Plastic Color Mismatch: Effect of Formulation and Processing Parameter

By

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Abstract

Color is a visual characteristic which imparts the ability to categorize different objects. When light strikes an object, there are three possibilities as to what can happen. The light may be reflected, refracted or scattered. Human perception of color is due to a combination of these. Plastics are polymeric materials. Polymers are generally colorless and need colorants in order to have some color. One or more pigments in certain ratios can be used to give a specific color to plastics. Slight variations in the proportions of either of the used pigments, or their dispersion, may result in a color that is different from the desired one. There can also be many other reasons for color mismatch such as processing or degradation effects.

This study utilized the historical data records of SABIC IP to look at effects of changing **Grade** on color (i.e. when the same color is produced from resin blends which may have different amounts of resins, pigments or additives). These records were also used to study effects of change in **screw diameter and configuration** on color (i.e. when the same grade of a color is produced on two different production lines, different screw diameters and configurations).

The effects of the processing parameters on the colors were also investigated by carrying out experiments using a twin screw extruder at SABIC IP's plant in Cobourg. Resins, pigments and additives were dry blended and extruded while being subjected to different processing conditions. Three processing parameters, namely temperature, rpm and feed-rate, were chosen for the study. A three level factorial design of experiments was used. An analysis of variance (ANOVA) has been utilized to find the effects of

individual parameters on color and the interaction between two parameters and their cumulative effect on color.

Acknowledgement

First of all I would like to thank ALLAH for helping me in completing my master's degree. After that I would like to highly acknowledge my supervisor Dr. G. M. Rizvi for all of his moral support and academic supervision throughout my stay in this project. I am thankful to my parents for their encouragement to pursue graduate studies. I would also like to thank all the SABIC staff members and my colleagues, especially Jamal Al-Sadi, for their continued support.

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Nomenclature

A = Light source represent normal light bulb

ANOVA = Analysis of variance

$\mathbf{D_o}$ = Outer diameter of screw

$\mathbf{D_r}$ = Root diameter

$\mathbf{d_{flight}}$ = Depth of flight

D65 = Light source represent average day light

Df = Degree of freedom

DOE = Design of experiments

F2 = Light source represent CWF lamp

F11 = Light source represent triple band lamp

K = Kelvin

kg/s = kilogram per second

L/D = Length to diameter ratio

MS = Mean Square

PC = Polycarbonates

rpm = Revolutions per minute

SS = Sum of squares

$\mathbf{T_g}$ = Glass transition temperature

MFI = Melt Flow index

μ = Micro 10^{-6}

Glossary

a* - Represents red to green axis on CIE 1964 color space.

b* - Represents yellow to blue axis on CIE 1964 color space.

CIE (Commission Internationale de l'Eclairage) – The International Commission on Illumination, the primary international organization concerned with color and color measurement.

Color space – Three-dimensional solid enclosing all possible colors. The dimensions may be described in various geometries, giving rise to various spacings within the solid.

Iilluminant – Mathematical description of the relative spectral power distribution of a real or imaginary light source — i.e., the relative energy emitted by a source at each wavelength in its emission spectrum. Often used synonymously with "light source" or "lamp," though such usage is not recommended.

L* - Indicate lightness and darkness on CIE 1964 color space.

Observer – The human viewer who receives a stimulus and experiences a sensation from it. In vision, the stimulus is a visual one and the sensation is an appearance. A phenomenon exhibited by a pair of colors that match under one or more sets of illuminants (be the real or calculated), but not under all illuminants.

Spectral power distribution curve – Intensity of radiant energy as a function of wavelength, generally given in relative power terms.

Spectrophotometer – Photometric device that measures spectral transmittance, spectral reflectance or relative spectral emittance.

Thermoplastics- Polymer that turns to a liquid when heated and freezes to a very glassy state when cooled sufficiently

Tristimulus – Of, or consisting of, three stimuli; generally used to describe components of additive mixture required to evoke a particular color sensation.

Thermosets- Polymer that irreversibly cure

Chapter 1

Introduction

North American compounding industry members have a very positive view of changing the concept of combining innovations and linking further growth and success in providing new solutions for North American markets and expanding innovation globally. In the year 2005, the total sale of compounded products by the North American compounding industry reached a figure of 4.5-5.4 billion kg (10-12 billion pounds), worth about $11 billion [1 & 2]. Over the last few decades, a significant increase in the Canadian plastics industry has singled out plastic production as an important industrial sector in Canada. In addition to having many advantages such as ease in manufacturing, being light weight, recyclable and cost effective, in comparison to other conventional materials, a major benefit of using plastics is the availability of a vast array of colors that add unique value to products, enhancing their attractiveness for consumers. However, producing the right color with minimal wastage has been a big challenge for plastic compounders who produce colored plastics for the plastic processing industry. In today's rapidly growing and highly competitive global market, this becomes even more challenging for compounders who are short lead-time suppliers of small lot sizes and mostly cater to the needs of prototype development. Success of all innovative product development depends directly upon the capability of a supplier to quickly realize the prototypes. Countries like Canada, who occupy a leading place in the present global market, essentially need to enhance their capability to develop prototypes in minimum possible lead-times.

Color mismatch problems in plastic arise due to many factors which include poor pigment dispersion, improper processing parameters during extrusion, and improper extruder selection. The dispersion of pigments has been very well studied in paints and coatings and has been the topic of numerous academic papers, textbooks and handbooks. The effect of processing parameters during extrusion on color has been discussed in detail in terms of food items. Most of the text on extruder selection covers different aspects of mixing between polymers, additives and pigments. The effect of different types of resins to produce plastics of the same color has rarely been addressed in terms of color science. However, combinations of different polymer blends have been studied extensively in terms of properties of material. Dispersion of these pigments in plastics has not been so thoroughly studied. A big difference between the two dispersion mechanisms is the high shear rates and the high processing temperatures and pressures involved in the manufacturing processes involving plastics. Other reasons for color variations include pigment or resin degradation or incorrect formulations. Nearly all the plastics used in manufactured products are colored.

1.1 Saudi Basic Industries Corporation (SABIC)

SABIC Innovative Plastics (formerly GE Plastics), is a recognized global industry leader in plastic and face such problems, at its manufacturing plant in Cobourg, Ontario. A core component of business at this plant is the supply of tailored plastics with customer specified colors. These are supplied to a large number of plastic manufacturers, all across the country and in the global market.

1.2 Collaboration between UOIT & SABIC

Companies like SABIC play a very important role in the rapid development of prototypes and hence facilitate innovation. Getting the correct color with minimum wastage is critical to such operations. Hence, SABIC is collaborating with UOIT (University of Ontario Institute of Technology) in conducting research to understand and resolve these issues. The collaboration between UOIT and SABIC provides a unique opportunity for carrying out experiments on lab scale equipment and as well as on production equipment. Use of production scale equipment for research studies is of course very expensive due to the large quantities (and cost) of materials involved, and as such, it is not very common. Due to the nature of operations at SABIC and their commitment to understanding these issues, this is a very unique opportunity that has been presented to academia.

1.3 Methodology

This research will conduct methodical scientific studies to improve color matching, color stability and consistency of compounded plastic materials. To tackle these problems, three concurrent sets of studies will be undertaken. The first set of studies involves data analysis of current and old production records at SABIC to look at the material and processing parameters that resulted in faulty batch productions. The second set of studies will specify and study the effects of controlled variations in processing parameters at the end of regular production runs and analyze the results of these changes. The third set of studies will utilize lab-scale equipment to observe effects of material and processing conditions. Parametric studies will be conducted to determine the effects of processing conditions and material formulations on the perceived colors in order to understand the basic scientific issues involved in color mismatch. The collected data will be correlated with existing and/or new models for predicting color matching formulations. Sensitivity analysis will be carried out to determine the effects of slight variations in pigment formulations on color. This will foster the basic scientific understanding of the effects that the processing and material parameters have on color perception when plastics undergo the commonly used manufacturing processes. In addition, this will also lead to refinements in existing model parameters or development of new models, which will be more efficient in predicting correct color matching formulations. The results from this project will be widely applicable in the plastic manufacturing industry. Extrusion is the primary process used by compounders. Therefore, these studies will provide additional insights into extrusion processing which will be applicable to a very broad segment of the plastic manufacturing industry.

The plastics manufacturing industry all over Canada is economically very important and has rapidly grown over the past few decades. Recognizing this importance, the Ontario government has recently provided $700,000.00 of funding for the formation of a state-of-the-art research facility, the Centre for Manufacturing Innovation (CMI), in collaboration with SABIC at their Cobourg plant.

1.4 Objective of Thesis

The scope of the overall project is very large (briefly presented above). The present study will cover the following aspects from the project.

1. Historical Data Analysis.
    a. Screw Diameter and Configuration: Using statistical model on the available production data in order to study the effect of screw diameter

and configuration when the same color is produced over different production lines.

b. Effect of Grade: Using a statistical model on the available production data in order to study the effect of changing grade when the same color is produced over the same line.

2. Processing parameters and color: To study the effects of processing extrusion parameters on color. Recommendations based on performed experiments.

This is a unique kind of study on plastic colorants which for which almost no literature is available.

1.5 Thesis Organization

Chapter 1 includes introductory words about the plastic industry and its economics in the North American region. It also very briefly highlights the problems of color mismatch in the plastic industry and covers the scope of the overall project and collaboration between UOIT & SABIC. Finally it discusses the main objectives of the present thesis. Chapter 2 will discuss background science especially about extrusion, color and ANOVA. It also includes a literature survey on the topic. Chapter 3 includes details about the experimental setup and experiment design. Chapter 4 covers the results and discussion section. Chapter 5 includes suggestions for future work.

Chapter 2

Background Science

2.1 Extrusion

Extrusion is the process of creating profiles or objects of fixed cross section. An extruder is essentially a screw rotating inside a barrel in which the motion of the screw moves material and generates friction and heat, which is used to melt plastic. They are normally mounted on a **base** in order to be on working height (3-4ft) [1]. The base is made quite heavy to avoid any possible movement or vibration.

The **drive motor,** which is mostly attached at the end of the screw, provides the power to move the material forward. The drive motor must be capable of changing speed. Variable frequency AC, or more commonly DC motors, are used because of their capability to change speed with change in input signal. One of the important parameters of the drive motor is its power capability (HP or KW). The power requirement for an extruder can be increased if the output increases, the barrel diameter increases, the screw length increases or high outputs are required [1]. It is also a function of material, i.e. materials with high viscosities and density need more power and vice versa.

Another important monitoring parameter for the extruder is its **current usage**. Current is measured in Amperes (abbreviated as amp) and measured through ammeter. Higher amps mean viscous or dense material and the extruder needs high power to move it forward. A too high ampere leads to motor burn out. Optimum temperature and screw speed should be used to avoid high amps. Too low processing temperatures result in more motor power requirement which may burn the motor, whereas too high of a temperature may result in degradation of the material.

The motor runs faster than the screw, so the gear reduction mechanism is required between screws and the drive motor. This reduction is achieved by gears and pulleys. Extruder rpm may vary from extruder to extruder depending upon the required output, material behaviour under high rpm, material residence time, etc. High rpm on a small extruder will not give enough residence time for the material in the extruder to melt and mix properly and may also cause vibrations, while low rpm in a large extruder may result in over heating of the resin which results in **extruder burn** in some areas. Typical speed for a 1-inch inside barrel diameter extruder is 50rpm [1]. Thrust bearing is used to prevent back movement of the screw.

The **barrel** of the screw is the area in which the screw rotates and material flows inside it. Barrels are mostly made from hardened steel coated with wear and corrosion resistant metals. The barrel's inside diameter is an important parameter in specifying an extruder's size and capability. Extruders on production scale would typically have a barrel diameter of 45mm or higher [1]. The outside surface of the barrel is covered with electrical heating elements which provide heat for resin to melt especially at start-up. The whole barrel is divided into different zones and heating elements because individual zones are controlled separately. Thermocouples are used to monitor the temperature of individual zones.

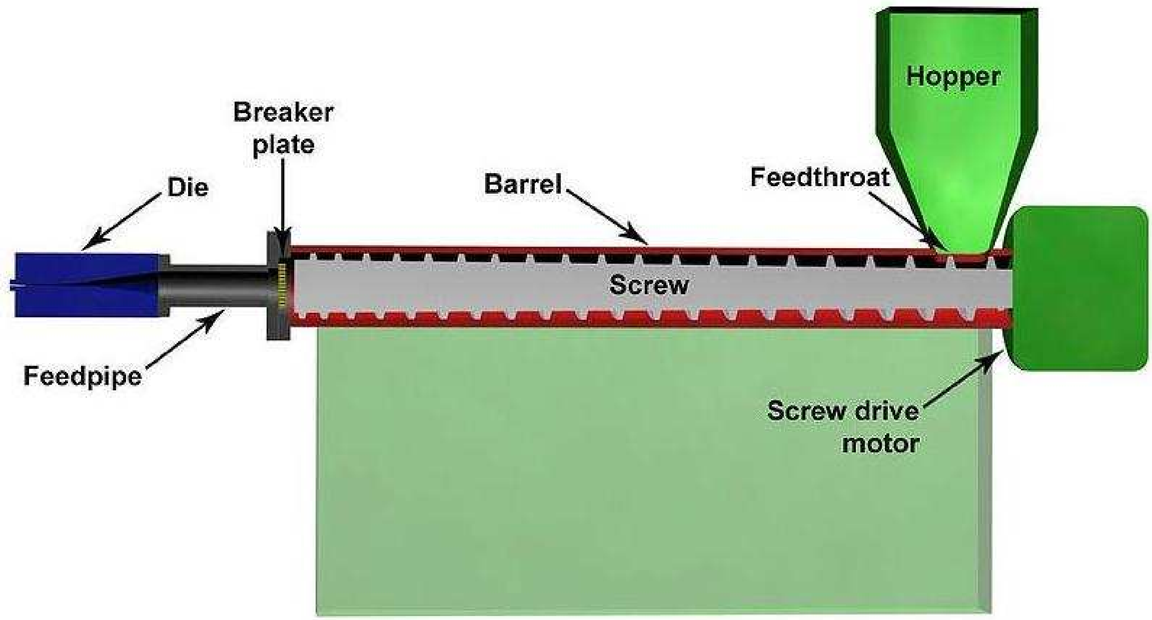


Figure 2-1 *A Typical Extruder Setup [3]*

These thermocouples are connected with a controller. Based on the resin processing temperature requirements, one can set the temperatures of individual zones.

Resin is generally fed to the barrel by using feeders. The opening on the top of the barrel connected with the hopper is called a feed throat. Material is generally fed on to the screw by gravity from the hopper, however mass flow rate of material can also be controlled by using a separate feeder. rpm of that screw feeder is adjusted such as input flow rate is achieved. The feeder can also be equipped with a load cell which is used to measure amount of material in the feeder and to control the feed-rate of the material. It can also be equipped with a mechanism for drying.

The main component of the extruder is its **screw**. The screw is attached to a gearbox. The screw is made from one of the two methods: 1) flights usually machined on a solid rod or 2) splines made on a rod and screw elements would be inserted onto it separately. Each helix turn is known as a flight which is like thread on a screw. The distance between the outside diameter of the screw and barrel wall is known as screw/barrel clearance. Depending upon the extruder diameter, this clearance usually varies with varying diameter, for example an extruder with the diameter of 25mm has a clearance of 0.1mm and an extruder with the diameter of 58mm has a clearance of 0.4mm. This clearance increases with wear and tear of the screw. After considerable wear, the screw should be rebuilt. Rebuilding of the screw is expensive and the cost can sometimes go up to 75% of the cost of a new screw [1] but it still will not give efficient output. In order to avoid wear, flights of the screw should be coated with wear resistant material. As mentioned above, the screw is the key element of the extruder and it performs several functions during extrusion, including imparting mechanical energy as part of the melting process, the mixing of different ingredients and creating pressure difference to convey material forward towards the die. The extruder may be a single screw or twin screw. An important parameter of the screw is its length to diameter ratio (L/D). This L/D ratio is also indicative of the ability of the screw to mix the ingredients of a compound. It is also a measure of energy needed to run the extruder. High L/D means good mixing and melting ability of the screw but high energy requirements. Typical range of L/D varies from 16:1 to 37:1 [1]. The root diameter is the diameter of the solid shaft. Screw's outside diameter is

$$D_o = D_r + 2d_{flight} \tag{2.1}$$

where d_{flight} represents flight depth which is the distance between flight top to outside the surface of the solid shaft. The screw's outer diameter is constant throughout the screw's length, but the root diameter changes. Due to change in root diameter, screw/barrel flight depth also changes. If root diameter is large then flight depth is small and vice versa. The change in screw flight depth results in imparting different shear rates on the material. The flight is inclined at an angle called pitch of screw. The most common pitch angle is 17.5^{o} [1], which is constant over the entire screw length. The width of the flight is typically $^{1}/_{10}$ of the distance between flights and also remains constant over the entire screw length.

Screw design and type vary for different materials, but generally all screws have the following three sections.

1. Feed Section
2. Compression Section
3. Metering Section

The **feed section** starts right below the feed throat. This is the portion of the screw from where solid materials (resin, additives, and pigments) enter the screw. This section has constant screw diameter and its purpose is to convey material away from the feeding zone. Root diameter remains constant which indicates constant flight depth. Material in this section will move under constant shear. The first few zones of the feed section should be lower in temperature. Immediately heating to a high temperature after the material enters would cause it to stick around the feed section and it will start rotating with the screw. This will block the incoming material and choke the throat of the hopper so that material flow will stop. This is called bridging of extruder. Bridging is worse in extruders with large diameters, at high rpm and especially with those materials that soften at low temperature. To prevent bridging, temperatures of the first couple of zones must be kept lower as compared to the other zones in the screw, so that material is heated gradually in the conveying zone. Chilled water is also used to prevent overheating.

The second section of the screw is the **compression section**. In this section, root diameter increases gradually, which results in a decrease in flight depth. This is the main section where mixing of resin with other ingredients like pigments and additives takes place [1]. This section is also known as the transition zone. Shear rate increases in this section due to reduction in fight depth. If a vent is needed, it is normally located after the transition section. Air and other volatile materials are usually purged out from this vent. The elimination of these volatile materials is important in making a pore free product. This section adds heat to the material. Heating in extruder zones are adiabatic which means there is no addition or removal of heat, but the mechanical energy from the motor is converted into heat due to friction [1]. Energy imparted on resin can be increased by reducing the pitch of flight. Lengths of each section vary for different materials.

The last section is the **metering section** which is at the end of the extruder and is connected to the die. Root diameter remains constant in this section. The flight depth in this section is less than in the feeding section. The material should be completely molten before it reaches the metering section. Shear rate is maximum in this section due to shallow flight depth. High pressure build-up in this section will push the material to the die zone.

Compression ratio is another important parameter in extrusion. It is defined as a ratio of flight depth in the feed section to the metering section. Compression ratio is actually a measure of energy imparted to the material. It ranges from 1.1:1 – 5:1.

A material enters the die zone after the metering zone. Depending upon the shape, friction in the die zone is normally higher than in the screw zones.

2.2 Color

Color is defined as the visual characteristic of an object as perceived by humans. It imparts the ability to categorize different objects [2, 31]. In order to determine a color we must define its "triad”. The triad of a color consists of, 1) a light source, 2) an object for which color is to be determined, and 3) an observer or a detector. In visual color matching, humans act as observers and their eyes as detectors. This means that in visual matching, color is defined as the interpretation of physical information thus adding another factor in the above defined triad which is "Human Psychology” [32].

2.2.1 Light Source: The first component of color Triad

In order to see color, a light source is needed (act as illuminants). Similar colors can be perceived differently under different lights. Light is an electromagnetic radiation. Human eyes can respond to these electromagnetic radiations in the range from 400nm to 700nm [3, 33]. This is known as the visible spectrum.

Wavelength interval, frequency interval and energy of pure hue in visible range are shown in table 2-1.

Table 2-1 *Wavelength, Frequency and Energies of Pure Hues [3]*

Color	Wave Length Interval (nm)	Frequency Interval (THz)	Energy of Pure hue KJ/mol
Red	~ 700-635	~ 430-480	171
Orange	~ 635-590	~ 480-510	193
Yellow	~ 590–560	~ 510-540	206
Green	~ 560–490	~ 540-610	226
Blue	~ 490- 450	~ 610-670	254
Violet	~ 450-400	~ 670-750	285

Table 2-1 shows that light with a higher wavelength appears red and light with a shorter wavelength appears violet. Red has the lowest energy while violet has the highest energy.

The sum of all the radiations of the visible spectrum produces pure white light. Intensity of the light source is also an important factor in the definition of color. Objects may look different under high intensity light as compared to dim light (low intensity light). All the above mentioned factors make it essential to select a light source before defining tolerances of color [2, 32]. Light sources can be described by their spectral power (energy distribution).

There are 2 types of light sources

1. Natural
2. Artificial

The sun is a natural source of light. The white light which can be divided into visible spectrum is termed as "polychromatic light". However, in order to define differences between colors, we cannot use the sun as a light source because the sun is dependent upon weather conditions. In cloudy weather objects may appear darker than on a sunny day.

So in order to differentiate between colors, we need to use artificial illuminants. Light from an artificial source depends upon temperature and relative spectral distribution of the light source.

Spectral Power Distribution of sunlight is shown in figure 2-2. **Black bodies** are an important group of light sources used to measure color [34]. The color temperature (absolute temperature measured in Kelvin K=C+273) of the lamp of a black body is an important factor [2]. An illuminant D65 is the blackbody heated to 6500K. CIE (Commission Internationale de 1'Eclairage) has defined a number of illuminants. Some of the most commonly used illuminants defined by CIE (with the temperatures at which they are heated) are defined below [2,32].

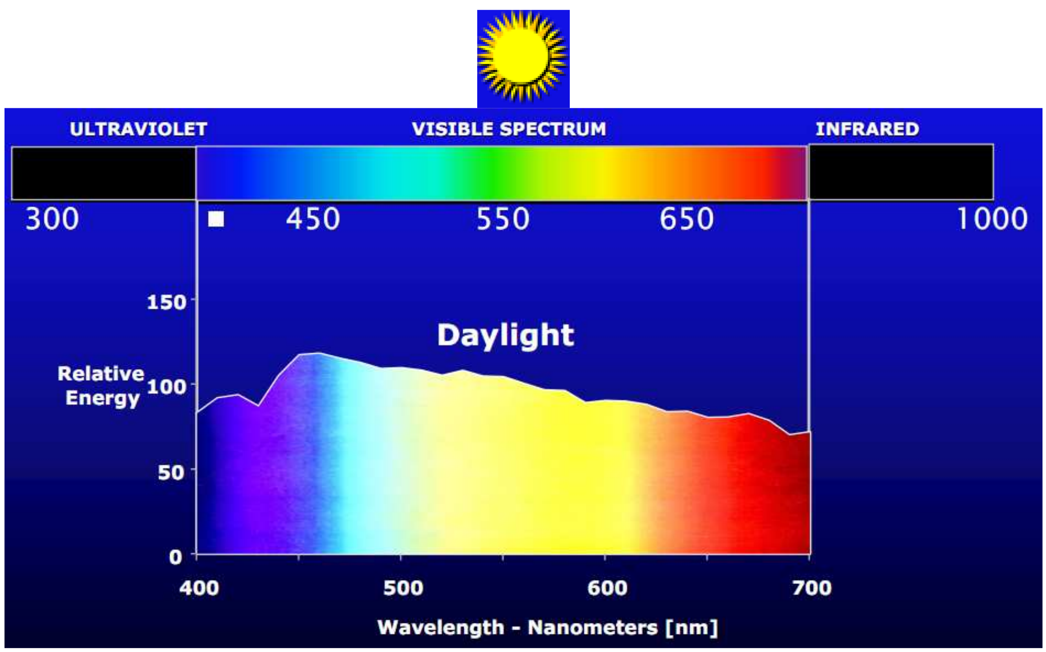


Figure 2-2 *Power Spectral Distribution of Sunlight [32]*

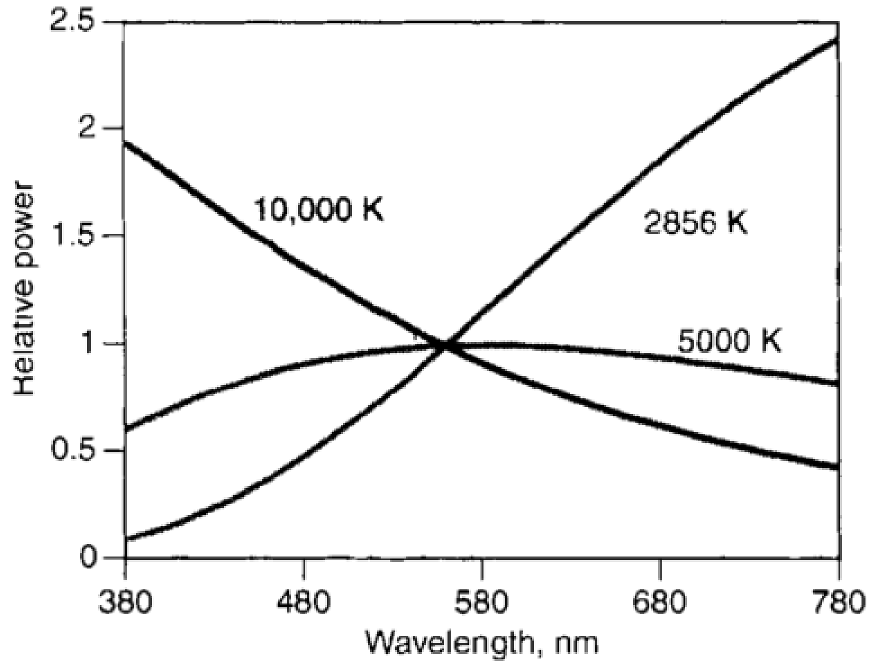


Figure 2-3 *Spectral Power Distribution of Black Bodies Heated at Different Temperatures (IES, 1981; Billmeyer and Saltzman, 1981).*

Table 2-2 *Black Bodies Temperature and Presentation [2]*

Light Source	Black body Temperature (K)	Presentation
D65	6500	Average day light
A	2856	Normal light bulb
F2	4230	CWF lamp
F11	4000	Triple band lamp

All these illuminants have different power spectral densities which mean that the same object will look different under different light sources. In order to measure color, external light should be blocked.

2.2.2 Object: The Second component of color Triad

Object is the second part of the color triad. Plastics are polymeric materials and generally polymers are colorless and need colorants in order to have color [2]. These colorants may be pigments or dyes.

When light strikes an object, there are 3 possibilities. The light be transmitted, absorbed, or scattered. Either one or a combination of all of the above will occur. First energy can be transmitted through the object. A small amount of radiant energy (about 4% [2]) will be scattered from the flat surface. This scattering is due to the refractive index. Different polymers have different refractive indexes which are why they will scatter light differently [2, 3]. Thus, two different polymers with different refractive indexes will scatter light differently, which ultimately means that they will look different even if they are colored by the same colorants. When an object is viewed from different angles it appears different. This property of material is called "flop".

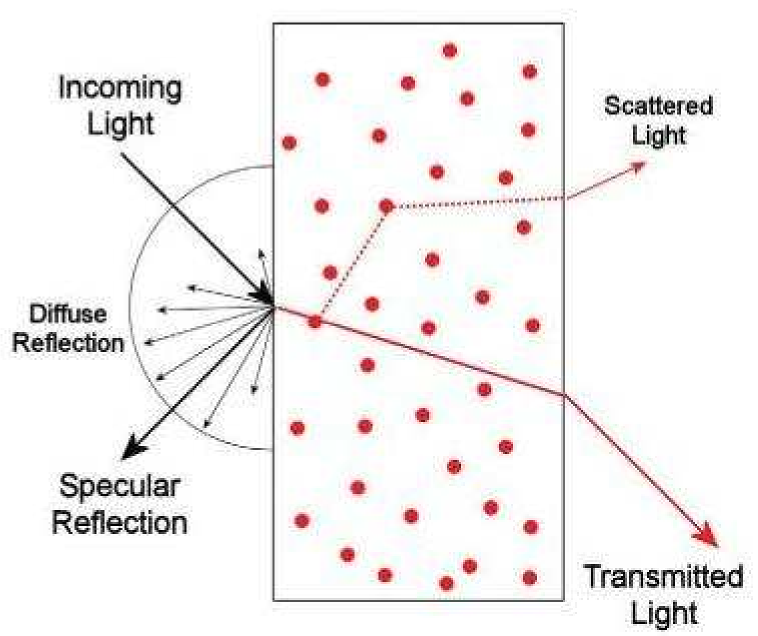


Figure 2-4 *Interaction of Light with Object [61]*

The second way an object interacts with light radiations is through absorption. Absorption is defined as the process in which the energy of light photons is taken up by matter. If an object absorbs all wavelengths of radiation it will appear black, and if it reflects all wavelengths of the radiations then it will appear white [35]. If some parts of the wavelengths of a radiation are absorbed then the object will appear colored. Leaves appear green because the pigment in leaves absorbs all radiations except green.

The third way an object interacts with light radiations is through scattering [36]. In plastics, scattering is caused by the presence of colorants (pigments, dyes). Pigments have their own refractive index so when mixed with resins, the color of an object will depend upon the accumulative amount of absorption and scattering. Particle size is an important factor that will affect the amount of scattering and ultimately the color of an object. Two materials with the same pigment but different pigment size will scatter light differently, which ultimately affects the appearance of the object [2, 37].

2.2.3 Observer: The third component of color Triad

The final part of the color triad is the observer. It can be a human observer or an instrument (e.g spectrometer). Based on the research of Whright and Guild in 1931, CIE proposed 2^o observer. It was believed that color detecting cones of the eye were located within 2^o arc of the fovea, which is why 2^o observer was chosen as a standard observer at that time. In 1960 it was observed that cones were present in a larger space of the eye than 2^o. Thus in 1964, CIE defined a new standard observer which covered all cones of the eye within a 10^o arc. This observer is known as 10^o standard observer [38].

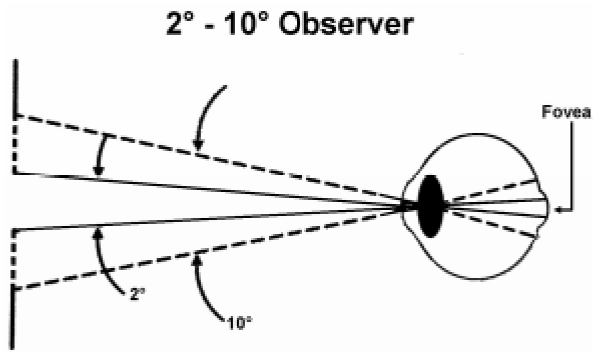


Figure 2-5 *Light at Different Angles [38]*

2.2.4 Color Language (Measurement)

Color can be measured in terms of hue, chrome and brightness. If we scale these three quantities then we will be able to define color numerically. Based on 20 hues, Albert Munsell was the first one to formulate color numerically. He devised a three dimensional color system in which brightness varies from 0 to 10, where 0 is a pure black hue and 10 is a pure white hue [39].

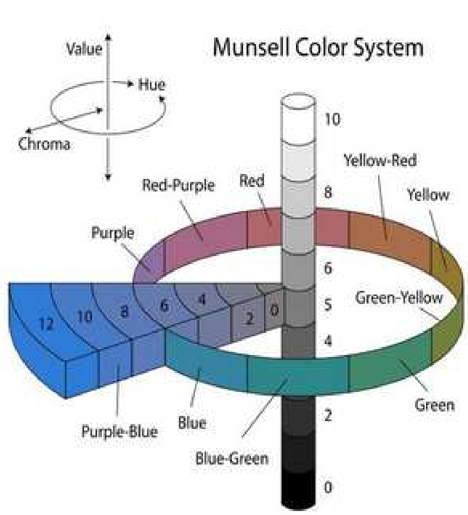


Figure 2-6 *Munsell Color System [3]*

Other methods to define color were developed and presented by CIE. These include L*, a*, b* and the Yxy color space system (based on tristimulus values XYZ).

In the Yxy system, XYZ tristimulus values are based upon three primary hues (red, green, blue) and all other colors are a mixture of these three [40]. XYZ are useful but not easily visually clear due to which in 1931, CIE [41] defined new color space in the XY graph in which light hues are in the center and saturation increases towards the edges. In 1964, CIE changed its recommendation to 10^{o} standard observer and developed L*, a*, b* space which gives a more accurate perception of color. This system is often termed as CIELAB and L*, a*, b* is known as tristimulus [2, 42, 40].

CIE color space is the most commonly used color space. It is a uniform color space where the color difference between two points is plotted on L*, a*, b* and can be visually observed. The L*a* and b* values are plotted in rectangular coordinates. Tolerances can be defined in terms of delta values of tristimulus. One can adjust the color by looking at these delta values.

Mathematically, tristimulus can be defined as below [43].

$$L^{*} = 903.9\left(\frac{Y}{Y_n}\right) \tag{2.2}$$

$$a^{*} = 500\left(f\left(\frac{X}{X_n}\right) - f\left(\frac{Y}{Y_n}\right)\right) \tag{2.3}$$

$$b^{*} = 200\left(f\left(\frac{Y}{Y_n}\right) - f\left(\frac{Z}{Z_n}\right)\right) \tag{2.4}$$

Where $f\left(\frac{X}{X_n}\right)$ can be defined as

$$f\left(\frac{X}{X_n}\right) = \sqrt[3]{\frac{X}{X_n}}\ when\ \frac{X}{X_n} > 0.008856 \tag{2.5}$$

$$f\left(\frac{X}{X_n}\right) = 7.87\left(\frac{X}{X_n}\right) + \frac{16}{116}\ \ when\ \frac{X}{X_n} \leq 0.008856 \tag{2.6}$$

$f\left(\frac{Y}{Y_n}\right)$ can be defined as

$$f\left(\frac{Y}{Y_n}\right) = \sqrt[3]{\frac{Y}{Y_n}}\ when\ \frac{Y}{Y_n} > 0.008856 \tag{2.7}$$

$$f\left(\frac{Y}{Y_n}\right) = 7.87\left(\frac{Y}{Y_n}\right) + \frac{16}{116}\ \ when\ \frac{Y}{Y_n} \leq 0.008856 \tag{2.8}$$

$f\left(\frac{Z}{Z_n}\right)$ can be defined as

$$f\left(\frac{Z}{Z_n}\right) = \sqrt[3]{\frac{Z}{Z_n}}\ when\ \frac{Z}{Z_n} > 0.008856 \tag{2.9}$$

$$f\left(\frac{Z}{Z_n}\right) = 7.87\left(\frac{Z}{Z_n}\right) + \frac{16}{116}\ \ when\ \frac{Z}{Z_n} \leq 0.008856 \tag{2.10}$$

And X_n, Y_n, Z_n represents tristimulus values of illuminants [38]

__Table 2-3__ values of Xn and Yn for Different Observer [32]

Illuminants	2^o Observer		10^o Observer	
	X_n	Z_n	X_n	Z_n
A	109.83	35.55	111.16	35.19
C	98.04	118.11	97.30	116.14
D65	95.02	108.82	94.83	107.38
F2	98.09	67.53	102.13	69.37
TL4	101.40	65.90	103.82	66.90
UL3000	107.99	33.91	111.12	35.21
D50	96.38	82.45	96.72	81.45
D60	95.23	100.86	95.21	99.60
D75	94.96	122.53	94.45	120.70

and $Y_n = 100$ for both 2^o and 10^o Observer and for all illuminants and deltas values can be expressed as

$$\Delta L^* = L^*_{Target} - L^*_{sample}$$

$$\Delta a^* = a^*_{Target} - a^*_{sample}$$

$$\Delta b^* = b^*_{Target} - b^*_{sample}$$

$+ \Delta L^*$ means sample is lighter than target.

$- \Delta L^*$ means sample is darker than target.

$+ \Delta a^*$ means sample is redder than target.

$- \Delta a^*$ means sample is greener than target.

$+ \Delta b^*$ means sample is yellower than target.

$-\Delta b^*$means sample is bluer than target.

The overall difference between target values and trismulus can be measured in terms of dE*[38-43].

$$dE^* = \sqrt{\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2}}$$

2.3 Analysis of Variance (ANOVA)

An ANOVA is an analysis of the variation present in an experiment. It is a test of the hypothesis that the variation in an experiment is no greater than that due to normal variation of individuals' characteristics and error in their measurement [44]. ANOVA is a statistical method used to find statistical significance of factors [45]. It consist of 4 main components.

2.3.1 Sum of Squares (*SS*)

The total sum of square of the model is defined as sum of the squared deviation from the mean due to the effect of individual term or the interaction between two term and sum of the squared deviation that are not explained by the model [46].

$$SS_{Total} = SS_{Model\ terms} + SS_{Residual}$$

Where $SS_{Residual}$ have two components [45-46]

$$SS_{Residual} = \mathrm{SS_{LOF}} + \mathrm{SS_{Pure\ Error}}$$

SS_{LOF} is the portion of the residual $SS_{Residual}$ that is due to the model not fitting the data. It is the weighted sum of squared deviations between the mean response at each factor level and the corresponding fitted value.

The pure error sum of squares ($SS_{\text{Pure Error}}$) is a measure of the effect contributed by error associated with repeatability. It is the corrected sum of squares of the repeat observations at each level of input and then pooled over all the levels of input.

2.3.2 Degree of Freedom (df)

Degree of freedom in ANOVA is defined minimum number of values required to specify all data points in the sample. N data points require N no. of degree of freedom. If the mean of the data is known and we have N data points, then we can say our df is N-1 [4] .

Total df for a given model is defined as

$$df_{total} = df_{Model\ Terms} + df_{Residual}$$

Where each model term has one df. df for residual is defined as

$$df_{Residual} = df_{\text{LOF}} + df_{\text{Pure Error}}$$

df_{LOF}vary with model. Linear models for scattered data have high df while quadratic or cubic model have less df for same scattered data. This is because of an increase in the number of points captured by estimated model regression.

2.3.3 Mean Square Value

It is the ratio of the sum of squares to the degree of freedom. Mathematically it can be expressed as [47]

$$MM = SS/df$$

Like SS and df, it is also calculated for both error and model term.

2.3.4 F-Value

The F Value for model terms is the test for comparing the variance related with that term with the residual variance. It is the ratio between mean square value for the term and mean square value for error [48].

$$F_{Term} = \frac{MM_{term}}{MM_{residual}} = \frac{\frac{SS_{term}}{df_{term}}}{\frac{SS_{residual}}{df_{residual}}}$$

The larger the F-value for the term, the more effective the term in the model. However F-value for LOF (lack of fit should be small) otherwise large error associated with the model term.

2.3.5 p-Value

This is the probability value for the term that is associated with the F Value for this term. It is the probability of getting an F Value of this size if the term **did not** have an effect on the response. In general, based on 95% confidence level, a term that has a probability value less than 0.05 would be considered a significant effect. A probability value greater than 0.10 is generally regarded as not significant [47-48].

2.4 Regression Calculation

Factors involved in calculation of ANOVA terms would either be quantitative or qualitative [4].Quantitative factors are also termed as categorical factors e.g. colors on different plastic production lines, or grade of color produced for a given line, etc. Qualitative factors are also termed as numeric factors e.g. temperature, rpm and feed-rate of extruder while producing a certain grade.

If we were to perform experiments on three temperature levels, namely 230^{o}C, 255 ^{o}C, and 280 ^{o}C, for a given rpm and feed-rate of extruder, and measure output values on these temperature levels, but we wanted to predict some value in between these temperatures such as 245 ^{o}C, the experimenter would then develop an interpolation equation. This equation is known as empirical model. Empirical model equation has constants associated with it and these constants can be determined by using given output data. The general approach to calculate these constants from the empirical equation is known as a regression calculation.

In a regression calculation, the least square method is used to calculate coefficients of the terms [44]. If the calculated regression captures all the points, then in this case residual (error) is 1. If calculated regression is not able to captures all the points drawn between input and output, then residual value will be less than one and there is an error associated with it.

A simple 3 input linear model can be predicted as [4]

$$y = a_0 + a_1x_1 + a_2x_2 + a_3x_3$$

Where y represents output and x_1, x_2, x_3 represents input. A more complicated linear model includes the interactions between two terms and interaction between all 3 terms as well.

$$y = a_0 + a_1x_1 + a_2x_2 + a_3x_3 + a_4x_1x_2 + a_5x_2x_3 + a_6x_3x_1 + a_7x_1x_2x_3$$

Similarly, quadratic and cubic model regressions can be calculated. Calculation of quadratic and cubic terms will also include square and cubic input terms.

Cubic regression can be calculated as.

$$\begin{aligned} y = a_0 + a_1x_1 + a_2x_2 + a_3x_3 + a_4x_1x_2 + a_5x_2x_3 + a_6x_3x_1 + a_7x_1x_2x_3 + a_9x_2^2 \\ + a_{10}x_3^2 + a_{11}x_1^2x_2 + a_{12}x_2^2x_3 + a_{13}x_3^2x_1 + a_{14}x_1^2x_1 + a_{15}x_2^2x_1 \\ + a_{16}x_3^2x_2 + a_{17}x_1^3 + a_{18}x_2^3 + a_{19}x_3^3 \end{aligned}$$

In general, by adding more terms you can improve lack of fit (residual) but it is more complex.

2.5 Response Surface Methodology (RSM)

Response surface methodology is a useful method by which interactions between two input variables and their mutual effect on output can be determined. The results are presented in terms of contours graphs, where two inputs will be on the X and Y-axis, while the output will be on Z-axis [50]. Contour lines connect the points with the same output value. For this case more than 2 input interactions and response surface can be found between different inputs and by fixing remaining inputs. Consider the 3 input variable (x_1, x_2, x_3) process mentioned above. Response surface can be plotted for three different combinations of inputs. These combinations include $(x_1, x_2, y), (x_1, x_3, y)$and (x_2, x_3, y). While plotting response surface between (x_1, x_2), other input x_3 will act as a constant [51] .

2.6 Design of Experiments -Factorial Design (DOE)

Multivariables are acting in most of the processes [49]. For example, in extrusion, variables are barrel zones temperatures, rpm of screw, feed-rate from feeder, etc. Factorial design includes variations of these variables at different levels.

The number of experiments in factorial design depends upon the level of each variable and for one categorical factor it is calculated as:

No. of Experiments =N^m.

Where N is the number of levels and m is the number of variables.

So let us say the number of experiments to evaluate a specific grade of a specific color by varying three extruder parameters (i.e. temperature, feed-rate, and rpm) at three different levels, is 27.

By using factorial design we will be able to

1. Vary individual input parameter with different combinations of other input parameters.

2. Applying ANOVA on factorial design in order to find effect of individual input parameter on output and to find the interaction between two input parameters and their mutual effect on output response.

3. Determining optimum conditions for controllable input by keeping in view un-controllable inputs.

So in short, the DOE is used to optimize the process for the best possible output.

Chapter 3

Literature Review

3.1 Plastics

Plastics are polymers of long chain molecules and may contain some additives in order to have certain properties [2]. Plastic may be made from natural or synthetic monomers [2, 3]. The word plastic is derived from the Greek πλαστικός (plastikos), meaning capable of being shaped or moulded, from πλαστός (plastos) meaning moulded [3].

All plastics can be categorized into two major types [2].

1. Thermoplastics

2. Thermosets.

A **thermoplastic** melts and turn into liquid at high temperatures and exists as a solid at room temperature [52]. They are mostly long chain molecule and can be moulded into the desired shape at elevated temperatures. These are different from thermosets because they can be melted whenever heated and can be remoulded as well [1]. Thermoplastics are elastic and flexible above glass transition temperature. Chemical changes in the composition of thermoplastics are negligible. Their molecular weight ranges from 10,000 to 20,000 amu [53].

Thermosets can either exist as solids or liquids at room temperature [1, 2]. When heated, initially thermosets will turn into liquids (low viscosity), but on further heating, they will turn into solid structures [64]. Thermosets cannot melt on reheating thus cannot be remoulded again [65]. Other than a normal covalent bond, there is a linkage between different chains of polymers known as cross linkage [64].

3.2 Polycarbonates

Brand name commonly associated with GE plastic (currently known as SABIC Innovative Plastic) [1]. The resins made from these types of polymers are often termed as Lexan®. Polycarbonates involve condensation of polymers that involve bonding of carbon with three oxygen atoms, a form of carbonates.

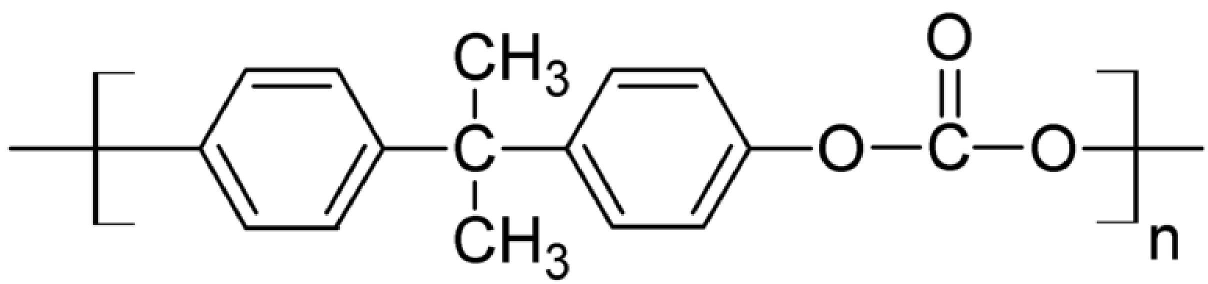


Figure 3-1 *Structure of Polycarbonates [3]*

The complex and aromatic structure of polycarbonate is responsible for its physical and mechanical properties. Polycarbonates are non-crystalline in nature but they are as strong as crystalline, e.g nylon and actel [67]. The mechanical performance is due to the large aromatic structure. Polycarbonates exhibit large resistance to the inter-molecular movement, which leads to high strength and high melting (softening) temperature [1]. A PC resin shows high resistance to creep. All these properties are due to the aromatic structure, pendent group and hydrogen bonding. Most polycarbonates are amorphous in nature. The combination of high optical clarity and toughness are unique properties that co-exist in PC. Polar nature of PC leads to moisture absorption. Absorption rate is high in melt, PC resins needed to be dried before processing [3, 68].

Uses : safety sheets and goggles, lenses, glazing panels, business machine housing, instrument casings, lighting fittings, safety helmets, electrical switchgear, laminated sheet for bullet-proof glazing, twin-walled sheets for glazing, kitchenware and tableware, microwave, cookware, medical (sterilizable) components [63]

3.3 Literature Review on Extruders

Extruders and their workings have been discussed in details previously. Many different researchers have worked on different aspects of SSE. For example Jiang has discussed residual time distribution in SSE [17]. Wilczyñski discussed theological properties [18] and morphology[19], of polymers in SSE using computer models. Based on experimental and historical data, Wilczyñski has determined performance of SSE to produce a good quality product [20]. Fenner has explained surging in SSE [21]. Advantages of SSE are that it is a proven technology with lowest capital cost [16]. About 90% of the extruders currently in use for production of plastic are single screw extruders [1]. Disadvantages include, high screw rpm with greater risk of burning at the screw tip, lower output rates, and inability of keeping melting temperature low with higher head pressure, and requirement of a drying system, Single screw extruders for polymer processing have typically had length to dimeter ratios (L/D) of 37:1 [1].

3.3.1 Twin Screw Extruders

Twins screw extruders are extensively used with materials that are sensitive to heat and provide better mixing than SSE [1]. Screws in the twin screw are intermeshed with each other. Barrel shape and linkage between the screw and motor used in twin screw extruders are different than those used in single screw extruders.

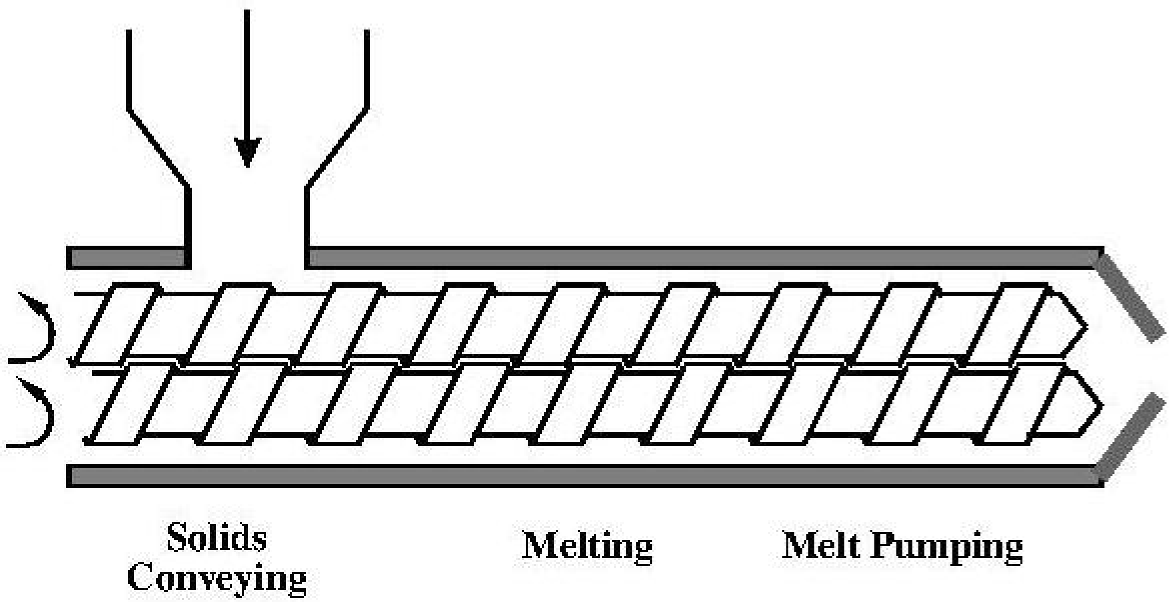


Figure 3-2 *Twin Screw Extruders [62]*

There are two different types of intermeshing screws:

1. Co-rotating
2. Counter-rotating

In **co-rotating** screws, both screws move in the same direction, either clock or anti-clock wise [6]. Material in co-rotating passes from screw to screw, material moves from the top of the 1st screw to the bottom of the 2nd screw, and then from the bottom of the other screw to the top of the 2nd helix of the 1st screw [1,6]. In this way, material gets high contact with the extruder barrel and thus there is efficient thermal heating [6]. Mixing in co-rotating is better than counter rotating. Shear rate is uniform all over the flow. Werner&Pfleiderer GmbH (W&P) is one of the leading producers of co-rotating twin screw extruders. Co-rotating extruders are mostly used for compounding. Their rpm vary from low range (10 - 20 rpm) to high range (up to 1200rpm). The screws in fully intermeshed co-rotating extruders can either be close fitting or open fitting [71, 72].

Close fitting screws will have large flight thickness and open fitting screws have small flight thickness. Close fitting screws have conjugated screw profiles (i.e. profiles in which the shape of the channel and flight is similar) [72]. Close fitting screws usually run at low rpm because of the high pressure in the inter-meshing region which may result in wear of elements. Close fitting twin screw extruders have good conveying capacities [6, 68].

In **counter rotating** twin screw extruders both of the screws move in opposite directions, if one moves clockwise then the other would move in a counter clock-wise direction [73]. Material is built up at the junction of the two screws, which is called the material bank [6]. This material bank is then conveyed along the screw length. Shear rate is maximum in between screws but low at other locations [1, 6].

3.4 Color in Extrusion-Literature Review

Color and its different aspects in general have been extensively investigated in literature, which ranges from color consistency [22] to color appearance [22] and its presentation in different workspaces [23]. The way in which color is perceived by humans is due to the presence of cones in the eye [2]. Anything can be colored by using dyes or pigments. Dyes are soluble while pigments are in-soluble in polymers [2]. Pigments are widely used in paints, plastic, glasses and various other industries for coloring purpose [25]. Pigments can be organic and in-organic [26]. Synthesis of color may contain one or more pigments in a given ratio [27-30].

Blanco and F. Apruzzese have discussed color changes taking place during extrusion for food products by using a neural network approach [7, 8]. S. Il has examined the effects on color of yellow maize grits when extruded in a counter-rotating twins screw extruder. He varied feed-rate, rpm, barrel temperature and moisture to study their effects on color of maize grits. He used the response surface method (RSM) to find the interactions between different processing parameters and their mutual effect on color [9]. Bhattacharya has also studied changes taking place in color tristimulus during extrusion using the RSM approach. In his study, he used a co-rotating twin screw extruder and showed the interaction between screw speed and temperature (extrusion parameters) and their mutual effect on color tristimulus. His findings show that L* and b* are mostly dependent upon barrel temperature during the cooking extrusion.[11]. Hanwu Lei used an image analysis technique to find the color changes taking place during extrusion in rice–glucose–lysine blend. He analyzed color of the extruder at different conditions. He considered five different variables which include both processing parameters and screw geometry. He used the statistical model (ANOVA) for his analysis [10]. He related color tristimulus with specific mechanical energy and temperature of the product.

3.5 Mixing

A number of different mixing processes take place in polymers [6]. When the different types of materials involved in mixing are all solids, then it is known as solid-solid mixing. An example includes the mixing of resins with power pigments. The mixing of polymers in molten form with solid fillers is known as solid-liquid mixing [70]. An example includes the mixing of pigments or additives with resins.

Generally all kinds of mixing can be divided in to two major categories:

1. Dispersive

2. Distributive

In **dispersive** mixing, size of cohesive components is reduced [6]. This type of mixing is also known as intensive mixing. An example is the addition of pigment in resins where the size of pigments would be reduced. Pigment particles agglomerate due to adhesive force such as in polar particles. Dispersive mixing aims at breaking such agglomerates, not changing particle size, in order to achieve proper surface finish [56].

Mixing that takes place in the absence of any cohesive resistance is known as **distributive** mixing [6]. It is also called extensive or simple mixing. In distributive mixing, particle size remains the same but material is spread over the whole area [54]. It can occur both in solids or liquids or solid-liquid mixtures [6, 55].

Polymers with fillers (e.g. additives) are known as compounds. The mixing of two different types of polymers is known as blending. An example of blends is the mixing of two different resins of polycarbonates [6]. If polymers are completely dissolved in each other and give single phase then it is known as miscible blends. This is quite un-usual for most of the polymers. Mostly polymers have limited solubility in each other and individual polymers retain their own identity [6]. These types of blends are known as immiscible or multiphase blends. The real objective in mixing is to obtain a single phase or a homogeneous distribution of phases. So mixing taking place between immiscible blends is generally dispersive in nature [71].

Many different researchers have worked on mixing during extrusion. For example, Zuilichem has compared mixing in twin and single screw extruder. He found that mixing in co-rotating screws is better than single screw extruders. Each screw has both dispersive and distributive elements. High shear rate in a twin screw leads to dispersive mixing [12]. Yerramill has explained the mixing effects in kneading elements of the screw. Dispersive mixing is dominant over distributive mixing in the tantalisation region of the kneading section [14]. Twin screw extruders are widely used in polymer industries and give efficient mixing and pumping [15].

Chapter 4

Experiment Design

Based on the historical data provided by SABIC and the properties of selected grades, experiments were carried out to study the effects of the processing parameters on the trismulus values and the dE. Three processing parameters were chosen for the study, namely barrel zone temperatures (measured in ^{o}C), rpm and feed-rate (measured in kg/hr). Two types of experimental methodologies were employed:

1. Study of General Trends (GT), while all except one parameter are kept constant
2. Design of Experiments (DOE), in which a number of parameters are varied simultaneously

GT: This set of experiments includes variation of processing parameters around the values on which SABIC usually operate for production of these specific grades on given a line (fixed screw configuration and diameter). Its involve variation of one parameter to see its effect on tristimulus (L*, a*, b*) and on color difference (dE*). Each parameter was varied at five different levels from minimum to maximum (range obtained from historical data). Variation of temperature is from 230^{o}C-280^{o}C with fixed rpm and feed-rate. The temperatures of the first two barrel zones were kept lower than the other barrel zones to avoid bridging [1]. Similarly rpm was varied from 700-800 and feed-rate from 20-30kg/hr.

Tables 4-1 to 4-3 shows the values of processing parameters on which experiments have been performed according to general trends.

Table 4-1 *Variation of Temperature (^{O}C)*

S.No	S.P Temperature (^{o}C)	S.P rpm	S.P Feedrate (kg/h)
1	230	750	25
2	240	750	25
3	255	750	25
4	270	750	25
5	280	750	25

Table 4-2 *Variation of rpm*

S.No	S.P Temperature (^{o}C)	S.P rpm	S.P Feedrate (kg/h)
1	255	700	25
2	255	725	25
3	255	750	25
4	255	775	25
5	255	800	25

Table 4-3 *Variation of Feedrate(kg/h)*

S.No	S.P Temperature (^{o}C)	S.P rpm	S.P Feedrate (kg/h)
1	255	750	20
2	255	750	23
3	255	750	25
4	255	750	27
5	255	750	30

Where, S.P set point. Actual values for temperatures were recorded within $\pm 3^{o}C$ of the set point temperature, while SP for rpm and feed-rate matches perfectly

4.1- DOE

Experiments according to DOE were also performed. Factorial design was chosen for DOE. Each parameter was varied on three different levels according to the factorial design. The advantage to using a 3 level factorial instead of a 2-level factorial (mostly used by many researchers) is it gives a more clear picture about the behaviour of tristimulus than would a 2-level factorial, because a 2-level factorial draws a straight line between maximum and minimum value and does not provide any information about in between behaviour.

Design-expert 7.1.6® was used for designing the experiments. All three process variables were used as numeric factors while grade was treated as a categorical factor. So total number of experiments was calculated as

$$No. Of\ Experiments = CN^{m} \tag{3.1}$$

where C=2, N=3 and M=3 which give us 54 experiments for two different grades with three process variables varied at three different levels.

Table 4-2 represents the combinations of temperature, rpm and feed-rate on which experiments have been performed.

***Table4-4**, 3 Level factorial design of experiment*

S.No	Temperature (^oC)	rpm	Feedrate (kg/h)
1	255	750	30
2	280	750	30
3	255	800	25
4	255	700	20
5	280	750	20
6	255	800	30
7	280	700	25
8	230	750	30
9	255	750	20

10	280	700	20
11	230	750	20
12	280	800	30
13	230	700	20
14	230	800	30
15	255	750	25
16	230	800	20
17	255	800	20
18	255	700	30
19	230	700	25
20	230	800	25
21	280	700	30
22	230	700	30
23	280	800	25
24	280	750	25
25	230	750	25
26	255	700	25
27	280	800	20

4.2- Experimental Setup

Planned experiments were performed in Centre for Manufacturing Innovation (CMI) at Coburg, Ontario, Canada. A co-rotating twin screw extruder made by Coperion Werner & Pfieiderer (model No. ZSK 26) was used. The extruder was connected with a controller. Specifications of the used extruder are

Figure 4-1 *Extruder*

Table4-5 *Extruder Specification*

Screw Diameter	25.5mm
Max.rpm	1200
Max Power	28KW
Channel Depth	4.55
Barrel/ Screw Clearance	0.1mm
No.of barrel Zones	9
No.of die Zones	1
Do/Di	1.55
L/Do	37
Dimensions (L*W*H)	2500*800*1800 mm

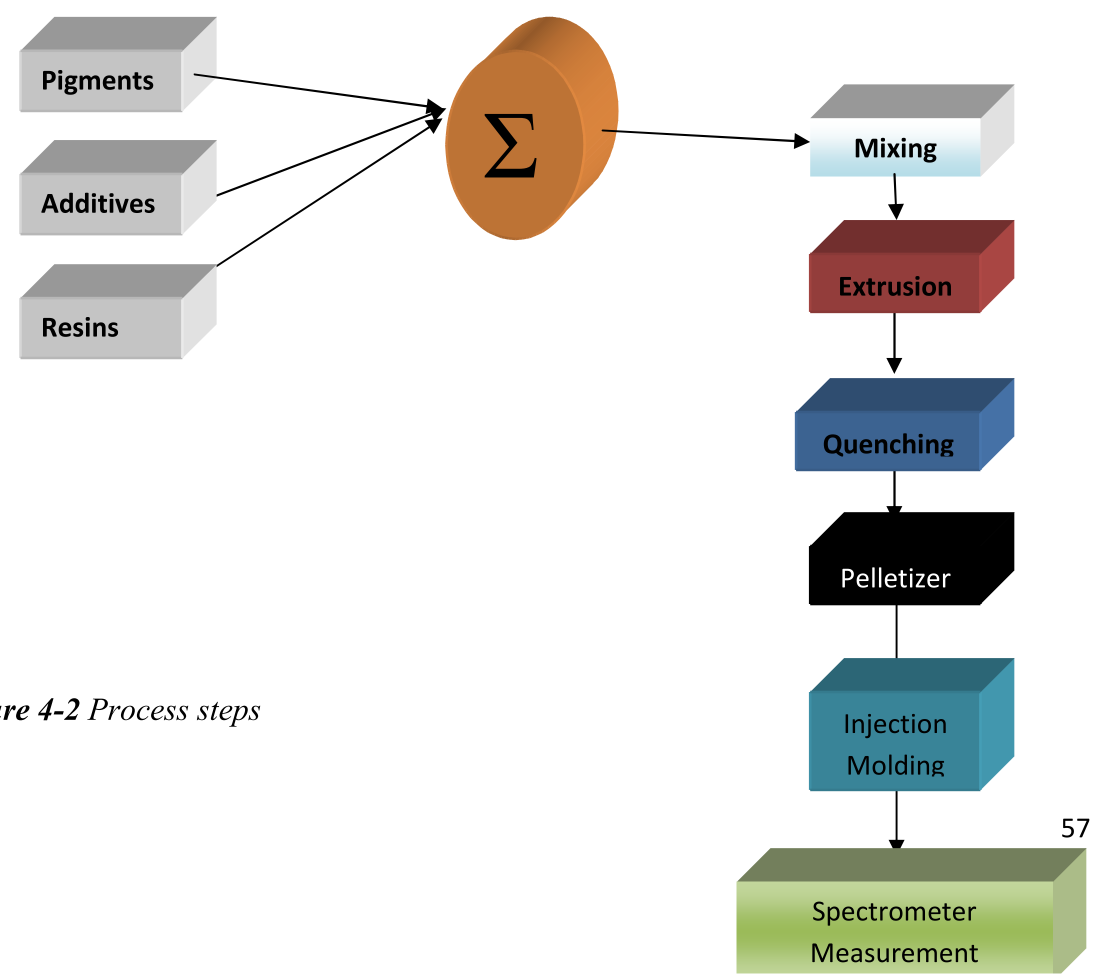


Figure 4-2 *Process steps*

4.3 - Experimental Procedure:

The extruder was set on the desired temperature. When the extruder reached the saturation level, then it was first purged with resin only. This is to remove any previous residue in it. Then the mixed blend of resins, additives and pigments was poured into the feeder. Feedrate of the blend is controlled through PLC. The feeder was equipped with a load cell, which was used to control the amount of material in the feeder.

After feeding, material passes through barrel zones where it gets heated. Zone one to five are conveying zones. Zones six to eight are mixing zones (contain mixing elements e.g. kneading blocks). Zone nine is again another conveying zone and then the molten materials enter the die zone. Then the molten melted material is drenched in cold water where it gets hardened. Then it passes through a drying section where moisture on the surface was removed by blowing pressurized air over it.

Figure 4-3 *Extrusion setup*

Afterwards, it enters into a pelletizer and is chopped into pellets. These pellets are then dried again using a dryer and then moulded into rectangular coupons /chips of 3*2*0.1 in (L*W*D) using injection moulding. Before taking the moulded samples, the moulding machine was first purged and based on recommendation of a SABIC expert, only the last samples for each experiment were collected.

Tristimulus values for these samples were measured by using spectrophotometer (Xrite, Model No. Color i7). Tristimulus values for the standard (target, desired color) were also measured. By using tristimulus readings of both the standard (target) and the sample, the dE was evaluated which is basically the geometric distance or the overall difference in color between the target and the sample.

Figure 4-4 *Injection Moulding*

Figure 4-4 *Spectrometers*

Chapter 5

Results and Discussions

The objective of this study was divided into two different stages. 1st is the historical data analysis and 2nd is the effect of processing parameters on color. In both cases, color deviation in terms of dE* was measured and analyzed.

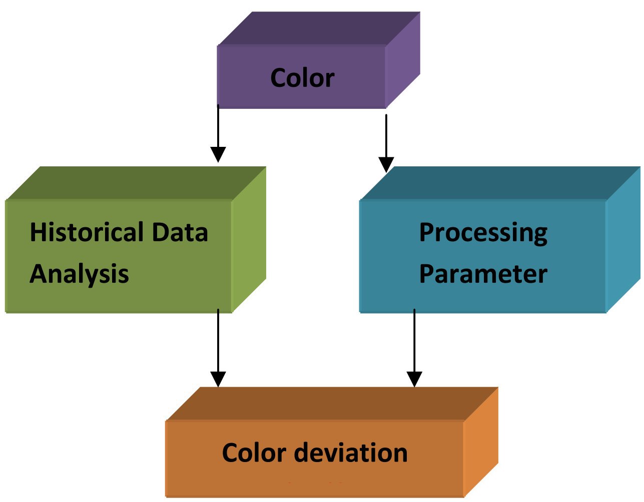


Figure 5-1 *Basic Flow Chart*

5.1- Historical Data Analysis- Identification of Pigments causing mismatch

Using the production records of SABIC, two different types of analysis were carried out which includes:

a. Effects of Changing Grade on color i.e. when the same color is produced from a resin or blend which may have different proportions or amounts of resins, pigments or additives.

b. Effects of change in screw diameter and configuration on color i.e. when the same grade of a matter is produced on two different production lines having different screw diameter and configuration.

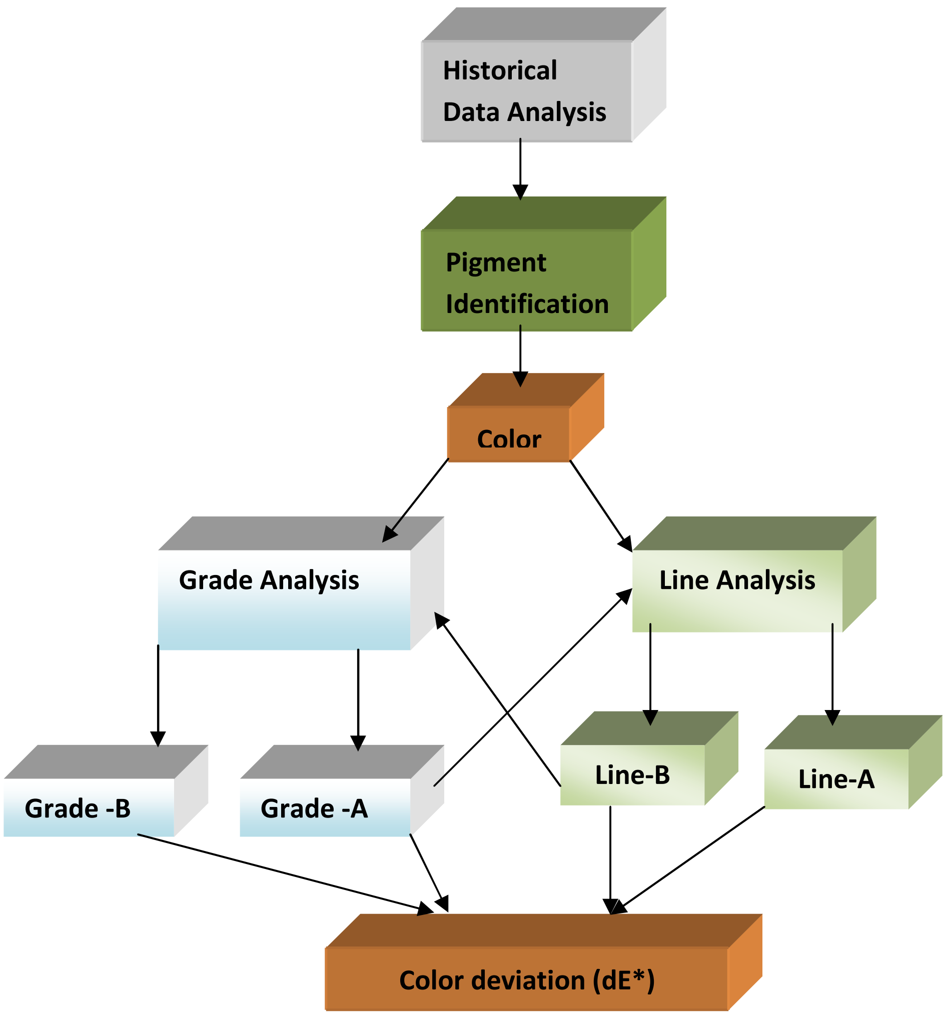


Figure 5-2 *Flow chart for historical data analysis*

Based on the historical records of SABIC IP for last two years (01/2009-06/2010), pigments causing most of the mismatch problems were identified. Then color made from these pigments showing high adjustments were identified. Each color has different grades. Production records for two different grades of the same color produced on the same production line were extracted in order to study the effect of changing resin amount in blend on color. Similarly, production records for the same grade produced on two different production lines (i.e. Line-1, Line-2) were extracted from historical data in order to study the effect of changing production line (diameter and configuration) on color.

5.2- Effect of changing grade on color

Using the production data from the compounding company (SABIC) for producing a specific color to achieve a desired value of CIE tristimulus reading (L*,a*b*) by using different grades (different amount of resins with same amount of pigments) on the same production line has been analyzed. Samples were measured against the desired values or target, and based on difference in values of $(L^*, a^*, b^*)_{sample}$ and $(L^*,a^*,b^*)_{target}$ adjustments were made. These adjustments were analyzed in terms of color difference (dE*). Analysis of variance (ANOVA) was applied to find the interactions between different pigments and their mutual effect on color difference (dE*) for two different grades. There are five inputs for chosen grades. Four are numeric (a pigment amount in PPH, namely A,B,C,D) and one is categorical with two levels each representing different grades (namely E). Linear regression was calculated. There were some aliased terms in the model which were neglected to avoid any misleading information in the model. Design Expert® (Version 7.1.5) from Stat Ease was used for statistical analysis.

5.2.1- Material

Table 5-1: *Color Formulation Used for Grade G1 & G2*

S-No	Type	PPH Grade-G1	PPH- Grade-G2
1	Resin-1	30	
2	Resin-2	70	100
3	Pigment-A (White)	1.76	1.76
4	Pigment-B (Black)	0.00968	0.00968
5	Pigment-C (Red)	0.01602	0.01602
6	Pigment-D (Yellow)	0.1084	0.1084

A blend of two Poly (bisphenol-A-carbonate) was used with pigments and additives. These resins were manufactured by SABIC IP. The amount of resin and pigment mentioned in Table 5-1 is suppose to produce a color with L*=67.28, a*=1.42 and b*=4.92. The meltflow index of resin-1 is 25 g/10 and for resin-2 is 6.5 g/10min.

5.2.2- Experimental

A single screw extruder was used for the production of these lots. Following is the line specification of the used extruder:

Table 5-2 *Operating Line Information*

Line	Extruder Size (mm)	HP (KW)	Maximum rpm	Number of barrels	L/D	No.of Die Zones
TSE	40	150	1000	9	37	4

Table 5-3 *Average Operating Conditions*

rp m	BZ 1	BZ 2	BZ 3	BZ 4	BZ 5	BZ 6	BZ 7	BZ 8	BZ 9	DZ 1	DZ 2	DZ 3	DZ 4
750	45.	76	212	242	252	257	260	261	263	262	276	265.	258.

5.2.3- Model Results

Table 5-4 *Figure ANOVA for Grade Analysis*

Source Terms	Sum of Squares (dE*)	df (dE*)	Mean Square (dE*)	F-Value (dE*)	p-value ,Prob > F (dE*)
Model	0.946677	8	0.118335	6.209409	0.0004
A-A	0.003808	1	0.003808	0.199838	0.6594
B-B	0.161159	1	0.161159	8.456526	0.0084
C-C	8.49E-05	1	8.49E-05	0.004453	0.9474
D-D	0.343672	1	0.343672	18.03362	0.0004
E-Grade	0.004328	1	0.004328	0.227114	0.6386
AE	0.005655	1	0.005655	0.296722	0.5917
BE	0.025708	1	0.025708	1.348987	0.2585
CE	0.297057	1	0.297057	15.58756	0.0007
Residual	0.400203	21	0.019057		
Cor Total	1.34688	29			

The Model F-value of 6.21 implies that the model is stasticially significant. There is only a 0.04% chance that a "Model F-Value" this large could occur due to noise.Values of "Prob > F" less than 0.0500 indicate model terms are significant. The analysis shows that pigment B, D and intercation between grade Pigment C (i.e. model term CE) are significant model terms. More data is required to find the missing interactions. Interaction of red pigment with grade is crucial. Similar results have been found by Farid and Rizvi [74].

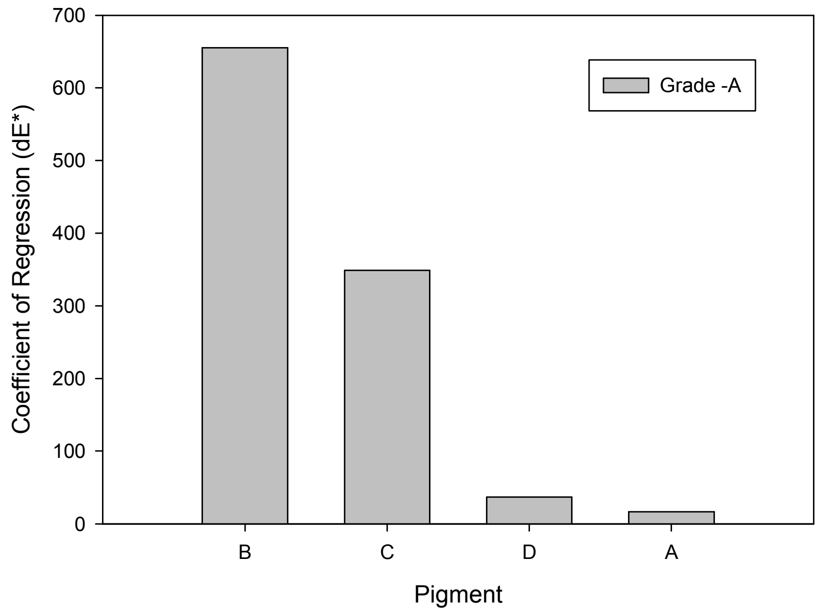


Figure 5-3 *Regression Coefficients for Grade-G1*

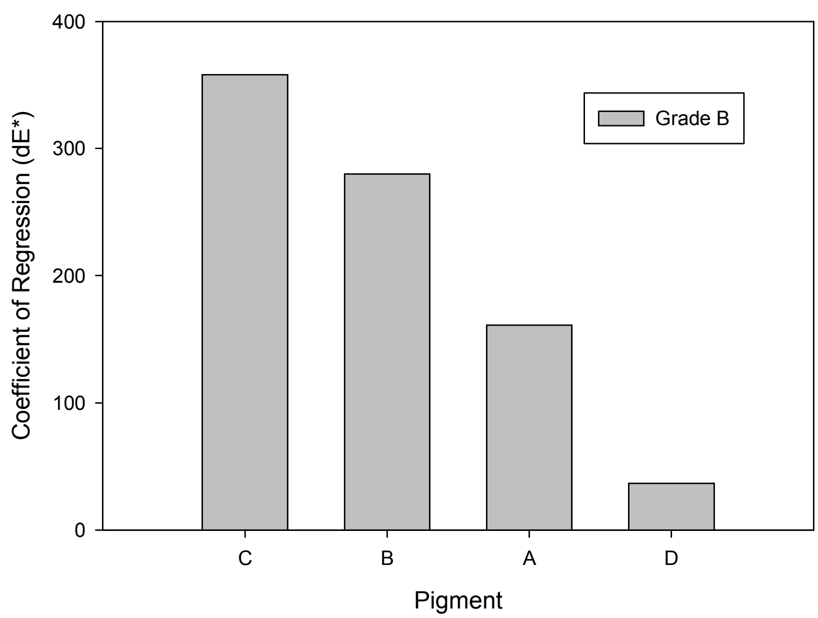


Figure 5-4 *Regression Coefficients for Grade-2*

Figures 5-3 and 5-4 show the difference in coefficients of the calculated regression for the pigments. Pigment A has a significant effect on the value of dE* for grade- G2 while having a negligible effect on grade G1. Pigment B has major effects on the value of dE in both grades but is more dominant in grade G1. In grade-G1 it is increasing the values of dE* and vice versa in grade-G2. Farid and Rizvi [74] have already predicted the drastic behaviour of this pigment. However, more quantitative data is required for better analysis. Pigment D does not have a prominent role.

5.3- Effect of changing Production Line on color

Using the production data from the compounding company (SABIC) for producing a specific color to achieve a desired value of CIE tristimulus reading (L*, a*, b*) at different extruders and with same grade has been analyzed. Samples were measured against the desired values or target, and based on differences in values of $(L^*, a^*, b^*)_{sample}$ and $(L^*, a^*, b^*)_{target}$ adjustments were made. These adjustments were analyzed in terms of color difference (dE*). Analysis of variance (ANOVA) was applied to find the interactions between different pigments and their mutual effect on color difference (dE*) on different lines with different screw diameters and configurations. The difference in contours in the interactions will reflect the effect of screw diameter and configuration. There are five inputs for the chosen grades. Four are numeric (pigments amount in PPH, namely A,B,C,D) and one is categorical with two levels, each representing different production (namely E) lines. Linear regression was calculated. There were some aliased terms in the model which were neglected to avoid any misleading information in the model. Design Expert® (Version 7.1.5) from Stat Ease was used for statistical analysis.

5.3.1- Material

Poly (bisphenol-A-carbonate) was used as a resin. This resin was manufactured by SABIC IP. The amount of resin and pigment mentioned in Table 5-5 is supposed to produce a color with the L* = 67.28, a* = 1.42 and b* = 4.92.

Table 5-5 *Color Formulation Used for Line Analysis*

S-No	Type	PPH	Grams (Batch of 6kg)
1	Resin	100	6000
2	Pigment-A (White)	1.76	105.6
3	Pigment-B (Black)	0.00968	0.5808
4	Pigment-C (Red)	0.01602	0.9612
5	Pigment-D (Yellow)	0.1084	6.504

The meltflow index of used resin is 6.5 g/10min. Low melt flow index shows that it is an extrusion resin. As melt flow is inversely porpotional to viscosites, the resin for the production of used resins is viscous.

5.3.2- Experimental

Data from two different extruders with the following specifications has been analyzed.

Table 5-6 *Operating Line Information*

Extruder	Extruder Size – Type (mm)	HP (KW)	Maximum rpm	Number of barrels	L/D	No.of Die Zones
Line-1	70-TSE	300	555	9	37	2
Line-2	58-TSE	350	860	9	37	4

Table 5-6 *Average Operating Conditions*

Line	rpm	BZ 1	BZ 2	BZ 3	BZ 4	BZ 5	BZ 6	BZ 7	BZ 8	BZ 9	DZ 1	DZ2	DZ 3	DZ 4
Line -1	213	245	260	266	287	269.	268	262	273.	213	245	260	-.	-
Line -2	540	52	88.	204	255	258	288	272	268.	267	269	295.75	269.	261

5.3.3- Model Results

Table 5-8 *ANOVA for Grade Analysis*

Source Terms	Sum of Squares (dE*)	df (dE*)	Mean Square (dE*)	F-Value (dE*)	p-value ,Prob > F (dE*)
Model	0.745623	7	0.106518	5.848401	0.0006
A-A	0.443483	1	0.443483	24.34965	< 0.0001
B-B	0.019177	1	0.019177	1.052902	0.3160
C-C	0.238772	1	0.238772	13.10988	0.0015
D-D	0.343672	1	0.343672	18.86951	0.0003
E-Line	0.39797	1	0.39797	21.85075	0.0001
AE	0.389767	1	0.389767	21.40034	0.0001
BE	0.130854	1	0.130854	7.184633	0.0137

Residual	0.400688	22	0.018213		
Cor Total	1.146311	29			

The Model F-value of 5.85 implies the model is significant. There is only a 0.06% chance that a "Model F-Value" this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicate model terms are significant. The analysis shows that A, C, D, E, AE, BE are significant model terms. More data is required to find the missing interactions. However it is quite obvious from the given analysis that changing production line affects the output color significantly and the observed color difference is due to change in production line. Each production line has a different number of screw elements (both mixing and conveying elements), which results in different amounts of shear on blend, which ultimately affects the color.

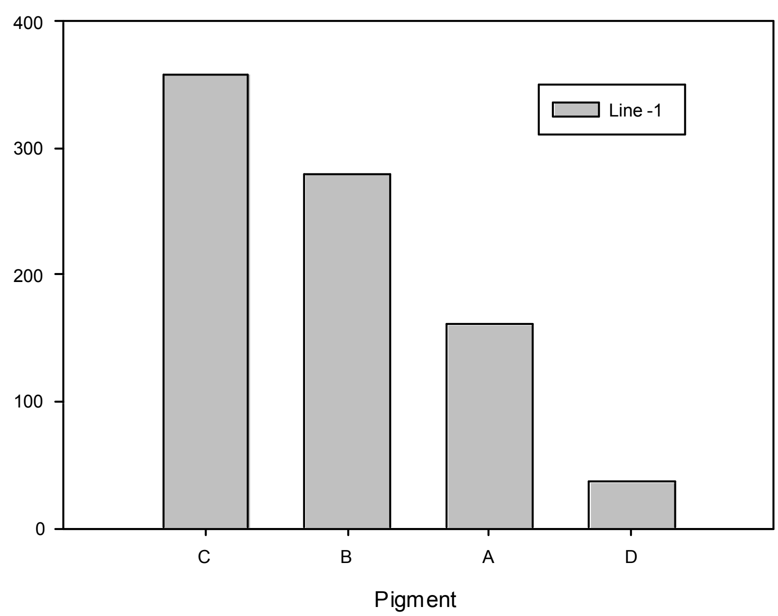


Figure 5-5 *Regression Coefficients for Line-1*

In figures 5-5 and 5-6, pigment A is showing completely different behaviour when processed on different lines. Similar opposite behaviour is observed in terms of pigment B. However pigments C and D are showing quite similar behaviour in both of the lines. It can be seen that the effect of pigment A is more dominant in line-2 while effect of pigments B, C and D are more dominant in line-1. As mentioned earlier, this can be explained in terms of shear mounted on pigments during processing.

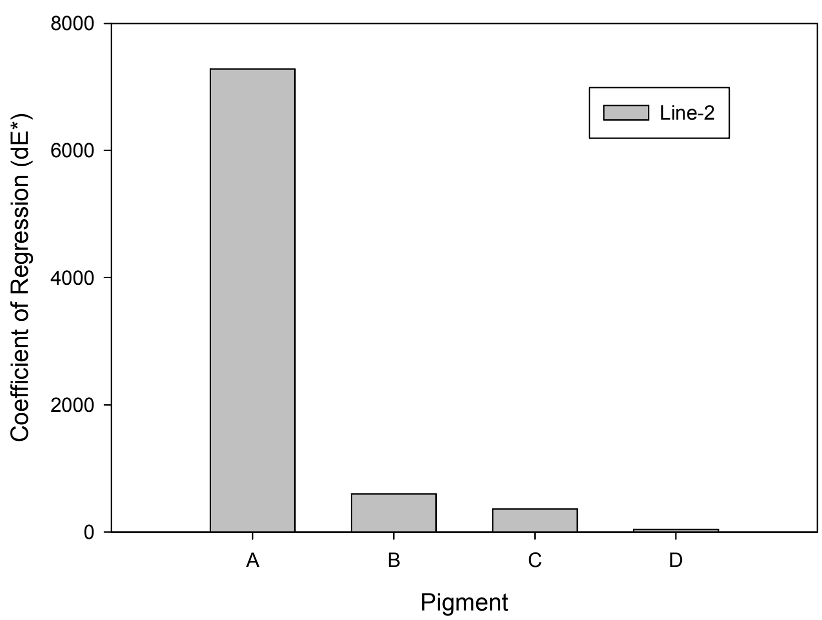


Figure 5-6 *Regression Coefficients for Line-2*

5.4 - Processing Parameters

The planned experiments were carried out at SABIC IP according to DOE and GT. Data generated from these experiments was analyzed using ANOVA. Each of the color tristimulus is separately analyzed below:

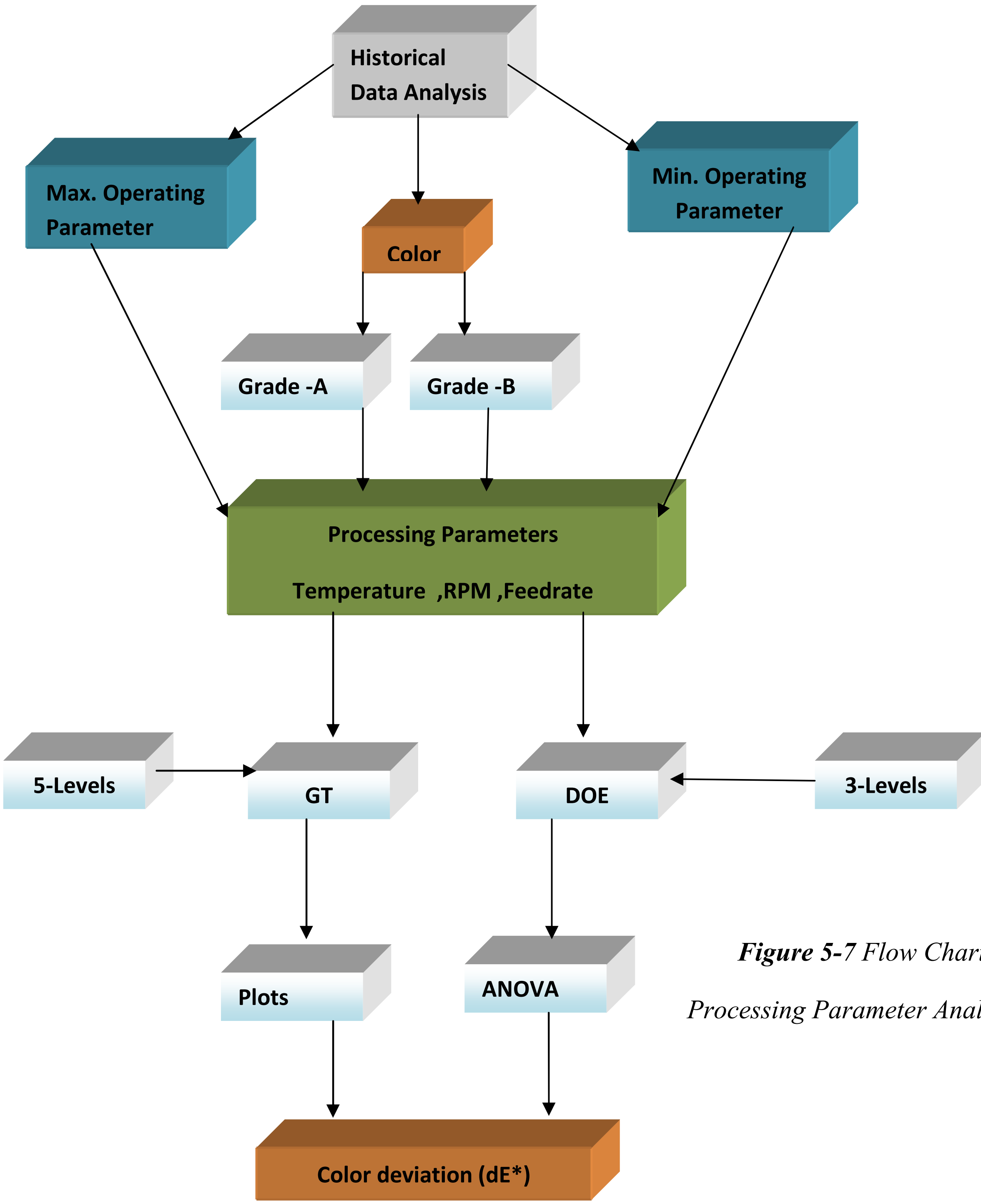


Figure 5-7 *Flow Chart for Processing Parameter Analysis*

5.4.1- ANOVA for L*

Table 5-9 ANOVA for L -Processing Parameter*

Source Terms	Sum of Squares (L*)	df (L*)	Mean Square (L*)	F-Value (L*)	p-value ,Prob > F (L*)
Model	6.168237	17	0.362837	10.28182	< 0.0001
A-Temp	0.483991	1	0.483991	13.71497	0.0007
B-RPM	0.044451	1	0.044451	1.259611	0.2692
C-Feedrate	0.000193	1	0.000193	0.005466	0.9415
D-Grade	4.951426	1	4.951426	140.3098	< 0.0001
AB	0.017334	1	0.017334	0.491209	0.4879
AC	0.104126	1	0.104126	2.950657	0.0944
AD	0.259675	1	0.259675	7.358482	0.0102
BC	0.019172	1	0.019172	0.543291	0.4658
BD	0.0325	1	0.0325	0.920963	0.3436
CD	0.071705	1	0.071705	2.031921	0.1626
A^2	0.012157	1	0.012157	0.34449	0.5609
B^2	0.003892	1	0.003892	0.110289	0.7417
C^2	0.086323	1	0.086323	2.446147	0.1266
ABC	0.000146	1	0.000146	0.004137	0.9491
ABD	0.007176	1	0.007176	0.203349	0.6547
ACD	0.067469	1	0.067469	1.911887	0.1753
BCD	0.006501	1	0.006501	0.184222	0.6703
Residual	1.270412	36	0.035289		
Cor Total	7.438649	53			

The Model F-value of 10.28 implies that the model is staticially significant. There is only a 0.01% chance that a "Model F-Value" this large could occur due to noise.Values of "Prob > F" less than 0.0500 indicate model terms are significant. For L*, A (temperature), D (grade) and AD (interaction between temperature and grade) are significant model terms.Similar results were obtained by other researchers while discussing color shift due to temperatures. For example, Apruzzese [8] shows that increasing temperature produces a darker product (i.e. L* increases with increase in temperature). p-Values greater than 0.1000 indicate that the model terms are not significant.

5.4.2- ANOVA for a*

Table 5-10 *ANOVA for a* -Processing Parameter*

Source Terms	Sum of Squares (a*)	df (a*)	Mean Square (a*)	F-Value (a*)	p-value ,Prob > F (a*)
Model	0.384597	17	0.022623	22.07308	< 0.0001
A-Temp	0.004011	1	0.004011	3.913552	0.0556
B-RPM	0.003633	1	0.003633	3.545038	0.0678
C-Feedrate	0.000411	1	0.000411	0.401187	0.5305
D-Grade	0.355537	1	0.355537	346.8896	< 0.0001
AB	0.000917	1	0.000917	0.894484	0.3506
AC	0.001001	1	0.001001	0.976694	0.3296
AD	0.007131	1	0.007131	6.957426	0.0122
BC	0.000817	1	0.000817	0.796804	0.3780
BD	0.001056	1	0.001056	1.03056	0.3168
CD	0.000883	1	0.000883	0.861924	0.3594
A^2	0.000395	1	0.000395	0.385854	0.5384
B^2	0.000938	1	0.000938	0.915476	0.3450
C^2	0.000998	1	0.000998	0.973896	0.3303
ABC	0.002025	1	0.002025	1.975747	0.1684
ABD	0.002501	1	0.002501	2.440211	0.1270
ACD	7.23E-05	1	7.23E-05	0.070579	0.7920
BCD	0.002269	1	0.002269	2.213343	0.1455
Residual	0.036897	36	0.001025		
Cor Total	0.421494	53			

The Model F-value of 22.07 implies that the model is significant. There is only a 0.01% chance that a "Model F-Value" this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicate model terms are significant. In case of a* , D (Grade) and AD (Interaction between temperature and Grade) are significant model terms.p-Values greater than 0.1000 indicate that the model terms are not significant.

5.4.3- ANOVA for b*

Table 5-11 *ANOVA for b* -Processing Parameter*

Source Terms	Sum of Squares (b*)	df (b*)	Mean Square (b*)	F-Value (b*)	p-value ,Prob > F (b*)
Model	6.190146	17	0.364126	31.56681	< 0.0001
A-Temp	0.119985	1	0.119985	10.40175	0.0027
B-RPM	0.005501	1	0.005501	0.476866	0.4943
C-Feedrate	0.000625	1	0.000625	0.054182	0.8173
D-Grade	5.946785	1	5.946785	515.5383	< 0.0001
AB	0.000301	1	0.000301	0.026098	0.8726
AC	0.006446	1	0.006446	0.558842	0.4596
AD	0.024457	1	0.024457	2.120267	0.1540
BC	0.013538	1	0.013538	1.173592	0.2859
BD	0.01028	1	0.01028	0.891168	0.3515
CD	0	1	0	0	1.0000
A^2	0.038849	1	0.038849	3.367877	0.0748
B^2	0.000788	1	0.000788	0.068286	0.7953
C^2	0.00019	1	0.00019	0.016491	0.8985
ABC	6.25E-06	1	6.25E-06	0.000542	0.9816
ABD	0.014917	1	0.014917	1.293165	0.2630
ACD	0.000474	1	0.000474	0.041098	0.8405
BCD	0.007004	1	0.007004	0.607205	0.4409
Residual	0.415264	36	0.011535		
Cor Total	6.60541	53			

The Model F-value of 31.57 implies that the model is significant. There is only a 0.01% chance that a "Model F-Value" this large could occur due to noise.Values of "Prob > F" less than 0.0500 indicate that model terms are significant. In this case A and D are significant model terms. Values greater than 0.1000 indicate the model terms are not significant.

5.4.4- ANOVA for dE*

Table 5-12 *ANOVA for dE* -Processing Parameter*

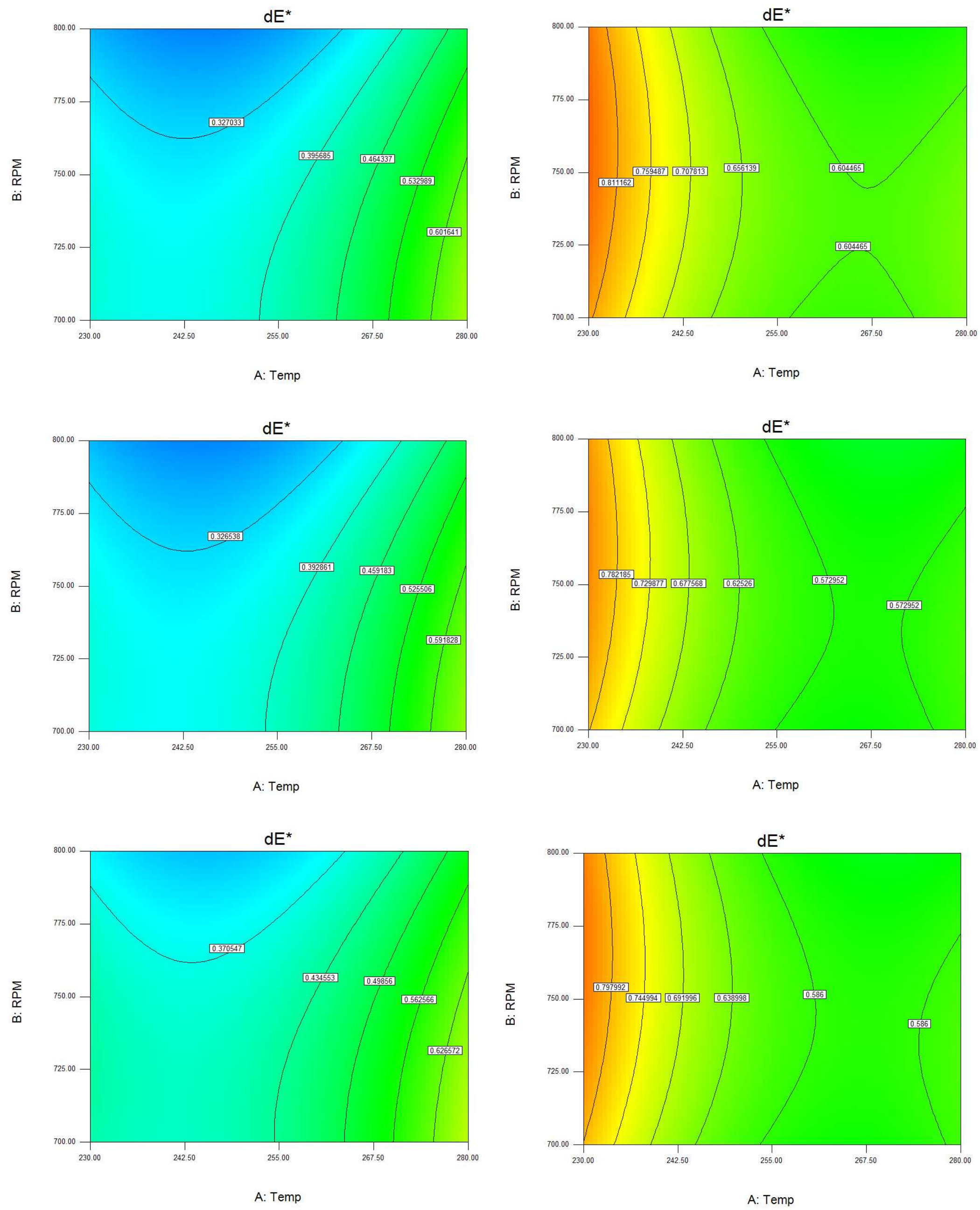
Source Terms	Sum of Squares-(dE*)	df-(dE*)	Mean Square-(dE*)	F-Value (dE*)	p-value ,Prob > F (dE*)
Model	1.463975	13	0.112613	6.927405	< 0.0001
A-Temp	1.52E-06	1	1.52E-06	9.35E-05	0.9923
B-RPM	0.038483	1	0.038483	2.367297	0.1318
C-Feedrate	0.000442	1	0.000442	0.027219	0.8698
D-Grade	0.695096	1	0.695096	42.75875	< 0.0001
AB	0.01191	1	0.01191	0.732614	0.3971
AC	0.000428	1	0.000428	0.026341	0.8719
AD	0.500627	1	0.500627	30.79603	< 0.0001
BC	0.000382	1	0.000382	0.023519	0.8789
BD	0.028439	1	0.028439	1.749392	0.1935
CD	0.007941	1	0.007941	0.488493	0.4886
A^2	0.163863	1	0.163863	10.08001	0.0029
B^2	0.010402	1	0.010402	0.639882	0.4285
C^2	0.00596	1	0.00596	0.366616	0.5483
Residual	0.650249	40	0.016256		
Cor Total	2.114224	53			

The Model F-value of 6.93 implies that the model is significant. There is only a 0.01% chance that a "Model F-Value" this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicate that model terms are significant.In this case D, AD and A^2 are significant model terms. Term A^2 represents nothing in actuallity, but it is used in quadratic curve fitting [5]. Figures 5-9, 5-10 and 5-11 show the interaction between different processing parameter at high, average and low levels.

a

b

c

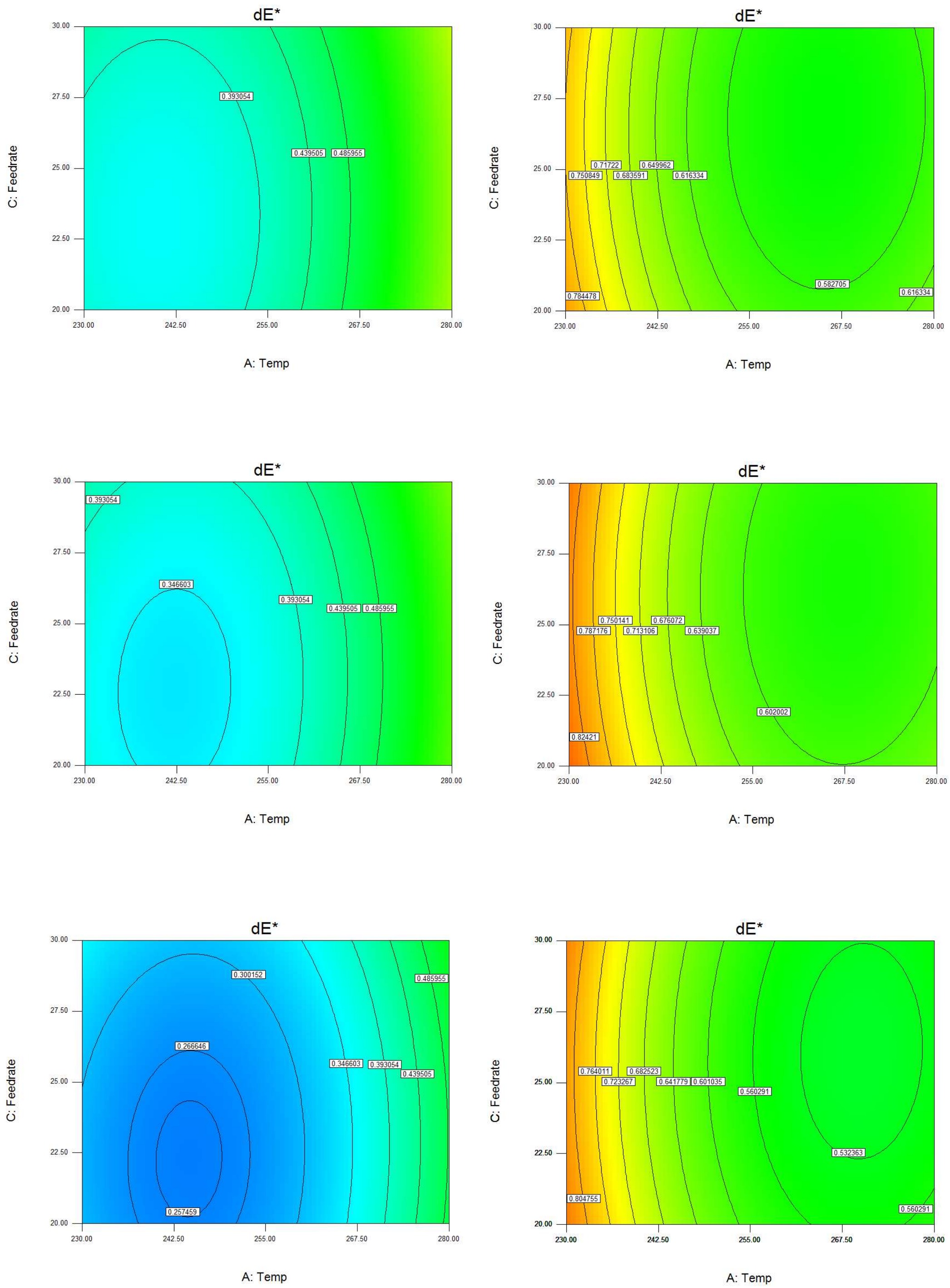


Grade G1 Grade G2

Figure 5-9 *Interaction between Temperature and rpm at a) FR=20 b) FR=25 c) FR=30 (kg/h)-dE* Measurement* a

b

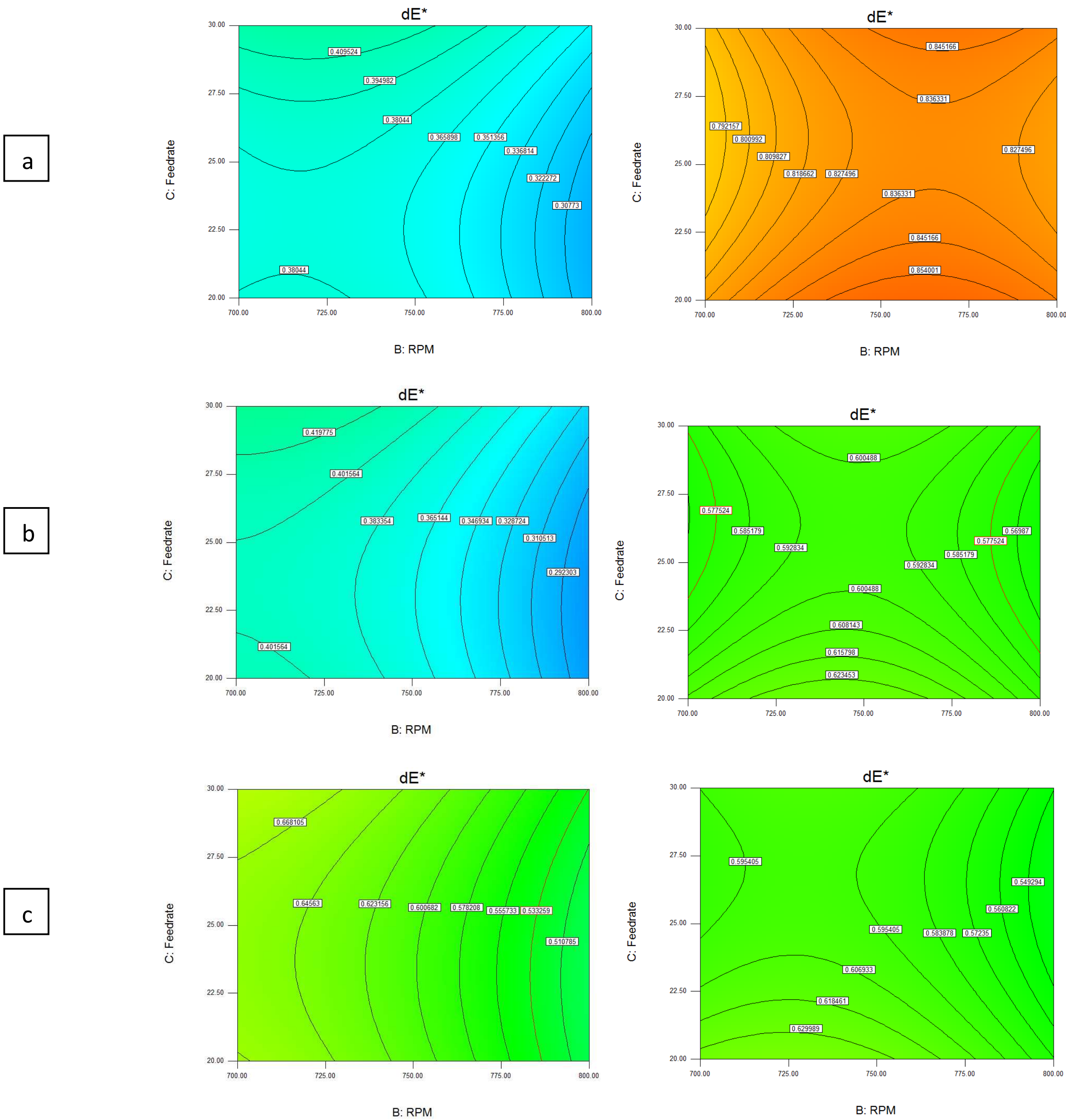
c



Grade G1

Grade G2

Figure 5-10 *Interaction between Temperature and Feedrate at a) rpm=700 b) rpm=750 c) rpm=800 dE* Measurement*



Grade G1 Grade G2

Figure 5-11 *Interaction between Feedrate and rpm at a) T=230C° b) T=255C° c) T=280C° dE* Measurement*

Process Parameter	Low	High	Avg
Temperature (C^o)	230	280	255
rpm	700	800	750
Feedrate (kg/h)	20	30	25

Grade G1: Large interaction between temperature and rpm is observed at low feed-rate when the temperature is low and the rpm is high. Decreasing rpm at low temperatures seems to increase the values of dE*. However at higher temperatures, the variation in dE* becomes less sensitive to rpm at low feed-rate, but it exhibits high values. This may be due to the degradation of either of the four pigments in a blend. IIo & Berghofer [9] have performed a similar study on food pigments and found similar results. However, it is important to mention here that different grades (resin+ pigments+ additives) behave differently due to differences in the nature of the blend. Pruzzese et al [8] discussed the effect of processing parameter on color in terms of measurement of L*. Results obtained by Sacchetti [58] also corroborate the present study. Another important aspect is the effect of feed-rate on dE*. High values of dE* are observed at low feed-rate and the acceptable color difference (dE*) window increases with increase in feed-rate. One of the possible reasons is that at low federate, material undergoes high shear and vice versa.

Grade G2: Quite different trends were observed for grade G2 than the trends of grade G1 for similar processing conditions. High interaction between temperature and rpm was observed at high temperatures and low feed-rate. Increasing the feed-rate increases the acceptable processing window of dE* in terms of temperature and rpm.

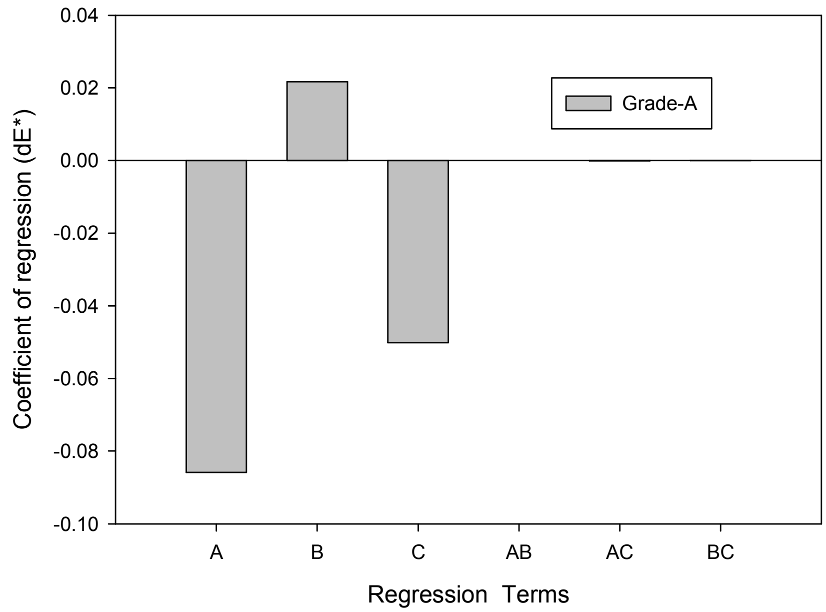


Figure 5-12 *Regression Coefficients for Grade-G1*

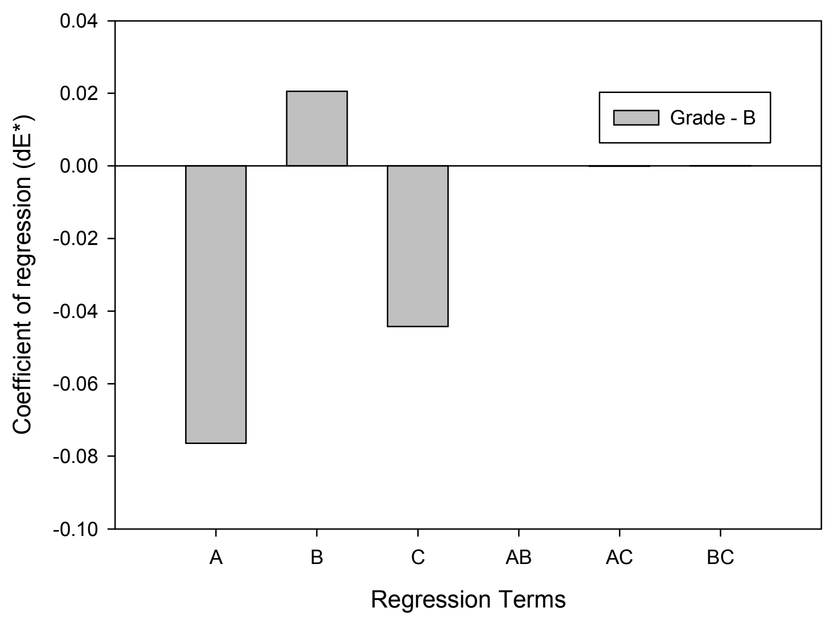


Figure 5-13 *Regression Coefficients for Grade-G2*

It is important to mention here that Grade G2 shows high values of dE* on all processing conditions as compared to Grade G1.

Figures 5-12 and 5-13 show the regression coefficients for processing parameters. A represents the temperature in ^{o}C. Column B represents the rpm and column C represents the feed-rate. Coefficient plots show that temperature is the most dominant factor effecting color difference. Feedrate is the 2nd most dominant observed parameter. Interactions between two parameters have negligibly small coefficients.

Note: The difference in the results of two grades actually reflects the effect of resins and additives

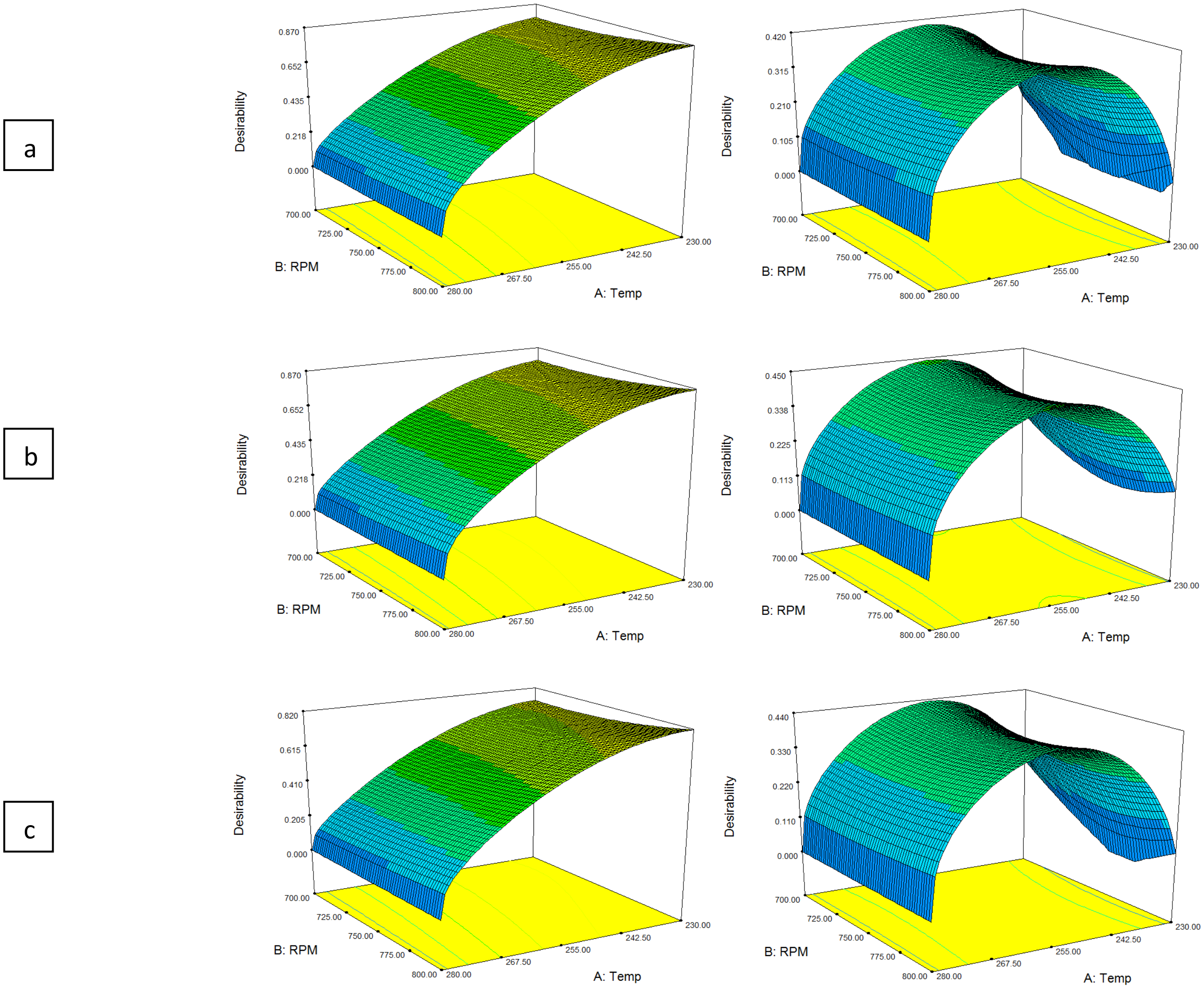
5.4.5- Optimization

Based on the above analysis, optimization was done using Design-Expert® Stat-Ease (version 7.1.6) in order to find optimum operating conditions. The goal was to find such processing conditions which minimize the dE* (i.e. the difference between the target and trial color). The dE* is listed in the following table for both of the grades. Desirability is how close the target is from the sample. Its value varies from 0 to 1, where 0 is not desirable and 1 is highly desirable. Stat-ease uses a method developed by Derringer and Suich. The details of this method are not the objective of the present study and can be found in literature. From the below Table it is clear that Grade-G1 has high desirability as compared to Grade-G2. It has been observed that desirability for Grade-G1 is high at low temperatures, high rpm and low feed rate. Similarly, desirability for Grade-

G2 is high at high rpm and at a temperature of 255°C with a feed-rate of approximately 25 kg/h. The effect of processing on high, average and low levels with respect to desirability is shown in Figure 5.14.

Table 5-13 *Optimal Operating Conditions*

Number	Temp	rpm	Feedrate	Grade	dE*	Desirability
1	230	799	22	Grade-G1	0.293186	0.872300702
2	230	799	20	Grade-G1	0.29636	0.869189876
3	230	799	26	Grade-G1	0.309473	0.856266552
4	230	783	20	Grade-G1	0.327399	0.838406267
5	230	700	23	Grade-G1	0.37429	0.790557932
6	230	700	23	Grade-G1	0.374373	0.790476838
7	230	700	21	Grade-G1	0.37585	0.788940913
8	230	700	26	Grade-G1	0.387875	0.776377181
9	255	799	26	Grade-G2	0.5616	0.444828762
10	250	700	26	Grade-G2	0.588236	0.444029188
11	251	700	26	Grade-G2	0.587898	0.444023145
12	252	799	28	Grade-G2	0.581136	0.441581796
13	250	700	28	Grade-G2	0.593809	0.44149582
14	254	799	29	Grade-G2	0.576748	0.436469569
15	254	800	22	Grade-G2	0.576776	0.434129631



Grade G1 Grade G2

a) FR=20 b) FR=25 c) FR=30 (kg/h)

Figure 5-14 *Desirability vs Processing Parameters*

5.5- Shear Rate

Shear rate is defined as the ratio between axial velocity and the distance between the screw and barrel surface. It is used in the viscosity calculation. Shear rate is measured in S^{-1}. Shear rate of the polymer flowing through an extruder with a diameter D moving with a speed N having channel depth d can be calculated as:

$Shear\ Rate = \frac{V}{d}$

where V is the axial velocity and d is the channel depth. Axial velocity can be found as

$$V = \frac{\pi DN}{60}$$

So,

$$Shear\ Rate = \pi * screw\ diameter\ (mm) * \frac{Screw\ Speed(rpm)}{channel\ depth(mm) * 60}$$

Shear rates in the extruder used at SABIC IP for experiments were calculated as:

D=25mm.

Channel depth = 4.5mm (for 100% degree of fill)

Screw to baffle clearance = 0.1mm

This shows us that shear rate is high when the distance between screw and barrel is small and minimum shear rate is observed at the center of the screw element where distance between barrel and screw surface is large.

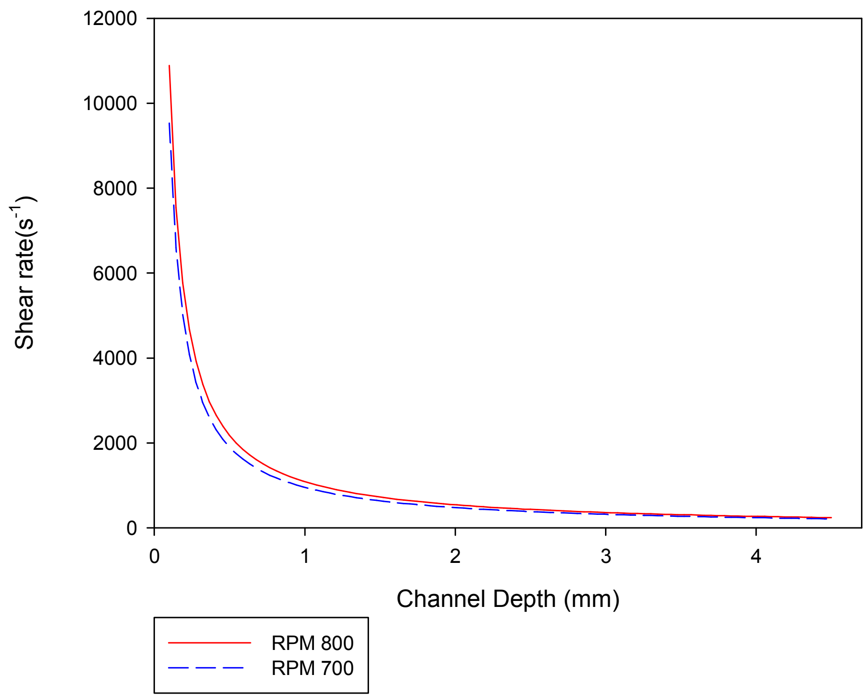


Figure 5-15 *Variation of shear rate with respect to channel depth for 100% Degree of fill*

5.6- Specific Mechanical Energy (SME)

Specific mechanical energy is defined as the ratio of drive motor power of the extruder to the throughput. It is measured in KWhr/kg. It is also abbreviated as SME.

$$\mathrm{SME} = \frac{\text{Drive motor power (KW)}}{FeedRate\ (\frac{kg}{h})}$$

Where drive motor power is a function of rpm (both actual and maximum), torque (load) and maximum KWs of motor.

$$\text{Specific Energy} = \frac{\left(\frac{\text{Actual Screw rpm}}{\text{Screw rpm}_{\text{max}}}\right) * \text{Maximum KW} * \frac{\%\text{Torque}}{100}}{FeedRate\ (\frac{kg}{h})}$$

SME for the DOE in the extruder used for experiments at SABIC IP was calculated as

$$\text{Maximum [kW]} = 28$$

$$\text{Screw rpm}_{\text{max}} = 1200$$

Table 5-14 *DOE for SME*

Run	Load (%)	SME-(kWh/kg)
1	56	0.326667
2	53	0.309167
3	54	0.4032
4	52	0.424667
5	50	0.4375
6	56	0.348444
7	52	0.339733
8	60	0.35
9	52	0.455
10	50	0.408333
11	56	0.49
12	53	0.329778
13	55	0.449167
14	59	0.367111
15	53	0.371
16	54	0.504
17	51	0.476
18	55	0.299444
19	56	0.365867
20	55	0.410667
21	53	0.288556
22	59	0.321222
23	52	0.388267
24	51	0.357
25	56	0.392
26	53	0.346267
27	50	0.466667

It is pertinent to mention here that temperature also affects SME. By increasing temperature, material becomes less viscous so it requires less motor power to move axially, hence load decreases which ultimately decrease SME for a given rpm and feed-rate. These statements are also verified by ANOVA.

Table 5-15 ANOVA for SME

Source Terms	Sum of Squares (dE*)	df (dE*)	Mean Square (dE*)	F-Value (dE*)	p-value ,Prob > F (dE*)
Model	0.093437	6	0.015573	136.9171	< 0.0001
A-Temp	0.005869	1	0.005869	51.6031	< 0.0001
B-rpm	0.011294	1	0.011294	99.29734	< 0.0001
C-F/R	0.076173	1	0.076173	669.7189	< 0.0001
AB	5.49E-07	1	5.49E-07	0.004827	0.9453
AC	3.28E-05	1	3.28E-05	0.288206	0.5973
BC	6.72E-05	1	6.72E-05	0.590484	0.4512
Residual	0.002275	20	0.000114		
Cor Total	0.095711	26			

The Model F-value of 136.92 implies the model is staticially significant. There is only a 0.01% chance that a "Model F-Value" this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicate model terms are significant. The analysis shows that A, B, C are significant model terms. The interaction given below shows that by increasing temperature, SME decreases which validates the hypothesis about the effect of temperature on SME. The interaction between Feed-rate and rpm and their mutual effect on SME at different temperatures is shown in figure 5-16.

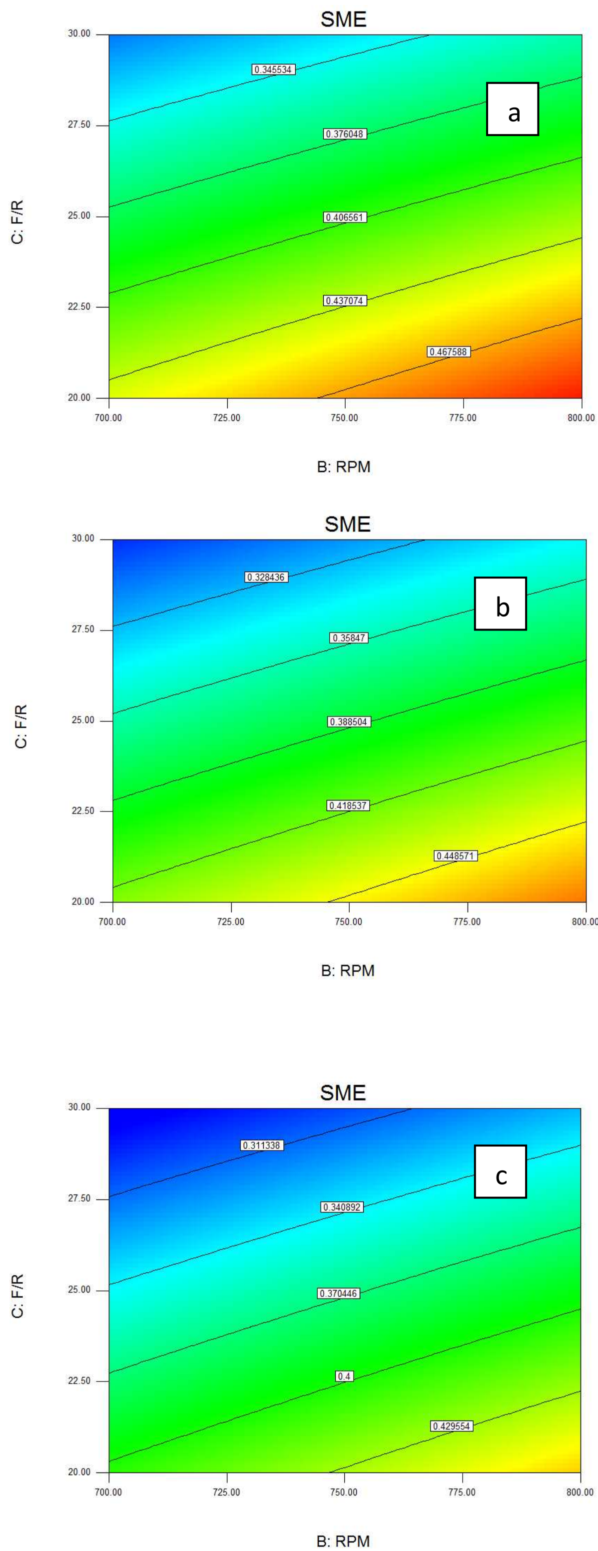


Figure: 5- 16 *Interaction between Feedrate and rpm at a) T=230C^o b) T=255C^o c) T=280C^o*

Similarly SME for GT experiments have also been calculated and presented below in tabular form.

__Table 5-16__ SME for GT

Level	Feed Rate (kg/h)	Specific Mechanical Energy (kWh/kg)
1	20	0.4328
2	23	0.3899
3	25	0.3850
4	27	0.3751
5	30	0.3609
Level	Temperature (Zone 3 ~ 10) (° C)	Specific Mechanical Energy (kWh/kg)
1	230	0.3789
2	240	0.3857
3	255	0.3850
4	270	0.3834
5	280	0.3883
Level	Screw rpm	Specific Mechanical Energy (kWh/kg)
1	700	0.3724
2	725	0.3789
3	750	0.3850
4	775	0.3978
5	800	0.4107

Chapter 6

Conclusions and Recommendations

6.1 Summary

In the present research, a number of problems causing color mismatch in the plastic industry have been studied. The whole project was divided into two different sets of study. The first group of study includes analysis of the two years production records data of SABIC IP. Two different sets of study have been performed under historical data analysis, which includes grade and production line analysis. In grade analysis, the objective was to study the effect of changing resin formulation on color and to study the pigment sensitivity in each grade. Two grades were selected for the study in which one grade was a blend of two polycarbonate resins and pigments the other was a blend of a single polycarbonate resin and pigments. In production line analysis, the objective was to study the effect of the production line (i.e. when same formulation was run on two different production lines having different screw diameters and configuration) on color and to study the pigment sensitivity in each line.

The second group of study was to investigate the effect of processing parameters on color. Three different processing parameters were chosen for the study which includes extruder temperature, screw rpm and feed-rate. Two different grades of the same color showing the highest number of adjustments in the historical records of SABIC IP were selected for study. Two different sets of experiments were performed which includes general trends and DOE. A lab scale extruder with a 26 mm screw diameter was used for the experiments.

6.2 Conclusions

For grade analysis, results show that pigments interact differently with different grades. Changing the amount of resin significantly affects pigment behaviour (i.e. pigment sensitivity) and ultimately, the output color of plastic on a given production line. The difference in the coefficients of figures 5-3 and 5-4 represents the effect of changing the amount of resin on color. In order to produce the right color, the interaction between resin blends and pigments must be understood prior to production.

For line analysis, results show that pigments behave differently under different production lines. This difference in behaviour of pigments is due to different shear rates in different production lines because of their different diameters and configuration. The difference in coefficients of the pigment PPH values in figures 5-5 and 5-6 actually represents the effect of changing screw configuration and diameter for a given formulation.

For processing parameters, results, shown in figure 5-12 and 5-13, show that temperature is the most influential factor. Increasing temperature reduces the color deviation for these particular highly adjusted grades. Similar results were observed for feed-rate, where increasing the feed-rate decreased the amount of color deviation. By increasing rpm, color deviation also increases, however the effect of rpm is not very significant as compared to temperature and feed-rate.

In general, there are hundreds of variables involved in color deviation, but proper understanding of formulation on a given production line and proper processing parameters leads to proper color production feed-rate.

6.3 Future Recommendation

Based on the performed analysis, the following are some future recommendations:

1. To obtain more historical data from SABIC IP and analyze with the current data in order to find the interactions between pigments and their cumulative effect on color.
2. More experiments should be carried out with other colors and on different grades.
3. New experiments should be designed in which range of rpm and feed-rate should be according to machine operating limits instead of SABIC records.
4. Effective shear rate calculations based on degree of fill in each screw element.
5. Screw configuration should be changed on the current machine to find the effect of screw configuration on color.
6. Perform test trials on actual production line.
7. Measurement and variation of residual time to see its effect on color.

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Appendix - A

Properties of polycarbonates

Table A-1 Properties of Polycarbonates [63]

	Units	
Density	Kg/m3	1190 - 1210
Price	CAD/kg	4.83 – 5.313
CO_2 Production	Kg/kg	3.92 – 4.34
Energy production	MJ/kg	101 – 112
Recycle fraction		0.45 – 0.55
Oxygen Index		24 – 26 %
Water Absorption		0.135 – 0.165 %

Composition

$(O\text{-}(C_6h_4)\text{-}C(CH_3)_2\text{-}(C_6H_4)\text{-}CO)_n$: :

Table A-2 Composition of polycarbonates [63]

Polymer Class	Thermoplastic :Amorphous
Polymer Type	PC
%filler	0
Filler Type	Unfilled

Mechanical Properties

Table A-3 Mechanical Properties of polycarbonates [63]

	Units	
Bulk modulus	GPa	3.834 – 4.026
Compressive modulus	GPa	2.35 – 2.47
Compressive strength	MPa	69 – 86.2
Elongation	%	110 – 120
Elastic Limit	MPa	59.1 – 65.2
Endurance Limit	MPa	23.7 – 30.8
Flexural modulus	GPa	2.27 – 2.34
Fracture Toughness	$MPa.m^{1/2}$	2.1 – 2.3
Hardness-Vickers	HV	17.7 – 19.6
Hardness-Rockwell M		70 – 75
Hardness – Rockwell R		104 – 115
Izod Toughness	KJ/m^2	63 – 95
Loss Coefficient		0.01639 – 0.01724
Modulus of Rupture	MPa	86.2 – 93.1
Poisson ratio		0.3912 – 0.407
Shape Factor		4.6
Shear Modulus	GPa	0.8291 – 0.872
Tensile Strength	MPa	62.7 – 72.4
Young Modulus	GPa	2.32 – 2.44

Thermal Properties

Table A-4 Thermal Properties of polycarbonates [63]

	Units	
Glass Temperature	^oC	142 - 158
Heat deflection Temperature	^oC	138 – 142
Heat deflection Temperature	^oC	121 – 132
Maximum Service Temperature	^oC	104 – 119
Minimum Service temperature	^oC	-43 – 7
Specific Heat	J/kg.K	1535 – 1596
Thermal Conductivity	W/m.K	0.189 – 0.205
Thermal Expansion	µstrain/ ^oC	120.1 – 124.9

Processing

3. *Table A-5 Processing properties of polycarbonates [63]*

	Units	
Linear mould Shrinkage	Mm/mm	5e-3 – 7e-3
Moulding Pressure Range	MPa	69 - 138
Processing Temp (Compression)	^oC	246 - 302
Processing temperature (Extrusion)	^oC	226 – 282
Processing Temperature (Injection)	^oC	266 - 322

Electrical

Table A-6 Electrical Properties of polycarbonates [63]

	Units	
Breakdown Potential	MV/m	15.98 - 19.17
Dielectric constant		3.1 – 3.3
Dissipation Factor		8.6e-4 – 9.4e-4
Resistivity	μΩ.cm	1e20 – 1e21

Durability

Table A-7 Durability of polycarbonates [63]

Flammability	Good
Fresh Water	Very Good
Organic Solvents	Average
Oxidation at 500C	Very Poor
Sea Water	Very Good
Strong Acid	Very Good
Strong Alkalis	Poor
UV	Good

Wear	Average
Weak Acid	Very Good
Weak Alkalis	Good