispersed, ultimately reducing the color shift (dE\*). Therefore, the shear-thinning observed at the crossover point showed a lower colour difference value, indicating improved pigment dispersion.[38]

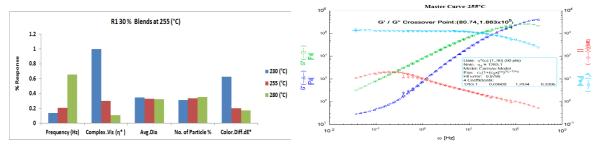


Figure 9. Effect of rheological parameters Figure 10. Master curve at reference on color 255°C w.r.t Hz

### 1.7. Modelling and Experimental Simulation of Viscosity

### 1.7.1. Compounded Grade Carreau model

Error! Reference source not found. shows the fitting parameters of the Carreau model for the blend at temperatures of 255°C. These simulated parameters for specific polymer blends, according to **Equation 2**, are given below. Note, C<sub>1</sub> indicates the absolute viscosity. The Carreau model is selected as a description for the viscosity behaviour of the compounds as it performs high regression values at various temperatures from 230, 255, and 280°C. Orchestrator software was used to plot the rheological characteristic curves and simulate the Carreau viscosity model by finding the four coefficients (C1, C2, C3, and C4); as expressed below in Error! Reference source not found.

Table 3. Carreau model parameters for SB (30/70%) at 230, 255, and 280°C

	Grade G3							
Temp	C1	C2	<i>C3</i>	C4				
230°C	4752.8	0.08	1.13	0.17				
255°C	1264.6	0.02	1.35	0.07				
280°C	447.2	-2.548x1019	-1.61x1018	1.11				

Table 4. Fitting parameters of Carreau model, PC blend (ML) with (WA), and (WOA)

Temp	Parameters (WA)				Parameters (WA) Parameters (WOA)				
°C	C1	C2	C3	C4	C1	C2	C3	C4	

# Jamal Al Sadi, Aysar Hawari

	230	1796.2	0.0724	2.8261	0.3169	6796.9	0.0848	1.3201	0.2096
Ī	255	452.79	0.0209	16.12	0.3232	1299.1	0.0223	1.6618	-0.002
Ī	280	191.87	0.0899	1.4858	1.0199	378.1	271.28	-18.67	0.9955

#### 1.7.2. Master Curves Careau Model

Master curves are normally extracted for extending material functions. This offers information about the short- and long-term behaviour of the materials. A master curve is of great value since it covers times or frequencies outside the range easily accessible via experimentation. Fitting the experimentally determined shift factors to a mathematical model permits the master curve to be shifted to any desired temperature. It offers information about the short- and long-term behaviour of the materials. The fitting parameters of the Carreau model for the master curve at the reference temperature of 255°C are given in and illustrated in *Error! Reference source not found.*.

	Processing Parameters								
Grade	C1	C2	<i>C3</i>	C4					
143-M3	1283.1	0.037	1.34	0.34					

Table Error! No text of specified style in document.. Carreau model for master curve

Тетр	Size-dia (micron)	Particle Nos (%)
230°C	0.78	48.9
255°C	0.79	52.4
280°C	0.84	55.3

**Table 7. Effect of Temperature on Dispersion** 

### 1.8. Effect of Temperature on Dispersion

Error! Reference source not found. illustrates the pigment size distribution at the three temperatures in order to represent the pigment size distribution. The data analysis showed that at the highest selected temperature of 280°C, 55.3% of the particles were approximately 0.84 microns in size compared to 52.4% of particles having a size of 0.79 micron at 255°C, and 48.9% of particles having a size of 0.78 at 230°C. Again, differences in color measurement are in good agreement with the particle size distribution in Error! Reference source not found. The results are shown at higher peaks (particle Nos %), reveals the lowest colour difference

Error! Reference source not found. illustrates the colour output in DE (deviation from target) at the investigated temperatures. The DE was 0.3 at 280°C and 0.35 at 255°C, in comparison to 1.1 at 230°C. The results agree with Table 7, which clearly shows a narrow peak in size distribution at 280°C, which drove the reduction in deviation from target due to better pigment dispersion.

### 1.9. SEM Characterization



The SEM micrograph,

**Figure**, depicts the presence of agglomerates in red pigments. It reveals primary particles that had a spherical shape in the vicinity of  $0.1~\mu m$  in size. The figure shows agglomerates consisting of primary particles of elliptical or cylindrical shapes with a diameter of approximately  $0.1~\mu m$ . Similarly, agglomerates were found in yellow, black and white pigments with primary particles in a spherical shape with  $10~\mu m$  and  $0.1~\mu m$  diameters.

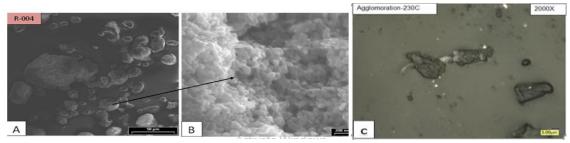


Figure 11. SEM micrograph of Red magnified Fig to 2000X pigments

Figure 12. DOM-agglomerations for 230°C

# 1.10. Influence of Viscosity on Pigment Dispersion

One of the significant parameters influencing the pigment dispersion is the viscosity of the (blend) matrix. The viscosity should be low for rapid pigment wetting, whereas for rapid de-agglomeration, the viscosity should be high. This indicates that, for optimum dispersion, there should be an intermediate and sufficient viscosity to promote both desired outcomes: dispersion and deagglomeration. **Table 2.** Processing conditions illustrate the color output in terms of DE (deviation from the target) at the investigated temperatures. The DE is 0.30 at 280°C and 0.34 at 255°C, compared to 1.1 at 230°C. It illustrates that the viscosity at 280°C and 230°C is quite adequate for wetting the particles. The reduction in viscosity and surface tension at the higher temperatures of 255°C and 280°C can be explained by enhanced wetting of pigments compared to the wetting prevalent at 230°C. This phenomenon can be explained by the Washburn equation given below. [39]

$$I(t) = \sqrt{\frac{C \cdot \bar{r} \cdot \gamma_L \cdot \cos \theta}{2 \cdot \eta}}$$
 (3)

I(t), C,  $\bar{r}$ ,  $\gamma_L$ ,  $\eta$ , and  $\theta$  represent the flow of the liquid, the pigment specific constant, the average pore radius of the agglomerate, the surface tension of the fluid, the dynamic viscosity of the fluid, and the contact angle between pigment and fluid, respectively. These results are also supported by **table 7**, which clearly shows that the highest peak of size distribution belonged to the blend processed at 280°C, which in turn led to a decrease in deviation from the target color values, i.e. lower DE, as presented in *Error! Reference source not found*. and **Table 2**. Moreover, comparing the peak height of the blends processed at the two temperatures of 255°C and 280°C reveals that a decrease in the particles size distribution peak by 4% changed the DE value from 0.30 to 0.34. However, concerning the re-agglomeration of particles, two phenomena are playing key roles: (1) the nature of the polymeric chain and (2) the higher the interfacial tension. These two phenomena can promote the reagglomeration of particles to form flocculate. [40, 41]

# 1.11. Morphology of Pigments

The morphology was examined by digital optical microscopy (DOM) and scanning electron microscopy (SEM). The optical microscopic graphs for the processing parameter samples are shown in **Error! Reference source not found.** through *Error! Reference source not found.* 

The degree of dispersion was enhanced at higher temperatures. For instance, dispersion at 255°C seemed to be somewhat better than that at 230°C. The micrographs in *Error! Reference source not found.* show that the higher the temperature is, the lesser the agglomeration evidence. **Error! Reference source not found.** 

Figure 13. SEM micrograph of polycarbonate grade compound at 230, 255, and 280°C

According to these results, the sample processed at 280°C showed better dispersed pigment than that processed at 255°C, which in turn, was better dispersed than that processed at 230°C

Viscosity plays a crucial role in determining pigment wetting, pigment agglomeration, pigment flow and dispersion, and ultimately the colour shifts. Particle size and colour difference are reduced, and a higher peak distribution occurs with increasing the temperature and feed rate. In general, the color differences were reduced at the center level of the three processing parameters at (i.e. 255°C, 750 rpm, 25 kg/hr), and colour output was improved

### 1.12. Blending Effects of PC Wt. % on Melt flow Index (MFI)

The actual MFI or viscosity may vary with formulations (i.e. the weight percent of resins, pigments, and additives in the blend). Therefore, the first study to be undertaken was to investigate the effects of blending ratios on the rheological properties of two polycarbonate resins (R1, R2) comprising the selected PC grade. To do this, blends were prepared in which the resin ratios were controlled in steps of 10%. This resulted in step batches were the percentage of each resin change from 100% to 0% in decrements of 10% for each subsequent batch.

It clearly shows a linear correlation with increasing R2 amounts. However, the increase in pressure difference from increasing the amount of R2 is proportional to the viscosity of the blends, where high viscosity corresponds to a low melt flow index. *Figure 14* Indicate that the variation in polycarbonate resin proportions and melt flow indexes significantly affected rheological properties and hence color deviations. As shown in **Figure 15**, an increase for Resin R1, processed in ML (WA&WOA), significantly increased the MFI value, which was inversely correlated with the viscosity values. The blend with additives showed higher MFI values compared to those without additives (WOA) and those processed with SB

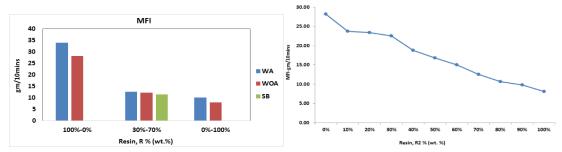


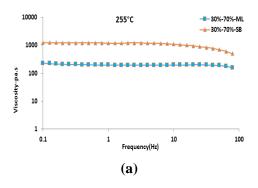
Figure 15: MFI variation versus compounded plastics

Figure 14. Variation in (melt flow index vs. polycarbonate resins)

## 1.13. Viscosity and Dispersion

Figure .a shows the viscosity and particle size distribution PSD measurements for the blends processed in Minilab (ML) and (SB). The blend processed in ML shows a lower viscosity than the one processed in SB. Figure .b shows the ML blend, a larger number of particles for 0-100 % PC (71%) had a small particle size, compared to 30-70 % PC(67%) and for 100-0 % PC(63%) for 100%. The average size of particles is around 1-3 microns, and the Nos of particles is 63-71%. The compounds produced with SB(30%) showed a higher viscosity, which could be due to the smaller particle size distributed in the resin matrix. Therefore, a higher shear heating would not affect the

color mismatch and degradation. The blends don't follow the 'rules of dispersion' and PSD s at varying shear rates



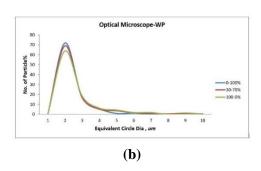


Figure 16 Viscosity (a) and particle size distribution (PSD) (b) for the blends in 0, 30,100 %

# 1.11. Effect of Processing Parameters on Pigment Dispersion

	Para	meter Lev	els				
Parameter	$^{\circ}C$	kg/hr	rpm	$L^*$	a*	<i>b</i> *	dE*
	230	20	750	67.913	1.396	14.763	1.096
Temperature (°C)	255	25	750	68.423	1.473	15.353	0.346
	280	30	750	68.655	1.523	15.443	0.303
	255	20	750	68.886	1.396	15.90	0.44
Feed Rate (kg/hr)	255	25	750	68.423	1.473	15.353	0.346
	255	30	750	50     67.913     1.396     14.763       50     68.423     1.473     15.353       50     68.655     1.523     15.443       50     68.886     1.396     15.90       50     68.423     1.473     15.353       50     68.803     1.51     15.64       00     68.096     1.1     15.32       50     68.423     1.473     15.35	0.320		
	255	20	700	68.096	1.1	15.32	0.633
Screw Speed (rpm)	255	25	750	68.423	1.473	15.35	0.347
	255	30	800	68.116	1.073	15.35	0.623

**Table** shows the grade was compounded with different processing parameters. The recommended optimal processing temperature to minimize the color deviation was 280°C, keeping speed and feed rate fixed at their midpoint levels. A similar procedure was used for both speed and feed rate.

	Para	meter Lev	rels				
Parameter	$^{\circ}C$	kg/hr	rpm	$L^*$	a*	$b^*$	$dE^*$
	230	20	750	67.913	1.396	14.763	1.096
Temperature (°C)	255	25	750	68.423	1.473	15.353	0.346
	280	30	750	68.655	1.523	15.443	0.303
	255	20	750	68.886	1.396	15.90	0.44
Feed Rate (kg/hr)	255	25	750	68.423	1.473	15.353	0.346
	255	30	rpm         L*         a*         b*           750         67.913         1.396         14.763           750         68.423         1.473         15.353           750         68.655         1.523         15.443           750         68.886         1.396         15.90           750         68.423         1.473         15.353           750         68.803         1.51         15.64           700         68.096         1.1         15.32           750         68.423         1.473         15.35           750         68.423         1.473         15.35	0.320			
	255	20	700	68.096	1.1	15.32	0.633
Screw Speed (rpm)	255	25	750	68.423	1.473	15.35	0.347
	255	30	800	68.116	1.073	15.35	0.623

Table8. Effect of processing parameters on color in terms of tristimulus values

### 1.12 Characterization at different Temperatures and thickness

Figurea. Show the case of particles embedded in plastic that were microtomed to the same thickness, e.g. B=70 μm. The thin slices were characterized at three processing parameters. Scattering efficiency increased rapidly as the average particle size decreased to approximately 0.2 microns, and the optimal number of particles was increased at higher processing conditions. The percent of particles of optimal size at the optimum temperature was 35.75933%.

*Figureb*. shows the case of particles embedded in plastic at the same parameter (e.g.  $230^{\circ}$ C) and microtomed to different thicknesses (e.g. 50, 70, and 90 μm). In general, the measured particle size number increased as thickness increased since the probability of seeing a fraction of a particle increased. The average pigment size measured approximately 0.1-0.2 μm.

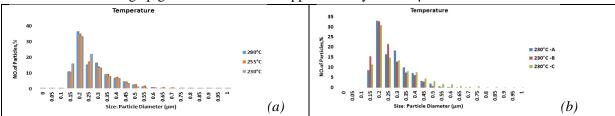


Figure 17. Particle diameter distribution for (a) temperatures 230,255,280°C and (b) for 230°C temperature layers at thickness A (50 μm), B (70 μm), and C (90 μm)

Smaller particles lead to higher suspension viscosities than larger ones. In addition, smaller particle sizes have higher surface area. This phenomenon typically increases the viscosity of the material and maintains the stability as well as material properties during the mixing process. The study will enable us to understand the dependence of processing conditions, de-agglomeration, and polymer blends' consequential colour stability and yield homogenous dispersions. It was also determined that the variation of processing parameters showed a significant effect on shear heat, viscosity, and rheological characterizations. As a result, color output was improved.

# **Conclusions**

IThe temperature and minute formulation of R1/R2 blends exhibit a strong shear thinning and a significant effect in lower viscosity value. The blends do not follow the rule of mixing in terms of viscosity. Formulation and temperature showed an excellent effect on controlling the viscosity and simulate the Carreau viscosity model at various temperatures and master curves. The optimal number of microtome particles was increased at higher temperatures and thicknesses. The average pigment size measured approximately  $0.1\text{-}0.2~\mu\text{m}$ . Microscope observation of morphology indicating improved pigment dispersion and excellent color matching at lower viscosity.

### References

- [1] Syang-Peng, Rwei. Polymer Engineering and Science, 2001
- [2] Wong, A and Lam, Y, J Polym Res 15:11–19, 2008
- [3] Mishra, A, Kumar. Scientific Journal & Industrial Research 67 (2008).
- [4] Dealy, J, and Wissburn, KF. In Melt Rheology and its Role in Plastics Processing: Theory and Applications Van Nostrand Reinhold. New York: Kluwer Academic Publishers, 1990.
- [5] Abdel-Goad, M, and Pötschke,P. "Rheological characterization of melt-processed polycarbonate multiwalled carbon nanotube composites." Non-Newtonian Fluid Mech, pp. 2-6. 2005.
- [6] Mange Sana, N, Chi kuku, RS, and Mainza, AN." Journal of the South African Institute of Mining & Metallurgy Saimm, 108, no. 4 (2008): 237-243.

- [7] Zaeh, M and Hess, R. "Impact of stabilizers on the color of pigmented polymer articles Geneva." Switzerland, Clariant Business Unit Additives Geneva, 2002.
- [8] Sanchez, P. M. Remiro, Nazabal, J. Appl. Polym. Sci., 50, 995-1005 (1993)
- [9] Liang, R. F. and. Gupta, R.K, 2000, Society of Plastic Engineering ANTEC, 2903 2907.
- [10] Lee S, Mather P, Pearson DMMK Journal of applied polymer science 59, 243-250, 1996
- [11] Fatoni, Rois. Product Design of Wheat Straw Polypropylene Composite. Waterloo: University of Waterloo, 2012
- [12] D. Meade, Introduction to colorant selection and Application Technology, in Coloring of Plastics Fundamentals, 2nd ed., R. Charvat, Ed., 2004.
- [13] B. Mulholland, "Effect of Additives on the Color & Appearance of Plastics," ANTEC, Ticona Engineering Polymers, 2007.
- [14] J. Birmingham, "Volatility of Titanium Dioxide Pigments in Polyethylene Film Extrusions," ANTEC, p. 3290–3294, 1995.
- [15] E. Jaffe, C. Campell, S. Hendi and F. Babler, "Rheologically Effective Organic Pigments," J.Coating Technol, p. 47–54, 1994.
- [16] D. Williams and M. Bevis, "The Effects of Recycled Plastics and Compound Additives on the Properties of Injection-Moulded Polypropylene Co-Polymer," J. Mater. Sci, p. 2834–2842, 1980.
- [17] A. Mishra and Kumar, Scientific Journal & Industrial Research, vol. 67, 2008.
- [18] D. J and K. Wissburn, In Melt Rheology and its Role in Plastics Processing: Theory and Applications Van Nostrand Reinhold, New York: Kluwer Academic Publishers, 1990.
- [19] K. H, H. Lee and J. Wook, "Rheological properties of branched polycarbonate prepared by an ultrasound-assisted intensive mixer," Korea-Australia Rheology Journal, vol. 19, no. 1, pp. 1-5, 2007.
- [20] P. R. Hornsby, "Rheology, Compounding and Processing of Filled Thermoplastics," Mineral Fillers in Thermoplastics I, pp. 155-217, 1999.
- [21] A.-G. M and P. Pötschke, "Rheological characterization of melt-processed polycarbonate multiwalled carbon nanotube composites," Non-Newtonian Fluid Mech, pp. 2-6, 2005.
- [22] C. K and W. Cook, "Structure-Property Relationships of Blends of Polycarbonate," Polymer engineering and science, vol. 43, no. 11, pp. 1727-1739, 2003.
- [23] B. V and P. Kitchloo, "Rheological characterization of polycarbonate resins and its applications," Society of Plastics Engineers , pp. 103-106, 2011.
- [24] H. S, K. Tapan and S. Chattopadhyay, "Dynamic and capillary rheology of LDPE-EVA-based thermoplastic elastomer: Effect of silica nanofiller," Polymer Composites, vol. 31, no. 3, p. 377–391, 2010.
- [25] S. E, A. Hossein and Behravesh, "Rheological Investigation of Wood-Polypropylene Composites in Rotational Plate Rheometer," Journal of Polymers and the Environment, pp. 998-1006, 2012.
- [26] A. S, W. Leelapornpisit, A. Derdouri and P. Carreau, "Morphology and Properties of Microinjected Polycarbonate/MWCNT Nanocomposites," in 66th Annual Technical Conference of the Society of Plastics Engineers, 2008.
- [27] M. Khan, "Rheological and mechanical properties of ABS/PC blends," Korea-Australia Rheology Journal, vol. 17, no. 1, pp. 1-7, 2005.
- [28] N. Mangesana, R. Chikuku and A. Mainza, "The effect of particle sizes and solids concentration on the rheology of silica sand-based suspensions," Journal of the South African Institute of Mining & Metallurgy / SAIMM, vol. 108, no. 4, pp. 237-243, 2008.
- [29] M. Zaeh and R. Hess, "Impact of stabilizers on the color of pigmented polymer articles Geneva," Switzerland, Clariant Business Unit Additives Geneva, 2002.

- [30] ASTM Standard D2244, 1993 (2000), "Standard Test Method for Calculation of Color Differences From Instrumentally Measured Color Coordinates," ASTM International, West Conshohocken, PA, www.astm.org
- [31] Olmsted, R., Advanced Color Formulation Technology, in Coloring of Plastics, Fundamentals, 2nd ed. edited by Robert Charvat, 2004.
- [32] Rauwendaal, C., Polymer Mixing, A Self-Study Guide. Hanser Publishers, Munich., 1998.
- [33] Giles, H.F., Wagner, J.R., Mount, E.M., Extrusion: the definitive processing guide and handbook.
- [34] J. Alsadi. Color Mismatch in Compounding Of Plastics: Processing Issues and Rheological Effects (Doctor of Philosophy in Mechanical Engineering, the Faculty of Engineering and Applied Science University of Ontario Institute of Technology). Ontario, Canada (2015)
- [35] J. Alsadi, U. Saeed, S. Ahmad, G. Rizvi and D. Ross, Processing issues of color mismatch: Rheological characterization of polycarbonate blends, Polymer Engineering & Science, Volume 55, Issue 9, September 2015, Pages: 1994–2001, Version of Record online: 8 DEC 2014, DOI: 10.1002/pen.24041
- [36] Jährling, M.a.F., A., *HAAKE Minilab -Compounder and Reactor*. Thermo Scientific, 2005.
- [37] ARES, G. *TA Instruments-Rheometer*. [cited 2015 June 20; Available from: https://www.ndsu.edu/cnse/mcal/equipment/RHMTR.html.
- [38] J. Alsadi Experimental Assessment of Pigment Dispersion in Compounding of Plastics: Rheological Characterization at the Crossover Points, Accepted on January 3, 2021, to Materials today: https://authors.elsevier.com/a/1cbQW7tbNaJzrU, published online Volume 45, Part 8, 2021, Pages 7344-7351, June 11, 2021
- [39] Washburn, E., The Dynamics of Capillary Flow. Phys. Rev. Vol 17(273,1921).
- [40] Xu, J., Hou, Z., and Li, T., *Novel Sample Preparation Method of Polymer Emulsion for SEM Observation*. Microscopy Research and Technique. Vol 70: p. 847–850,2007.
- [41] Klanjsek, G.M., Kunaver, M., Mozeti M. and Hrovat, A. . 148(1): p. 2203 2207, 2004.