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Comparative Investigation on Dispersion And Processing Conditions of Polycarbonate Composites Evaluated By Microscopic Methods

*Jamal. AlSadi[1], Khalid. Zouhri[1.2.3]

[1]*Department of Mechanical Engineering, Higher College of Technology Abu Dhabi, UAE*
[2]*Department of Mechanical Engineering, University of New Haven, 300 Boston Post Road West Haven, CT 06516.*
[3] *Department of Mechanical Engineering-Engineering Mechanics, Michigan Technological University, 1400 Townsend Dr., Houghton, MI 49931, USA*
*Corresponding Author: *Jamal. AlSadi[1]*

ABSTRACT: *The objective of this paper is to evaluate the influence of various processing parameters on the dispersion quality of polycarbonate compound. Experimental data were compared with historical data records. The information can be used to obtain an optimum processing condition at which an extensive range of particles may be dispersed to the composition. The influences of parameters, pigment size distribution, and morphology on the pigment dispersion were also studied. The procedure consisted of dispersing the pigment using a co-rotating twin extruder and then injection mold to produce color chips; these were processed with three different parameters (temp, feed rate, and speed), at three different levels.*

The information on pigment dispersion was obtained using a Digital optical microscope (DOM), Scanning Electron Microscope (SEM), Particle size analyzer (PSA), and Micro Ct Scanner (MCT scanner) -image analysis. The optimum objective of this study is to evaluate pigment dispersion in PCs composition, by understanding the relation between the processing conditions, particle size distribution, and the color difference output. This will help us in understanding additional dispersion issues that have a bearing on color variations for different PCs compound grades.

Keywords: *Dispersion, processing Parameters, Microscopic Methods, Characterization, Color.*

I. INTRODUCTION

Color is necessary for life, as it is in plastic.As plastics continue to enter consumer and industrial markets, color plays an increasingly important role in producing pigments used in the polycarbonate grade; extruded compound needs to have excellent dispersion properties and uniform particle size. Oversized particles can be formed during the processing of the pigment in PC blends. Many pigments are a result of the surface energy of pigments causing attractive hydrodynamic forces between the crystallites. The attractive forces increase with decreasing particle size; small particles tend to combine more easily. Color shift depends on many physical properties of the pigment such as particle size and shape, size, distribution; degree of initial aggregation; surface characteristics; defects and voids in the crystal lattice structure; and additives.

Pigment dispersion affects the color difference and mechanical characteristics of an extruded polycarbonate compound. Consequences of poor dispersal on the PC compound can be the form of mostly visible agglomeration that can cause a high color difference. Other color variation problems that occur are poor gloss, low Chroma, color change and poor opacity/transparency. Mechanical issues that can develop because of a poorly dispersed pigment in polymeric materials. These dispersion problems result in product rejection for grade manufacturers, leading to loss of productivity. Therefore, Poor dispersion generates recycling that requires re-formulation of the grade to get better dispersing pigments in their products. Masterbatch manufacturers are required to provide pigments with improved distribution and to control the processing conditions. Dispersion tests for pigments in plastics have developed over time to display, as well as prevent the possible situation which causes poorly dispersing the pigment. Many types of pigment dispersion test methods developed; they include particle size analysis (PSA), optical microscopy (DOM), scanning electron microscopy (SEM) and micro ct scanner examination.

Processing parameters and shear rate (rheology) are essential for improving dispersion. The processing material at three different conditions and three levels (temp, speed, and feed rate) can have a significant influence on the color and dispersion of a pigments. Several researchers have established an optimization study of particles for opacification of polymer, to find a better size and shape pigment for use in the plastics and paints industry (1-7). The optimum color strength of pigment obtained at a full reduction in the primary particle size. This is due smaller particle is the higher surface area and consequently stronger the color [8, 9] There are two types of reduction in the particle size, shear, and collision. The best quality of the dispersion degree depends on the characteristic of Pigment volume concentration, size, dwell time, rotation speed, energy input, and temperature [10, 11]. The dispersion degree depends on the amount of energy input, i.e. the highest intensity speed and the residence time in the mixing chamber. The pigment particle size distribution (PSD)ranges between 50 and 500 nm to achieve steady dispersion process and to produce a stable colloidal system with color strength, purity, gloss, and highest opacity is obtained in this range [8,9,10,11]. However, an intensity of scattered light by a particle depends on molecular weight, size, and shape [12].

1.1 Image analysis (DOM)

In this paper will review various microscopic evaluation techniques that can be used to examine dispersion to identify the pigment particles, calculate their area, and generate an area distribution. The micrographs of primary particles where they exist in small aggregates held together by van der Waals or London forces. The particles combine to form agglomerations. The size and shape of the particles have a significant effect on color variation as well as other physical properties in plastics. Dispersibility is the degree into which agglomerates separate under shear into primary particles, to achieve maximum color properties and minimum color difference. The particle shape and size are also crucial factors in determining pigment performance, and the color difference (dE), can be considered the ultimate measure or evaluation of dispersibility.

When creating colored compounds or masterbatches from pigments, the goal is to perform a proper dispersion by wetting the pigment surface with the polymer, breaking up agglomerates, and separating pigment particles. The dispersion of pigment particles results in the highest possible color strength. Obtain a proper distribution. Compounders typically should use a twin-screw extruder, additives, and adjusted processing conditions. Therefore Pigment suppliers improve pigment dispersion characteristics by balancing particle shape, size, and final surface treatment.

1.2 Pigment Particle size measurement

Particle size measurement will help to establish terminology used to describe the pigment particles that comprise commercial products. Because pigments are metallic products, they do not exhibit exact and uniform size, but instead, exist in a range of sizes. The scales have been named by Honigman [13] as crystals and crystallites, aggregates, and agglomerates. Particle size is important. The more significant particle size is more reflective and has more sparkle than smaller particles. The tiny particles have produced more sheen and a satin finish. In this study, size measurement of particles is an important science that can only be declared here.

Particle size analysis (PSA), scanning electron microscopy (SEM), digital optical microscopy (DOM), Micro Ct scanner, and image analysis (areas) are all used to study pigment particles, their size, and their distribution. In this study, the particle size distributions of compound grade, under the effect of variable processing parameters as microscopically measured by a DOM or SEM instruments, do not exhibit uniform sizes, that comprises the light scattering are incredibly different.

1.3 Color variation and Particle size distribution

Due to the effects of absorption and scattering with particles, changing a pigment's particle size to larger or smaller changes its hue. As a general rule, hue changes with increasing or decreasing particle size distribution. Narrower particle size distributions are superior for chroma. Since hue shifts with particle size, it is necessary for the pigment manufacturer to control particles size tightly. Pigments are produced in batches and blended, both to meet a color standard and uniformity. Mixing a wide range of particles is a blending of different hues, and therefore results in loss of chroma. The material properties of inorganic pigments are crucial to their application. Scattering and absorption are functions of particle size. Control properties associated with particle size and morphological structure and size distributions help to determine product Chroma.

The other important points, which relates to color change, is the Particle size. The larger pigment sizes are more reflective and have more sparkle than smaller particles. The higher the aspect ratio of a pigment, the higher the hiding power. Characteristically, the narrow particle size distribution shows a cleaner effect, and the broader provides greater hiding power. Particle geometry will also influence the visual effect; this mainly applies to metallic and spherical particles were used. Therefore, the shapes offer different levels of reflectance and opacity [14]

For a proper color appearance, the pigment dispersion is essential.Shearing forces are a requirement for overcoming the surface forces in the pigment particles that hold together the agglomerates. Additional related factors include the chemical composition, structure of the particle, size, and distribution. Other related issues are viscosity, molecular weight distribution, additives in the pigment system, interactions between the pigment and the polymer [15, 16]

A few studies have been carried out by various researchers regarding the effects of processing parameters by the dynamic mixing in a screw extrusion during the compounding of polymers [17, 18]. The dispersion of pigments in viscous polymeric media based on the three distinct stages which can be recognized: wetting, de-agglomeration and distribution, and stabilization [19, 20]. The processing conditions have significant effects on the structure of the grade composites; such as a shear stress, temperature and residence time [21].These, in turn, influence the properties of the processing material. The properties of polycarbonate compound strongly depend on the dispersion state of pigments, and the evaluation of pigment dispersion is therefore essential.

Scanning electron microscopy (SEM) and digital optical microscope (DOM) have been used widely to estimate pigment dispersion. They can provide direct information on pigment size distribution and the effect of processing parameters. It is also an important technique to evaluate the overall dispersion/distribution of pigment particles at the microscopical level, these in turn by using (DOM) and (SEM). Vermogen et al. [22] suggested a statistical image examination method to describe the clay dispersion and the extent of agglomeration. His observation was based on TEM and OM, using histograms of the dispersion parameters of the tactoids of different classes [22, 23]. In the early study, the decrease in viscosity due to the presence of additives and increase in temperature improves the surface tension of polymer blends and thereby increases the wetting properties of pigments. Furthermore, the focus extended to the color output of polycarbonate grade -3, which presents that decreasing the viscosity improves the color output due to better wetting and de-agglomeration of pigments. However, the study will enable us to understand the dependence of wetting, de-agglomeration and the consequential color shifts of polymer blends [24, 25]

In the previous study, the polycarbonate compound behaves like an elastic solid due to a high modulus. Higher storage G' and loss modulus G" occur at increased frequency and lowered temperature.As temperature, shear rate and frequency increase, the melt viscosity is decreased and exhibits a shear thinning. Therefore, the processing conditions have significant effects on dispersions [24, 25, 26, 27].The objective of this study is to investigate the effect of processing parameters with the dispersion of the pigment in the plastic. The study will evaluate how the particle size distribution and analyzes their relationship within processing parameters to obtain accurate results, a various dispersion and morphological techniques were used to assess the effects of optimum processing parameters.The objective was examined by the existence of the agglomeration, the shape, and size of the particles traveling through the flow.

The main focus was extended to establish a quantitative methodology which has been developed by combination of PSA, SEM, μCT Scanner, and DOM observations to describe pigment dispersion in polycarbonate composites (G3) This method improves the evaluation of rheological properties, dispersion of pigments and in reducing the color differences, required to process the material with reduced wastage.

II. EXPERIMENTAL

2.1 Materials

The material used was a blend of two different Lexan polycarbonates, having different a Melt Flow Index; (MFI) for R1 was 25g/10min and that for R2 was 6.5g/10min. Also, it contained four different color pigments (black, white, red, yellow) and three additives (a stabilizer, light stabilizer and weather resistant) –See Table-1

2.2 Procedure

The additives were mixed with the resins at a 100:0.86 ratio and were batch blended by a super floater in ratio per weight to assure higher consistency. Extrusion was performed on co-rotating intermeshing-TSE to ensure a uniform melt mixing. Upon exiting the die, the extrudate was quenched in cold water, dried using air and then converted into pellets using a pelletizer. These pellets molded by using injection molding into three rectangular color chips (3x2x0.1") size. These were processed at 85 ton, at about 1000 PSI, and at 280 °C. The specimens then dried in the lab at room temperature and measured at three different spots. The color difference (dE*) tristimulus values of these coupons were measured and characterized by the spectrophotometer, using the CIELAB Color Space system. It provides three tristimulus color readings L*, a*, and b* values [28]. Three parameters were varied individually to three different levels. While keeping the other parameters fixed, one setting changed; the others remained fixed at Federate 25kg/hr, screw speed 750rpm, and temperature of 255°C, while studying their effects on color shifts.

For the microscopy dispersion test, a tin sheets were prepared either by molded color chips, hot presses or-or microtome into thin slices, for examination under the microscope. Color chips were used to get the images

into DOM at magnification (1000X-5000X). Itillustrates the characterization of the pigment size distribution for Temp, screw speed and feed rates, at a fixed temperature of -255 °C, screw, the speed of – 750 rpm and feed rate of -25kg/hr. (SEM) Joel 5500 is a high-resolution scan system. The samples covered with a thin conductive colloidal graphite coater used to characterize the compounding material, having a magnification of 3000X-1000X, and SEM-SM-600 was used to characterize pigment raw material without coating. The acceleration voltage was 20 kV, having a magnification of 3000X.

The measurements for the center point treatment chip were carried out by using X-ray micro CT scanner, with processing at a speed of 750 rpm, and at fixed Temp 255C and FR 25kg/hr. The data collected at 32kV and 187μA; the image characterized on a high-resolution X-ray detector; 10 megapixels (4000 x 2300), 12 bit digital cooled CCD camera. The images of the micrograph show a consistent distribution, which can be of uniform shape and have spherical particles of narrow peak size distribution.

2.3 Set up Processing Parameters (General Trends)

Producing a specific color plastic using extrusion compounding requires proper operating conditions. Changes in the operating conditions will affect color. In the present work, the operating conditions were varied in a controlled manner to study their effects on color. Three parameters including temperature, speed, and feed rate were varied individually to three different levels while keeping all other parameters fixed (GT). Strong interactions were observed between the operating conditions and color as shown in Table2

The experiments were set up as follows: The recommended processing temperatures were =230°C, 255°C and 280°C with a speed of (750 rpm) and flow rate of (25 kg/hr) fixed. A similar procedure used for both the speed and flow rate. The following were recommended: processing speeds of 700 rpm, 750 rpm, and 800 rpm with a flow rate (25 kg/hr) and temperature (255°C) fixed. Lastly, the recommended flow rate was 20 kg/hr, 25kg/hr, and 30 kg/hr, with a constant speed (750 rpm) and temperature (255°C). [29-34].

III. RESULT AND DISCUSSIONS

The extrudate pellets of the compounded material were processed at different parameters, e.g., temperature, feed rate, and screw speed; then these pellets were compressed by the injection molder in rectangular shape chips. Each parameter produced three samples which were measured for their color output properties and the particle size distributions.

The most common method used to determine color approval or rejection is the Delta E* or "dE*." However, the CIELAB system uses the "dE as a reference amount for setting tolerances. Consequently, there are many quality sectors which can accept this value without considering the other aspects of color such as lightness, chromaticity, and hue. The methods of color evaluation is calculated by

$DE^* = [(DL^*)^2+(Da^*)^2+(Db^*)^2]^{1/2}$………………………….. (1)

The three aspects of the tristimulus color sample are (lightness/darkness, red /green shade, and yellow /blue shade). Ultimately, the CIELAB system compares a sample to a standard and makes a numerical determination based on the perceived color difference. There are variance color differences (dL*, da*, db*, dC*, dE*): where dL* is in negative for darker, da* is in positive for more red, db* is in negative for bluer, dC* is chromaticity, DE or dE* is the total color difference ,the lower color difference is at the center point, the negative dC* and db* values indicate the chromaticity of bluer hue originating from incomplete dispersion .

3.1 Effect of temperature

3.1.1 Effect of temperature on color values at 750 rpm and 25 kg/hr

Figure 1 demonstrates the results of a the color difference (dE*) at variant temperature 230ºC, 255ºC, and 280ºC. It has shown a reduced value of color difference when increasing the temperature, at a fixed feed rate (25kg/hr) and speed (750rpm). However, the best color difference value (dE*) is at higher temperatures (255 ºC, 280°C, dE*=0.3). The target values (Tg) are CIE (L*, a*, b*) which equal (68.5, 1.43, 15.7). Color differences are (dL*,da*, db*, dE*, dC*) .The data represented at 230 ºC is the higher color difference dE*. The negative color values of dC*, da*, dl* and db* indicate that the green-blue hue originates from an incomplete dispersion of the pigments. Pigment particles in this sample show incomplete dispersion and higher agglomerations than the other two samples.

The total color difference dE* decreases at 255°C. The negative of color values of dC*, dl* and db* indicates that the green-blue hue originates from the incomplete dispersion of the pigments. Pigment particles in this sample are shown to have a better dispersion and lower agglomerations than the 230°C sample.

At temp 280°C, dL* is positive for a lighter color, da* is positive for more red, db* is negative for bluer. DE or dE* is the total color difference. Note that dC* is always negative at three temps, which is an indication for low chromaticity. The negative of dC* and db* color values indicate that the chromaticity of the bluer hue originates from an incomplete dispersion. In general, the total color difference variation is reduced when the temperature increases. According to these results, 280 º C samples had a better-dispersed pigment than 255°C,

which in turn was better dispersed than 230 ºC. Again, the reduction in color measurement is in good agreement with the image of less agglomeration or lower particle size and with a higher peak of particle size distribution as shown in Table3 and Figure.2.

3.1.2 Evaluate Dispersion at Variant Temperatures

Figure 2 illustrates the pigment dispersion with an increase in temperature. The peak of the distribution becomes narrow; 55.3% of the particles lie at approximately 0.84 microns at 280 ºC, in comparison 52.4 % of particles lie at 0.79 at 255°C, and 48.9% of particles lie at 0.78 um at 230°C. Manufacturing the PCs grade samples at temperatures of 230, 255, and 280 °C at fixed 750 rpm, and 25 kg/hr. The morphology was tested by scanning electron microscopy (SEM), Using a Joel 5500 LV at a 20 kV accelerating voltage, preceding to the SEM analyses.The samples were vacuumed by SC7620 Sputter Coater using PELCO conductive colloidal graphite coater. The results were shown in Figure 12 (a, b, C). The degree of dispersion is achieved at higher extrusion processing temperatures, such as 255°C, which seems to be somewhat better than 230 °C. The micrographs in Fig. 12(a-c) show that at higher temperatures lesser agglomerates are evident at 280C than at 230C. A higher temperature has good pigment dispersion with a lesser amount of agglomerates observed

3.2 Effect of Feed rate

3.2.1 Effect of Feed rate on color values at 750 rpm and 255 °C

The color values of dL* and db* show minimum value at the central points, but da* is increased with an increasing feed rate at a fixed speed 750 rpm and Temp 255 °C. The feed rate increases from 20 kg/hr to 30kg/hr, while the temp 255°C and speed 750 rpm are fixed. Color differences are (dL*, da*, db*, dE*, dC*,): the data represented at 20kg/hr shows the higher color difference dE*, db*, dl* and dC*. The higher value in dC* is due to the positive color values of dl* and db*, which indicate that the light-yellow color value is positive. A pigment particle in this sample shows incomplete dispersion and higher agglomerations than the other two samples. At 25kg/hr is shown a positive in da* of the color value and positive in da* and dl* at 30kg/hr. It indicates a slight reduction in the color difference. See figure 3.

3.2.2 Evaluate Dispersion at variant Feed rates.

In general, the total color differences decrease when the feed rate is increased. According to these results, the 30kg/hr sample had a better-dispersed pigment than the 25kg/hr sample, which in turn was better dispersed than 20kg/hr. Figure 4 shows the pigment dispersion with an increase in the feed rate. The peak of the distribution becomes narrow, e.g., the feed rate of 30kg/hr, at peak 53.8% and the particles sizes lie at approximately 0.8 microns, in comparison to 52.4 % of particles lie at 0.8 at 25kg/hr and 47.8% of particles lie at 0.97 um at 20kg/hr. The particle size distribution graphs for the samples used in this study are in good correlation with the feed rates. Again, differences in color measurement are in good agreement with the image and particle size distribution analysis results. See table 4.

3. 3 Effect of speed

3.3.1. Effect of speed on color values at 255 °C, 25kg/hr

The speed increases from 700 rpm to 800 rpm, while the feed rate at 25Kg/hr and Temp at 255 °C rpm are fixed. The color values of dL* and db* are lower than the target values at three levels; except the da* color difference value is slightly positive at 750rpm (See Fig.5). Fundamentally, this is an indication that the red inorganic pigment was better dispersed in the center point than in 700 and 800 rpm samples. Color differences are (dL*, da*, db.*, dE*, dC*,): dL* is in negative for darker, da* is in positive for more red, and db***** is in negative for bluer.DE or dE* is the total color difference. The lower color difference is at 750 rpm; dC* is negative for chromaticity. The negative dC* and db* values indicate the chromaticity of a bluer hue originate from the incomplete dispersion. The transparent polycarbonate plastics do not scatter light; consequently, to achieve a certain opacity level, white pigment is added to create scattering. Therefore, it affects the apparent color strength [35, 36, 37]. The negative of chromaticity may be due to the low Chroma in the compounded plastic grade of the translucent polycarbonate resins.

At low speed, e.g., 700 rpm, the extruded material has shown a lower shear heat or lower shear rate. Agglomeration, can in principle, take place in zones with low shear.

By increasing the speed to 750 rpm, deagglomeration occurs in zones of high shear. Raising the screw speed higher than 750 rpm, the color differences are decreased, because of the relatively low overall shear forces through the entire mixing zones, which separate the pigment particles immediately from the composition.

Also, directly after raising the speed to 800rpm, extruded resin material was increased with shear heat transition, and the PC composites were confined to higher shear rates at the higher speed of 800 rpm. The difference in the state of the pigment dispersion was also proven by measurements of the color differences between the 700rpm,

750rpm, and 800rpm.The most commonly used system in plastics is CIE 1976 (L* a* b*) color space or CIELAB, which allows the specification of color regarding a three-dimensional space.

The data are represented in Fig. 5. The total color difference dE* increases at 700 and 800, but decreases at the center point 750rpm. The negative dC*, da*, and db* values indicate the green-blue hue, originating from the incomplete dispersion of the pigments. Pigment particles in sample 750rpm were better dispersed than in the other two samples. Again, differences in color measurement are in good agreement with the image and particle size distribution analysis results.

3.3.2 Evaluate Dispersion at variant speed

Extrusion was carried out at a screw speed of 700, at the center point (750rpm), and 800 rpm, for which a screw speed of 750rpm was used and found a significantly lower color difference. The results of the particle size and dispersion analyses are shown in table 5. The dispersions were characterized with DOM analysis in combination with the image (SEM) and MCT. Scanner. With the increase of screw speed, the peak of the distribution becomes narrow, except at the lower and higher speeds, the average size of the particle is, 0.96, 0.79, and 0.88um for 700,750,800rpm respectively. At 750rpm higher peaks are shown in Figure .6 in comparison to 42.8% at 700rpm and 44.98% at 800rpm. The Pigment size distribution (PSD) graphs for the samples used are in good correlation with the speeds in the process. See table 5

3.4 Dispersion and Morphology of Compounded Grade:

Dispersion is characterized by:

3.4.1 S3500 Microtrac- Particle Size Analyzer (PSA)

The particle size analyzer (PSA 3500) is a laser diffraction particle size analyzer for both wet and dry dispersions with minimum effort, which delivers rapid, precise particle size distributions measuring over the nanometer to millimeter particle size ranges. To characterize the diameter of a pigment, Table 6 shows the variation of the particle as analyzed and also used in a wet state in the wet test; the pigment was reduced to the lower value. When in the water it has a non-ionic surfactant such as Triton X100. Then the particle analyzer employs the fact of scattered light from multiple laser beams projected through a stream of particles. Consequently, the amount and direction of the light scattered by the particles are measured by an optical detector array, and then analyzed by the Microtrac Software "Micro-flex."

Table 6 shows that the sizes are in diameter for the four pigments. The sizes decrease as the time or ultrasonic power increases. It illustrates the average dia of four "as raw pigments" with the variation in ultrasound time. It can be seen that average particle dia decreases to a value of 0.2 μm ±0.1 at 150 seconds, except black pigments having dia of 4μm. It means that the increase in ultrasound time increases the dwell time and induces more energy to de-agglomerate the particles. The results of Table 6 are quite comparable with the results of primary particles of pigments, presented in Figure 7.

This process will improve the dispersion, reduce the size of pigment particles and agglomerates by increasing ultrasonic power and ultrasound time. This is basically due to adhesion forces through the process of surface wetting, which enables agglomerated particles to be dispersed with quite little energy input. Shown, the dispersion operation determines the amount of power and duration of ultrasound required to disperse the sample to its primary particle size. Using three red lasers has the effect of increasing the range of measurement, resulting in flexibility for analyzing a large number of samples. Also, the benefits of using Microtrac S3500 are more advantageous by permitting the users to measure complex particles with precision. This is difficult to measure with other particle analyzers [38].

3.4.2 Digital optical microscope (DOM)

Digital optical microscopy (DOM) is used to characterize the PCs -grade samples. It is prepared in the following processing parameters of the extruder, at temperatures of 230, 255, 280 °C, and at fixed other parameters of temperature (255°C), feed rate (25kg/hr), and speed (750 rpm). Magnification is 1000X. The evaluation was made of the degree of pigment dispersion in color chip samples; the aim of the present test is to quantify the changes in the state of the pigment dispersion caused by the variation of processing parameters. The color samples were injection molded into three rectangular color- chips (3x2x0.1") size for a spectral color measurement. The CIE(L*,a*,b*) color values were measured with a spectrophotometer.

The figures (8-10) showed the pigment dispersion with the increase of screw temperature, feed rates, and screw speeds; the peak of the distribution becomes narrow at approximately higher processing parameters as were shown in figure (1-6).

The significance of this approach is the proportional relations between the Number of particle % and color differences (dE*) at variable temperatures, screw speeds and feed rates as were shown in figure (1-6).

As judged from morphological micrographs, agglomeration of the pigment clearly occurred at a lower processing temperature parameter, e.g., 230 °C; therefore, at higher processing temperatures e.g. 255 and 280°C are employed to reduce the color differences at a constant rate at (0.3) and to increase pigment wetting, reduce or prevent pigment agglomeration, improve the pigment dispersion, and reduce the color shifts.
The pigment distribution and morphological micrographs, agglomeration of pigments are shown in different modes:

Figure 8 illustrates DOM-Uniform particle distribution, tested for (a) chip sample at 750 rpm(center point) and lower color difference (dE*) and magnification 1000X, (b) microtome sample at 280 ºC(Lower dE*) and magnification 2000X. Figure 9 illustrates DOM-Agglomerations Agglomerations at different processing conditions for (a) at 700rpm (b)230°C, (C) 800 rpm and (d) 30 kg/hr ,(samples were measured by 50micron chip , cut by microtome, magnification 2000X. Figure10: Micrograph of R1-30% agglomerates were exist at low processing parameters at: (1) 230 ^{o}f (4000X): (2) 20kg/hr (5000X): (3) (a) 700rpm (5000X) and (4) 800rpm (5000X)

3.4.3. Scanning Electron Microscope (SEM) -JSM600

The morphology was examined by scanning electron microscopy (SEM). Fig.7, A (SEM) -JSM-600 was used to characterize the raw pigments and the color compounded grade to verify the presence of agglomerates. Figure 7 depicts the presence of (a) agglomerates in black, white, yellow and red pigments. The SEM and DOM micrographs show that a relatively major agglomerated pigment could be observed at lower processing parameters more than at higher processing. The degree of dispersion is obtained at higher extrusion processing parameters, e.g., temperatures, feed rate, and speed. The same results have been shown in the graphs (1-6)

3.4.4- Scanning Electron Microscopes SEM-Joel 5500

SEM-Examined the morphology by using a Joel SEM at a 20 kV accelerating voltage. Figures (11, 12, 13,) depicts the presence of agglomerates and primary particles in compounded grade. Three parameters were varied individually to three different levels. In the SEM micrographs, relatively major agglomerated pigment could be observed at lower processing parameters more than the higher processing.
Figure 11, 12, and 13 illustrate: SEM micrograph of the compound grade at (a) three parameters (Temp, Screw speed, and Feed rate) (b) three levels (Low, medium and high).Agglomerations were higher and noticed at lower processing parameters.

3.4.5 3D X-ray Micro CT Scanner -Sky Scan 1172

The sample that has been obtained from (30/70%) color-Grade.3 Processed at 25 kg/hrs. 255 C° and 750rpm (Center level). The grade compounds were molded at 282 C°, examined the morphology by X-ray micro tomography measurements. The datasets collected for the tomography were 360° angular range with a step size of 0.4° between the images , the image was characterized on a high-resolution 3D, X-ray detector; 10 mega pixel (4000 x 2300) pixels) 12 bit digital cooled CCD camera; it shows a consistent distribution and a spherical pigment shape.

Figure 14 illustrates: compounded PC grade (3) , process at 750rpm, 255 ^{o}f and 25kg/hr : (a) agglomeration's (b) particle shape (C) pigment distribution, using a µCT scanner .Agglomerations were lower at this processing parameters and the images were very clear by using micro Ct scanner. The images of the micrograph show a consistent distribution.

IV. SUMMARY AND CONCLUSION

The processing parameters of the PC molded samples were investigated using a co-rotating twin extruder at a variant temperatures of 230,255,280 ºC, variant feed rates of 20,25,30kh/hr and at variant speeds of 700,750,800 rpm The results showed that: The interaction relationship between tristimulus color values (dL*, da*, db*, dE*) and processing parameters showed that the minimum color difference throughout the experiment was 68.42for L*, 1.47 for a*, 15.35 for b*, and 0.34 for dE* respectively. According to these results, the 280 ^{o}C sample had a better-dispersed pigment than at 255°C, which in turn, was better dispersed than at 230°C; again the 30kg/hr sample had better-dispersed pigment than the 25kg/hr, which in turn, was better than the 20kg/hr.

During the processing parameter of speed, the sample at low speed, e.g., 700 rpm, the extruded material has shown alow shear heat or a low shear rate. Agglomeration can in principle, take place in zones with low shear. By increasing the speed to 750 rpm, deagglomeration occurs in zones of high shear. By raising the screw speed beyond 750 rpm, the color differences start to decrease up to 775rpm. This is because of the relatively low overall shear forces through the entire mixing zones.

The color difference values (dE*) decrease significantly with the increase of the temperature and feed rates. Therefore,it shows a consistent higher peak distribution. Equally, increasing the speed to a medium range (the i.e., center point at 750 rpm) shows a significant minimum color difference; therefore,it shows a consistent higher peak distribution and a spherical pigment shape. At a higher screw speed of 800rpm, these processing parameters can generate large shear forces and amounts of a frictional heat state that may arise and affect the heat stability of pigment and damage other components of the polymer matrix. The high velocities raise the material temperature; the failure of appearance, physical properties, or degradation are possible. Currently, processing parameters are the most common pigment dispersion process used in the manufacture of color concentrates. Selecting the best color concentrate for your specific polymer application requires careful consideration of pigment particle size and surface area. This spectacle is employed to increase pigment wetting, reduce or prevent pigment agglomeration, improve the pigment flow and dispersion, and ultimately reduce the color shifts; particle size and color difference are reduced, and a higher peak distribution occurs when increasing the temperature and feed rate; in the case of the speed, however, the color is reduced at center point. The color differences are cut at the center point and high processing conditions and ultimately improve color output.

At higher processing parameters, the samples (Temp) were found to be a very lower color difference and having a bluer (-db*) and more red color difference (+ da*) and lighter color + dL* with weaker Chroma (-dC*) compared to the Speed sample. The (speed) chips was found to be higher color difference and negative (-dl*) compared to both positive dl* for (temp and feed rate) samples

4.1 Conclusions

In general, the center point at (255 ºC, 25kg/hr, and 750rpm) sample was evidence that the lowest color difference for the three samples was prepared with a higher consistency, which is consistent with the historical data. At lower processing parameters, this example shows incomplete dispersion and higher agglomerations. Small particle size such as red pigments type often possess high surface areas and therefore exhibit higher degrees of (' Redness '), than larger particle size pigments. Within a given small particle size range, resulting in higher melt viscosities and slightly improved dispersibility. The overall particle size profiles of the high processing samples were smaller in comparison to the lower processing application, and it was launched as a successful commercial product for grade compound applications.

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List Of Tables

Table 1: Composition of compounding material for polycarbonate -Grade (3)

S.No	Constituents	Material Name	PPH	Unit
1	PC1	Bisphenol A (BPA)	30	gm
2	PC2	Bisphenol A (BPA)	70	gm
3	F 1	Weather resistant (L)	0.035	ml
4	F 2	Stabilizer (Liquid)	0.065	ml
5	F 3	Light Stabilizer	0.2	gm
6	White	White Pigment	0.278	gm
7	Black	Black Pigment	0.036	gm
8	Red	Red Pigment	0.175	gm
9	Yellow	Yellow Pigment	0.071	gm

Table 2 –Average Operating Conditions – Temperature, screw speed and Feed rates Variation (General Trends (GT))

Speed RPM	BZ1 (°C)	BZ2 (°C)	BZ3 (°C)	BZ4 (°C)	BZ5 (°C)	BZ6 (°C)	BZ7 (°C)	BZ8 (°C)	BZ9 (°C)	DZ1 (°C)	F. Rate (kg/hr)
750	70	195	230	230	230	230	230	230	230	230	25
750	70	195	255	255	255	255	255	255	255	255	25
750	70	195	280	280	280	280	280	280	280	280	25
700	70	195	255	255	255	255	255	255	255	255	25
750	70	195	255	255	255	255	255	255	255	255	25
800	70	195	255	255	255	255	255	255	255	255	25
750	70	195	255	255	255	255	255	255	255	255	20
750	70	195	255	255	255	255	255	255	255	255	25
750	70	195	255	255	255	255	255	255	255	255	30

Table 3 Particle size distribution related to temperatures and color variation

Temps(C)	dE*	No. of Particle %	Avg. Pigment size
230.00	1.10	48.90	0.84
255.00	0.35	52.40	0.79
280.00	0.30	55.30	0.78

Table 4 Particle size distributions related with feed rate and color variation

Feed rate(kg/hr)	dE*	No. of Particle %	Avg. Pigment size
FR-20	0.44	47.8	0.97
FR-25	0.34	52.4	0.83
FR-30	0.32	53.8	0.84

Table 5. Particle size distribution related to screw speed and color variation

Speed(rpm)	dE*	No. of Particle %	Avg. Pigment size
N-700	0.633	42.82	0.96
N-750	0.346	52.43	0.79
N-800	0.623	44.98	0.88

Table 6: Average particle size for four pigments using wet analysis with varying ultrasound time at 30W

Black		White		Yellow		Red	
Time/sec	Dia/mic	Time/sec	Dia/mic	Time/sec	Dia/mic	Time/sec	Dia/mic
0	40	0	8.1	0	3.2	0	4.2
30	20	30	5.2	30	2.9	30	2.8
60	10	60	2.2	60	1.7	60	1.8
90	6.2	90	1.1	90	1.2	90	1.1
120	4.2	120	0.8	120	0.7	120	0.6
150	2.2	150	0.4	150	0.3	150	0.2

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Figure 14: PC compounded grade-3, Process at 750rpm, 255 ^{o}C and 25kg/hr.: (a) Agglomeration's (b) particle shape (C) Pigment distribution, using a µCT Scanner

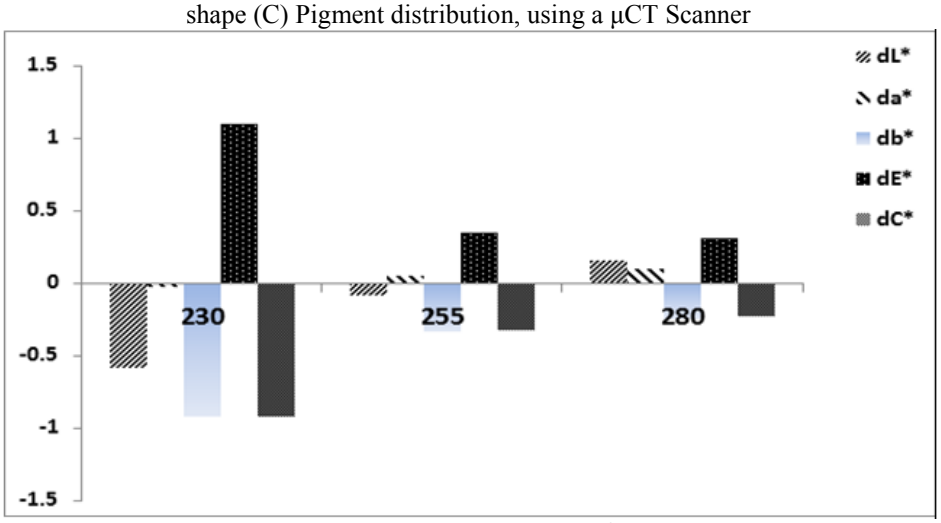


Figure 1 .CIELAB Colour differences for 230, 255 and 280 ^{o}C temperature parameter samples.

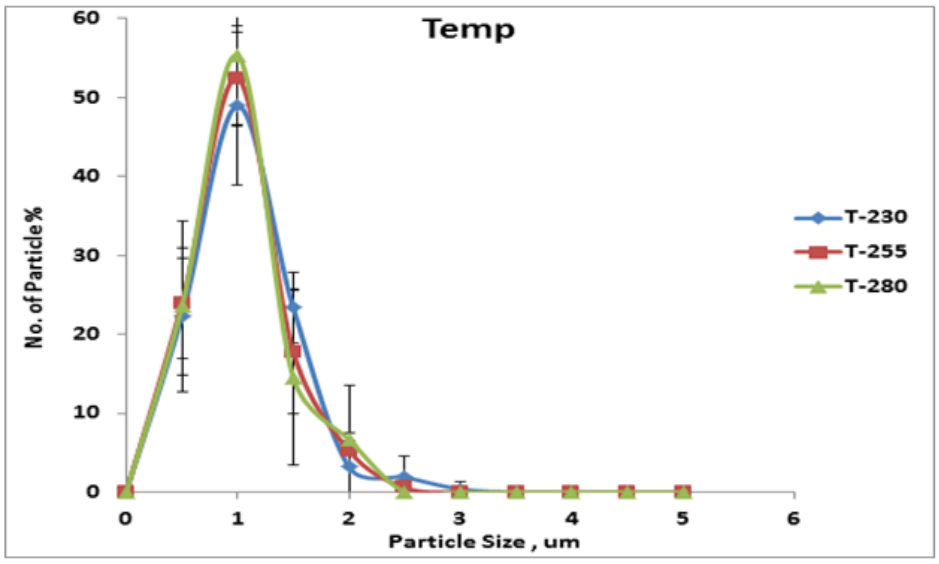


Figure. 2. Pigment size distribution for Temperature parameters, at fixed 25 kg/hr and 750rpm

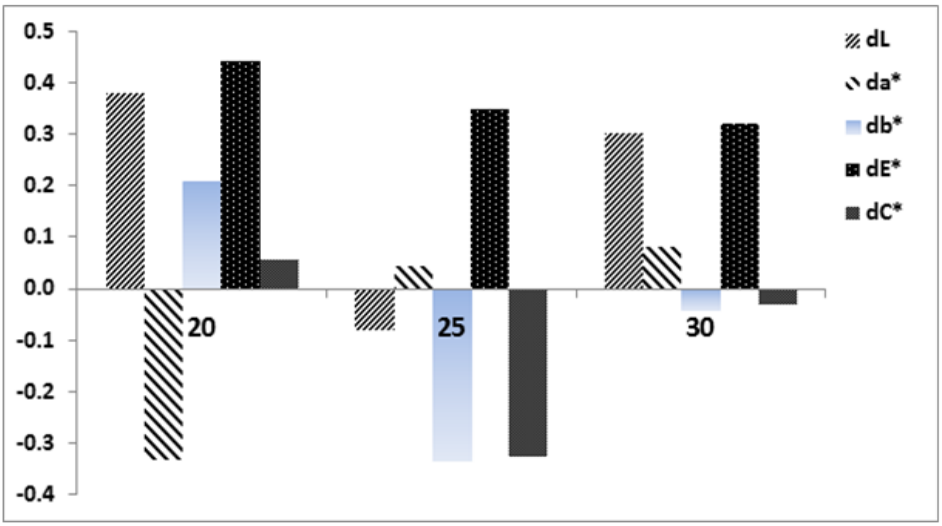


Fig. 3. CIELAB Colour differences for 20, 25 and 30 kg/hr. Feed rate parameter samples

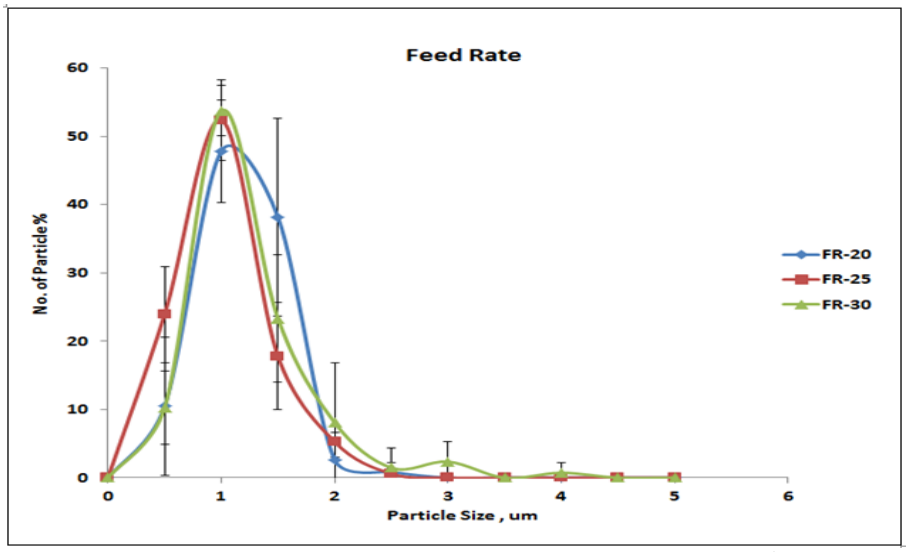


Figure. 4. Pigment size distribution for Feed rate parameters, at fixed 255 ^{o}C and 750rpm

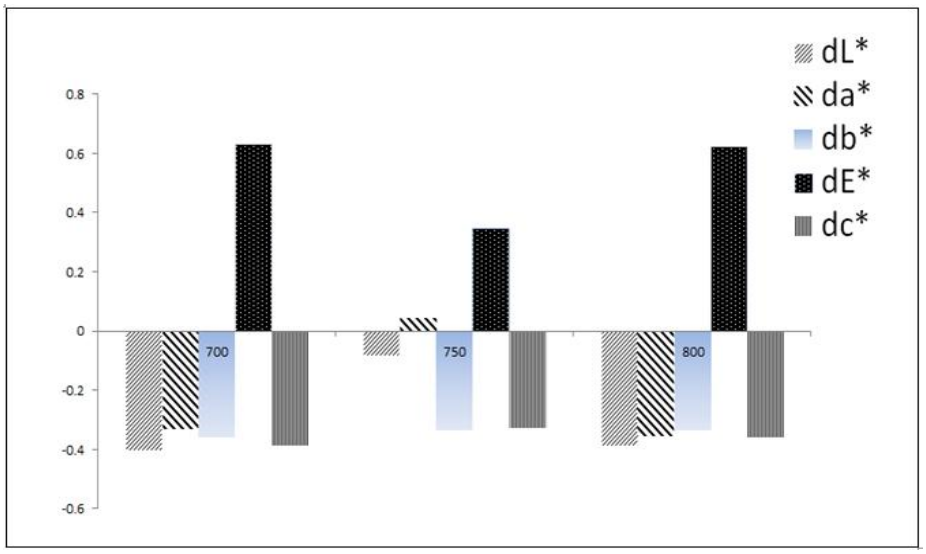


Figure 5. Cielab colour differences for 700, 750 and 800 rpm speed parameter samples

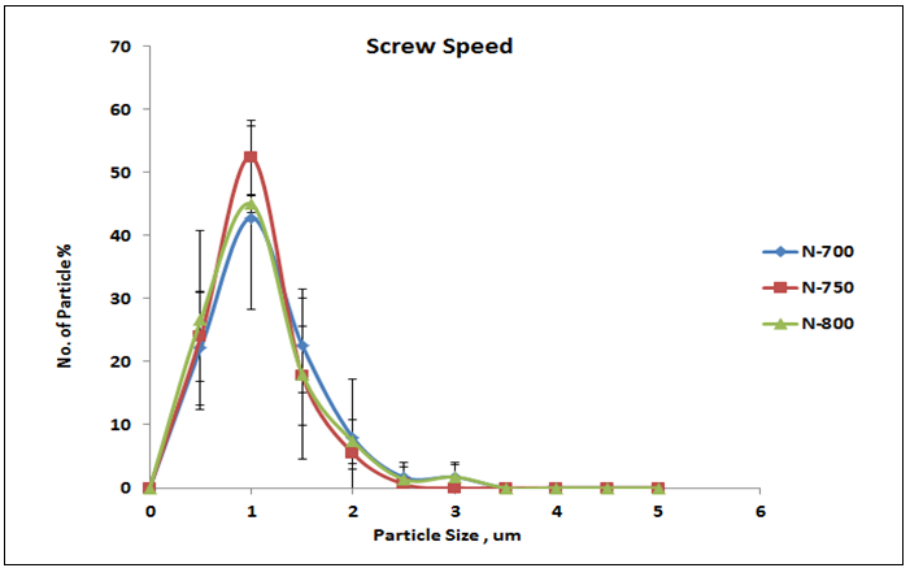


Figure.6. Pigment size distribution for Screw Speed parameters, at fixed 255 ^{o}C and 25kg/hr

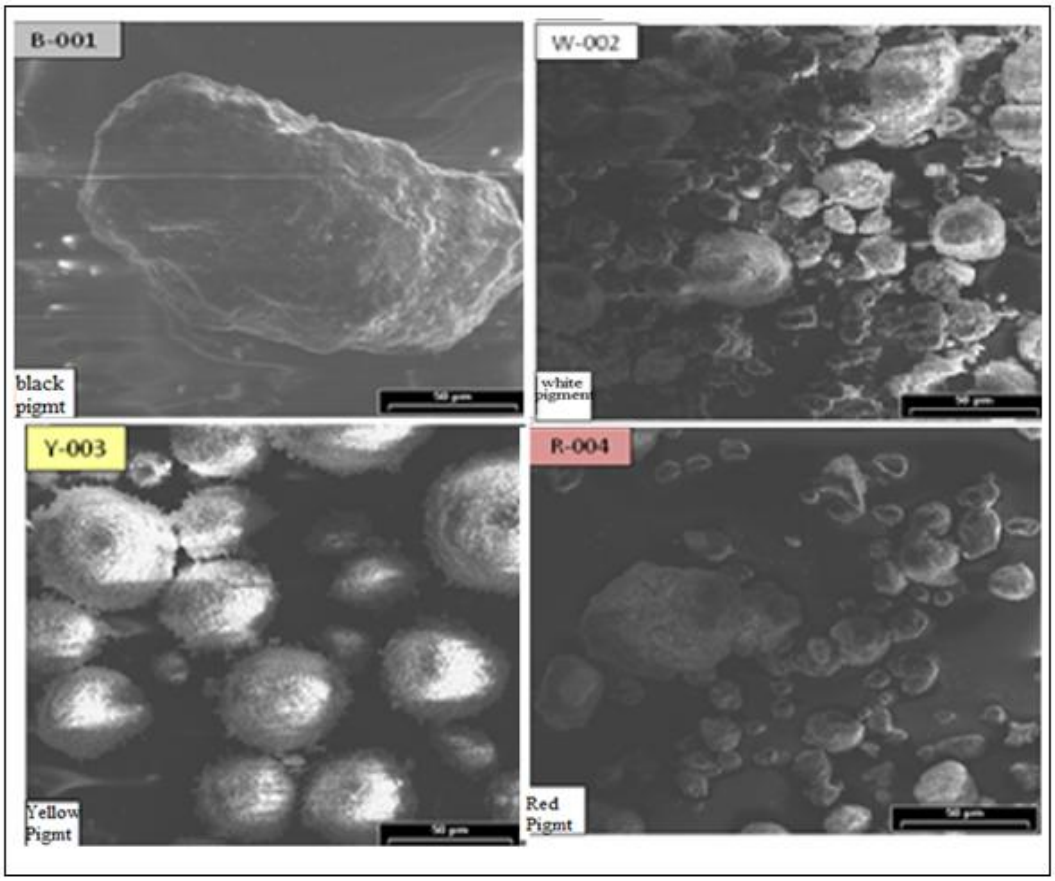


Figure 7: SEM micrograph of pigments (1) white (2) black (3) Yellow (4) Red,3000X (Agglomerations, Raw Pigments).

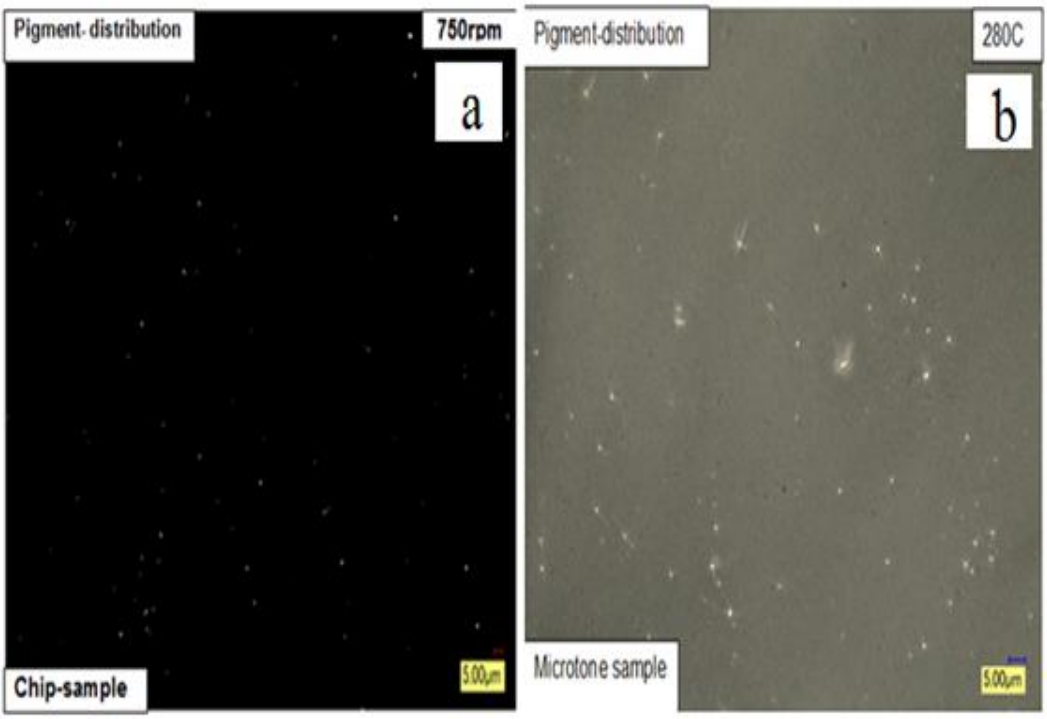


Figure 8: DOM-Particle distribution, tested for (a) chip sample at 750 rpm (Centre point) and magnification 1000X, (b) microtone sample at 280^{o}C and magnification 2000X

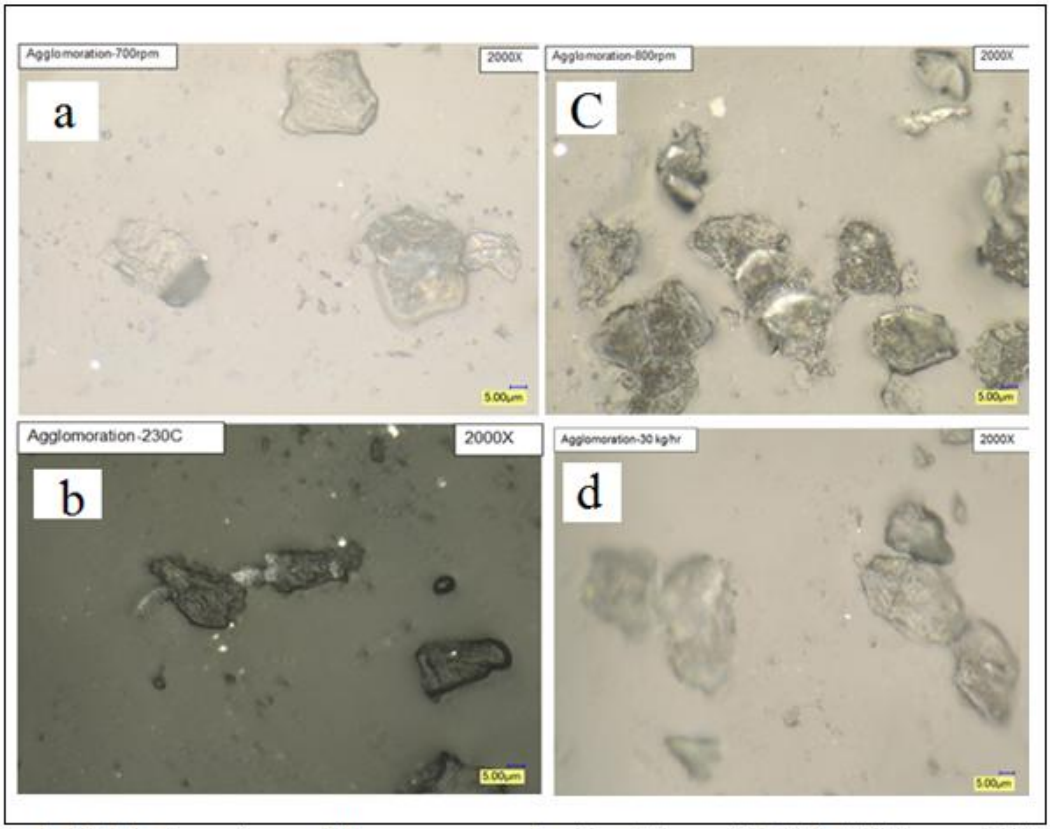
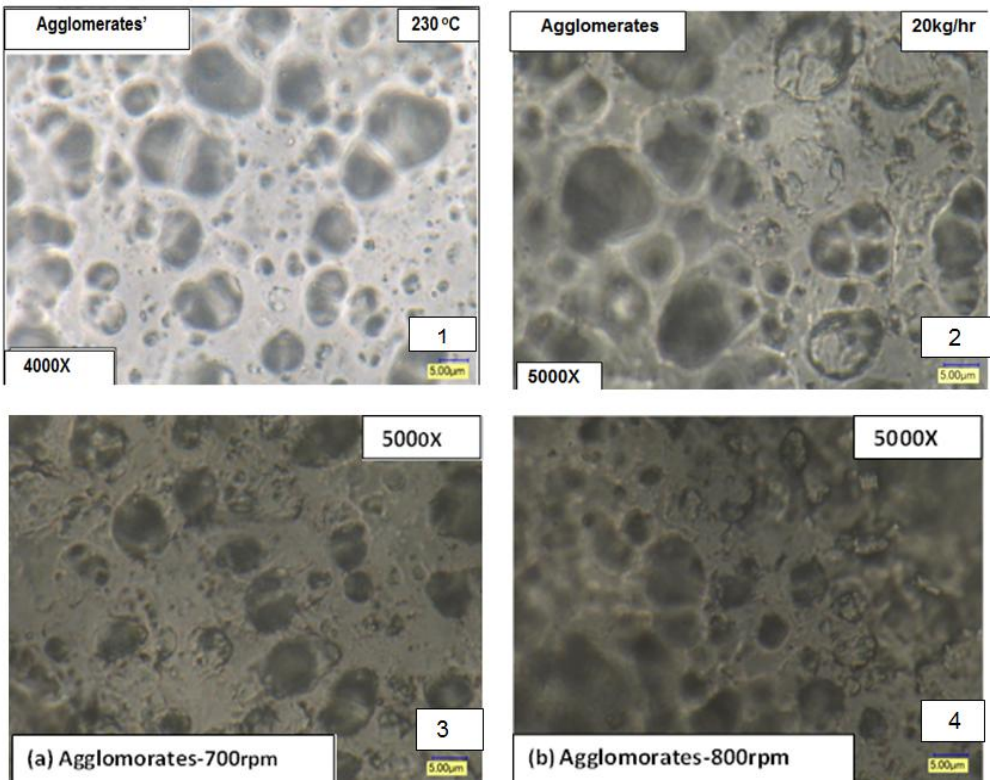


Figure 9: DOM-Agglomerations at different parameters for (a) at 700,rpm (b)230 ^{o}C ,(C) 800 rpm ,and (d) 30kg/hr samples , magnification at 2000X ,(b) measured by 50micron chip ,cut by microtone magnification 2000X,

Figure10: Micrograph of R1-30% agglomerates' (at low processing parameters) at: (1) 230 ^{o}C (4000X): (2) 20kg/hr (5000X): (3) (a) 700rpm (5000X) and (4) 800rpm (5000X)



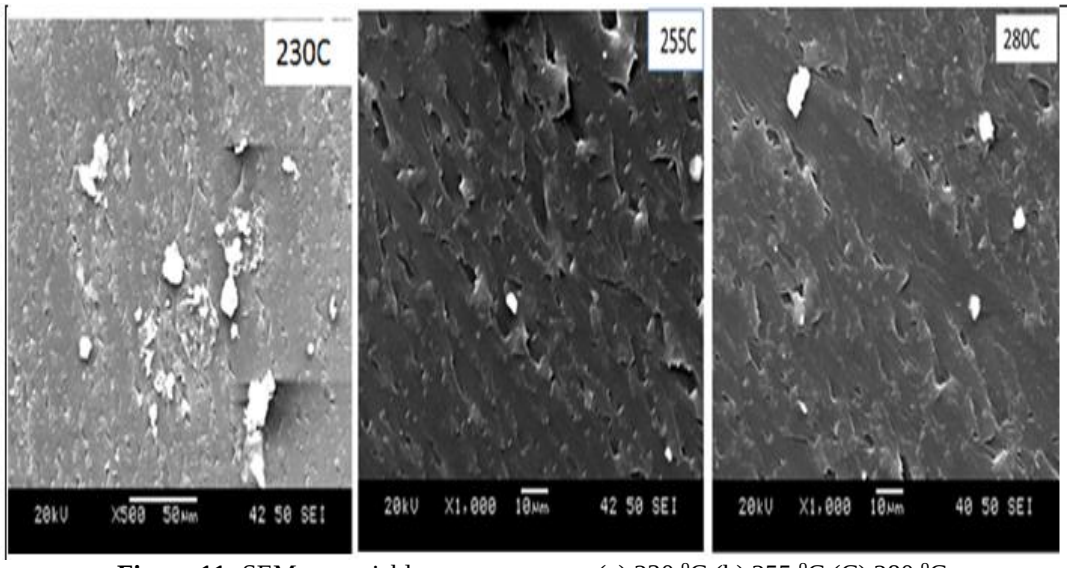


Figure 11: SEM- at variable temperature at: (a) 230 ^{o}C (b) 255 ^{o}C (C) 280 ^{o}C

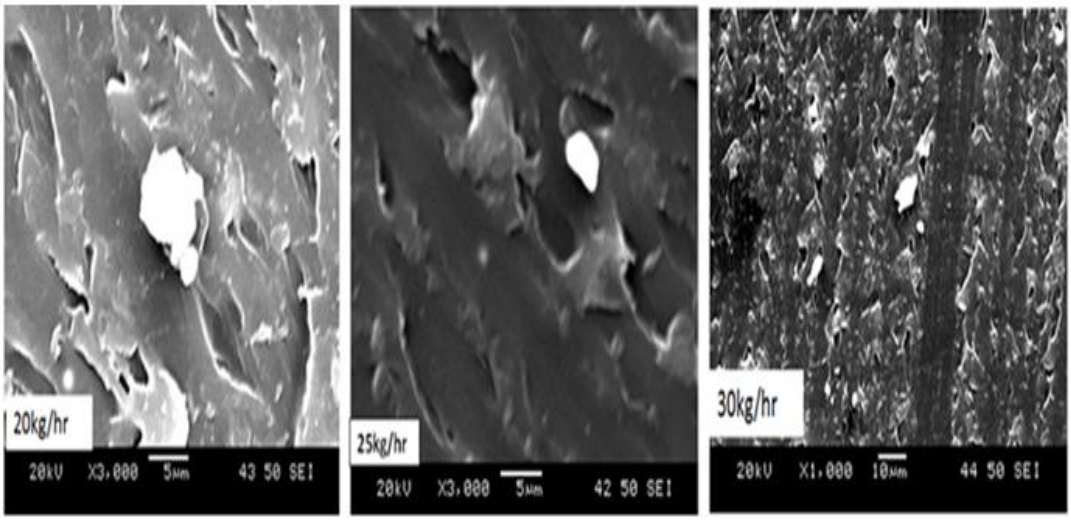


Figure 12: SEM- at variable feed rate at: (a) 20kg/hr (b) 25kg/hr (C) 30kg/hr

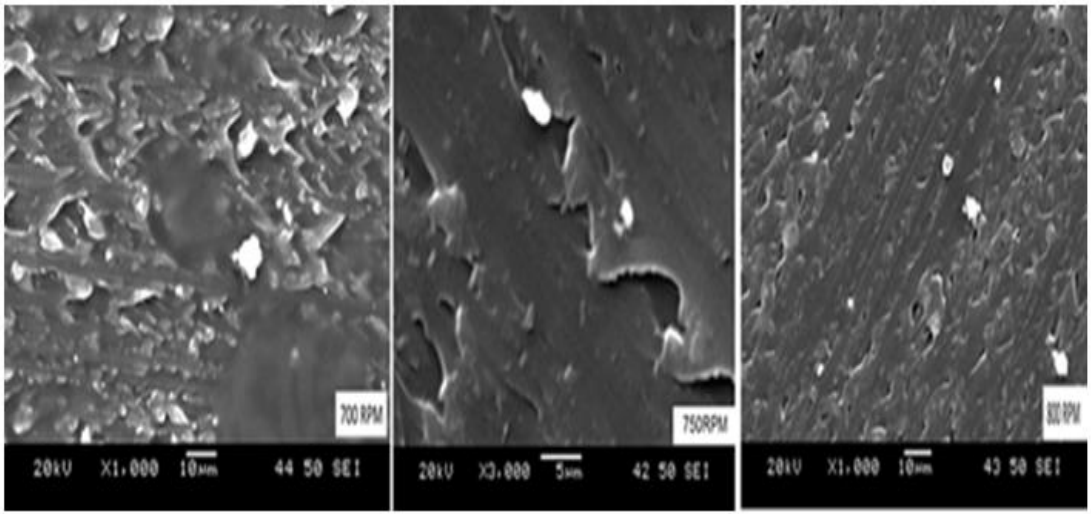


Figure 13: SEM – at variable screw Speed at :(a) 700rpm (b) 750rpm (C) 800rpm

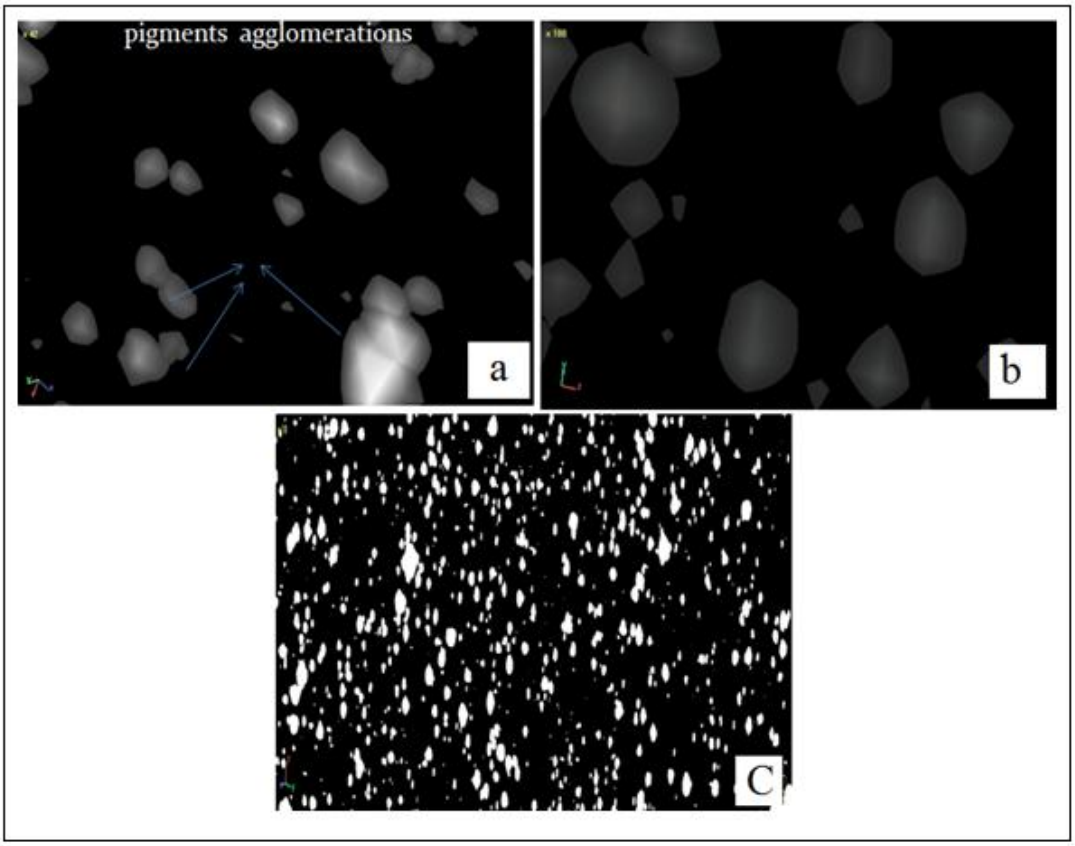


Figure 14: PC compounded grade-3, Process at 750rpm, 255 ^{o}C and 25kg/hr: (a) Agglomeration's (b) particle shape (C) Pigment distribution, using a µCT Scanner

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