***“EFFECT OF PARTICLE SIZE OF CALCIUM CARBONATE ON POLYPROPYLENE COMPOSITES”***

*A MAJOR PROJECT-II REPORT SUBMITTED IN THE PARTIAL*

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**RAVI NARAIAN PANDEY.**

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**DEPARTMENT OF APPLIED CHEMISTRY & POLYMER TECHNOLOGY**

**DELHI TECHNOLOGICAL UNIVERSITY,**

**BAWANA ROAD, DELHI-110042.**

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**CERTIFICATE**

This is to certify that the dissertation entitled *“****EFFECT OF PROCESSING PARAMETERS ON THE MECHANICAL PROPERTIES OF POLYPROPYLENE”*** has been submitted by ***Ravi Naraian Pandey*** as partial fulfillment for the degree of **Master of Technology in Polymer Technology, Delhi Technological University, Delhi**. This is a record of his own work carried out by him under my supervision and guidance. The matter embodied in this major thesis report has not been submitted for the award of any other degree or diploma.

 **Prof. G. L. Verma**

 Head, Department of Applied Chemistry &Polymer Technology,

 Delhi Technological University, Delhi-110042.

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*First of all I pray to “****GYANDAYINI MATA SARASWATI”*** *with deep of my heart without whose grace nothing is possible for me.*

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*I feel pleasure in submitting my major project-II entitled* ***“*EFFECT OF PROCESSING PARAMETER ON THE MECHANICAL PROPERTIES OF POLYPROPYLENE*”*** *under the esteemed guidance and supervision of* **Prof. (Dr.) G. L. VERMA *.***

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**Place: Delhi**

**Dated:16/07/12 *RAVI NARAIAN PANDEY***

***Namami shami shana nirvana rupam, vibhum vyapakam brahma veda swarupam.***

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**ABSTRACT**

A polymer is rarely used as a pure material and the baseline physical, chemical and rheological properties such as molecular weight, strength, stiffness and viscosity are often modified by the addition by the fillers or by blending with another polymer.

However, as many polymers are immiscible compatiblisation and graft processing polymer blends are very important technique to increase miscibility of the blends as well as improve to chemical, physical and mechanical properties.

Reactive extrusion or melt processing is one of the most appropriate techniques for improving polymer properties. Compatiblisation and graft polymer processing are often carried out under reactive extrusion conditions. This technique is an efficient approach because it is easy, inexpensive and short processing time. Although reactive extrusion has numerous advantages one of the limitations is degradation of the polymer under the high temperatures and mechanical stresses encountered.

In the polymer industry , because of increasing customer demand for improved product quality ,optimizing the polymerization process by decreasing product costs and controlling the reactions during the polymerization has become more important. It can be said that any method used for monitoring the polymerization process has to be fast, accurate and reliable Both online and inline process may be involved in in process monitoring. The primary information from in- process monitoring is used for identifying and understanding molecular structure and changes, optimizing and improving process modeling and understanding whether the process is under control. This also involves considering whether the products have the required properties.

Talc filled polypropylene composites are designed to improve the properties and to lower the overall cost of engineering plastics. In this study the effects of adding talc with variable particle size on the mechanical and thermal properties of polypropylene was investigated. A composite of Polypropylene with constant concentrations (24%weights) of talc was prepared on twin screw extruder (Co-rotating). The composite showed improved mechanical as well as thermal properties on addition of different size of filler. It is also observed that mechanical properties as well as thermal properties increase with decrease in particle size.

 **CHAPTER 1**

 **INTRODUCTION**

1.1Introduction

Polymer science or macromolecular science is the subfield of material science concerned with polymers, primarily synthetic polymers such as plastics. The field of polymer science includes researchers in multiple disciplines including chemistry (pure and applied), physics, and engineering.

This science comprises three main sub-disciplines:

* Polymer chemistry or macromolecular chemistry, concerned with the chemical synthesis and chemical properties of polymers.
* Polymer physics, concerned with the bulk properties of polymer materials and engineering applications.
* Polymer characterization is concerned with the analysis of chemical structure and morphology and the determination of physical properties in relation to compositional and structural parameters.

Particle filled polymer composites have become attractive because of their wide applications and low cost. Incorporating inorganic mineral fillers into plastic resin improves various physical properties of the materials such as mechanical strength, modulus and heat deflection temperature.

In general the mechanical properties of particulate filled polymer composites depend strongly on size, shape and distribution of filler particles in the matrix polymer and good adhesion at the interface surface. Polypropylene (pp) is among the most widely used engineering thermoplastics in automobile (car bumper, dash board), home appliances, and packaging and consumer applications because of its excellent mechanical properties.

In this investigation Talc of variable particle size is added to PP. Influences of the addition of these fillers on the mechanical and thermal properties are examined.

Such studies can lead to many benefits of which the following are probably the most important (Brydson, 1981).

(a)To understand processing faults and defects which are of rheological origin and hence to make logical suggestions for adjusting the processing conditions for either minimizing or completely removing the fault.

(b)To make a prior intelligent selection of the best polymer or polymer compound to use under a given set of circumstances.

 (c)They can lead to qualitative and to some extent quantitative, relationships between such factors as output, power consumption, machine dimensions, material properties and operational variables such as temperatures and pressure.

Since there are so many development in polymer types and grades, in addition with fact that most of the processing occur in the molten state, better understanding of the rheological characteristic of polymer systems is beneficial to the design of polymer processing equipment, with the ability for predicting the energy requirement, optimizing the processing conditions and correlation with the structural development (Hornsby, 1999; Han, 1976).

**1.2 Polymer Composites**

Composites materials are formed by combining two or more materials that have quite different properties. The different materials work together to give the composite unique properties. Composites represent an important class of engineering material. New requirement and new product has led to drive for more and better polymer composites.

Lightweight high performance engineering plastics had replaced metals in many applications as polymers are relatively cheap and large volume structural materials (Sheldon, 1982). The wide application of polymer composites ranges from the manufacturing of high tech engineering structures such as tanks, pipes, aircraft interior

furnishings and support beams, to the making of leisure and sporting items such as golf

clubs and balls, skis, racquets and boats. Basically, polymer composites may be classified into the following categories (Alger, 1990):

* Polymer-polymer combinations (polymer blends)
* Polymer. gas combinations (expanded, cellular or foamed polymers)
* Polymer. rigid filler combinations of:
* Polymer-fiber
* Polymer-particulate filler
* Polymer-hybrid filler

In these types of composites, the intention is to obtain some enhancement of one or

more of the mechanical properties, i.e. reinforcements, although cheap fillers are primarily used as diluents, to reduce cost without much effect on the properties. For polymer particulate filler, an improvement of stiffness or wear resistance is the main objective. Using the above classification, the present study comes under the polymer hybrid filler category, which the polymer matrix is polypropylene while calcium carbonate is combined in different compositions to form a fillers systems. A dispersion agent is also added in CaCO3 to enhance the dispersion of the filler.

Historically, thermoplastic polymers have been used extensively as matrix material for injection molded and short fiber reinforced composites. In the early 1980.s, polymer chemist developed advanced thermoplastic matrices suitable for higher performance composites. The result is a new class of composites with short processing time, damage tolerance superior to that of traditional thermosetting polymer matrix composites and an adequate solvent resistance (Miller, 1996).

At this moment, using of this mixed fiber and particulate composites has been recognized for many years. Growth of hybrid systems has been particularly rapid in the last decade or so, encouraged by the demand for high performance engineering materials, especially where low weights is great importance. Although individual classes of fillers or fibers can contribute some desirable properties, the real interest in composites is in optimizing the different contributions from different types of fillers.

Turning to specific applications, polypropylene composites has the potential to be used in automotive applications such as door trims, bumpers, panels and interior furnishings.

**1.3 Polymer Nanocomposites:**

A **nanocomposite** is a multiphase solid material where one of the phases has one, two

 or three dimensions of less than 100 [nanometers](http://en.wikipedia.org/wiki/Nanometers) (nm), or structures having nano-

scale repeat distances between the different phases that make up the material.[[1]](http://en.wikipedia.org/wiki/Nanocomposite#cite_note-ajayan-0) In the broadest sense this definition can include porous media, [colloids](http://en.wikipedia.org/wiki/Colloids), [gels](http://en.wikipedia.org/wiki/Gels) and [copolymers](http://en.wikipedia.org/wiki/Copolymers), but is more usually taken to mean the solid combination of a bulk matrix and nano-dimensional phase(s) differing in properties due to dissimilarities in structure and chemistry. The mechanical, electrical, thermal, optical, electrochemical, catalytic properties of the nanocomposite will differ markedly from that of the component materials. Size limits for these effects have been proposed,[[2]](http://en.wikipedia.org/wiki/Nanocomposite#cite_note-kamigaito1991-1) <5 nm for [catalytic](http://en.wikipedia.org/wiki/Catalytic) activity, <20 nm for making a hard magnetic material soft, <50 nm for [refractive index](http://en.wikipedia.org/wiki/Refractive_index) changes, and <100 nm for achieving [superparamagnetism](http://en.wikipedia.org/wiki/Superparamagnetism%22%20%5Co%20%22Superparamagnetism), mechanical strengthening or restricting matrix [dislocation](http://en.wikipedia.org/wiki/Dislocation) movement .

**1.3.1 NANOCOMPOSITE STRUCTURE**

In general, the structure of polymer/clay nanocomposites is classified according to the level of intercalation and exfoliation of polymer chains into the clay galleries.
Various parameters including clay nature, organic modifier, polymer matrix and method of preparation are effective.

**1.3.1.1PHASE SEPARATED STRUCTURE.**

When the organic polymer is interacted with inorganic clay(unmodified),the polymer is unable to intercalate within the clay layers and the clay is dispersed as aggregates or particles with layers stacked together within the polymer matrix. The obtained composite structure is considered as “Phase separated”.

**1.3.1.2 INTERCALATED STRUCTURE:**

When one or more polymer chains are inserted into the inter layer space and cause to the increasing of the inter layer spacing, but the periodic array of the clay layer is still exist, the intercalated nanocomposite is formed.

**1.3.1.3 EXFOLATED STRUCTURE:**

Exfoliated or delaminated structure is obtained when the insertion of polymer chains into the clay galleries causes to the separation of the layers one another's and individual layers are dispersed within the polymer matrix.

**1.4 EXTRUSION TECHNOLOGY:**

Extrusion is one the most versatile processes, it converts a plastics material into product

of the specific cross sections by forcing the material through an orifice called die under

controlled conditions. There are specific requirements that must be satisfied concerning

both equipment and the raw material. The equipment must be capable of providing

sufficient heat to soften the material, sufficient mixing actions to homogenize the melt

and then sufficient pressure to force the melt through filter pad and die.

Extrusion may be continuous (theoretically producing indefinitely long material) or semi-

continuous (producing many pieces). The extrusion process can be done with the

material hot or cold.

Commonly extruded materials include [metals](http://en.wikipedia.org/wiki/Metal), [polymers](http://en.wikipedia.org/wiki/Polymer), [ceramics](http://en.wikipedia.org/wiki/Ceramic), [concrete](http://en.wikipedia.org/wiki/Concrete) and

foodstuffs.

Hollow cavities within extruded material cannot be produced using a simple flat

extrusion die, because there would be no way to support the center barrier of the die.

Instead, the die assumes the shape of a block with depth, beginning first with a shape

profile that supports the center section. The die shape then internally changes along its

 length into the final shape, with the suspended center pieces supported from the back

 of the die.

**1.4.1HISTORY**

In 1797, [Joseph Bramah](http://en.wikipedia.org/wiki/Joseph_Bramah) patented the first extrusion process for making lead pipe. It

Involved preheating the metal and then forcing it through a die via a hand driven

Plunger. The process wasn't developed until 1820 when Thomas Burr constructed the

 First hydraulic powered press. At this time the process was called squirting. In 1894,

Alexander Dick expanded the extrusion process to copper and brass alloys.

**1.4.2 PROCESS**

The process begins by heating the stock material (for hot or warm extrusion). It is then

 loaded into the container in the press. A dummy block is placed behind it where the

ram then presses on the material to push it out of the die. Afterward the extrusion is

stretched in order to straighten it. If better properties are required then it may be heat treated or cold works.

The extrusion ratio is defined as the starting cross-sectional area divided by the cross-

sectional area of the final extrusion. One of the main advantages of the extrusion

process is that this ratio can be very large while still producing quality parts.

**1.4.3 FUNDAMENTALS OF EXTRUSIONS**

**1.4.3.1PRINCIPLES**

Although the solid state extrusion is known, in the everyday language extrusion is a melt

deformation process in which the following operations situation takes place:

The required materials (polymers, additives) are fed through a feed hopper into a barrel

The polymeric materials soften by the heat from the external heaters and generated by frictional forces.

The energy needed to convert the cold particle into a hot viscous melt depends on the

 specific heat, heat of melting, the maximum temperature of the extrusion.

The transfer of heat into individual pellets by thermal conductivity.

Thermal conduction is important in removing the heat from the melt in order to solidify it.

The pressure required for the continuous extrusion can be generated by the extruder screw.

 **1.4.4 CLASIFICATION**

**1.4.4.1Batch –type**

* Ram extruder
* Reciprocating screw extruder

**1.4.4.2 Continuous-Type**

**1.4.4.2.1 Screw less extruder**

* Disk extruders
* Drum extruders
* Other extruders

**1.4.4.3 Screw extruder**

* Single screw extruder
* Twin screw extruder
* Multiple screw extruders.

**1.4.4.4 MELT EXTRUSION: SINGLE SCREW**

In injection molding the purpose of the screw extruder is simply to obtain a melt, a

Dedicated extrusion machine works on the principle but also must mix, homogenize and

Melt the material.

Higher back pressures may be generated in single screw extrusion machines compared

to injection molding machines and the screws may be longer for better mixing

**** Fig: 1.1 single screw extruder.

(Source: internet)

1.2.5 SCREW DESIGN

There are five possible zones in a thermoplastic screw. Since terminology is not standardized in the industry, different names may refer to these zones. Different types

of polymer will have differing screw designs, some not incorporating all of the possible

zones.

Fig 1.2: A simple plastic extrusion screw

(Source: internet)

Most screws have these three zones:

* Feed zone. Also called solids conveying. This zone feeds the resin into the

extruder, and the channel depth is usually the same throughout the zone.

* Melting zone. Also called the transition or compression zone. Most of the resin is

 melted in this section, and the channel depth gets progressively smaller.

* Metering zone. Also called melt conveying. This zone, in which channel depth is

again the same throughout the zone, melts the last particles and mixes to a uniform

temperature and composition.

In addition, a vented (two-stage) screw will have:

* Decompression zone. In this zone, about two-thirds down the screw, the channel

suddenly getsn deeper, which relieves the pressure and allows any trapped gases

(usually moisture or air) to be drawn out by vacuum.

* Second metering zone. This zone is like the first metering zone, but with greater channel depth, and repressurizes the melt to get it through the resistance of the screens and the die.

Often screw length is referenced to its diameter as L:D ratio. For instance, a 6-inch

(150 mm) diameter screw at 24:1 will be 144 inches (12 ft) long, and at 32:1 it is

192 inches (16 ft) long. An L: D ratio of 24:1 is common, but some machines go up to

32:1 for more mixing and more output at the same screw diameter. Two-stage (vented)

screws are typically 36:1 to account for the two extra zones. Each zone is equipped

with one or more [thermocouples](http://en.wikipedia.org/wiki/Thermocouple) or [RTDs](http://en.wikipedia.org/wiki/Resistance_temperature_detector) in the barrel wall for temperature control.

**1.5 Problem statement**

For years, fillers have been used extensively to improve the mechanical properties of polymeric material. Besides the increment obtained in stiffness, hardness, abrasion resistance, and reduced cost of the filled material, the addition of filler to polymer also modify their flow behavior and consequently their process ability.

The vast resources of CaCO3 in Malaysia have driven Malaysia Plastic Manufacturers to switch to use CaCO3 as a cheaper alternative to fill PP composites. In fact, CaCO3 is five times cheaper than talc and it takes an overwhelming proportion of the filler market in plastics, although in PP it occupies second place behind talc. Study on the hybrid filled PP composites of talc and CaCO3 had been conducted by Leong *et al.* (2001) in order to get a balance material properties and cost.

It has been well documented that the general behavior of adding fillers to polymers will increase the viscosity and decrease melt elasticity of the polymer.

Such a theoretical study will be useful for designing better processing equipment and determining optimal processing conditions. Thus, the effect of filler loading, type and treatment of the filler will be investigated with regards to the flow behavior and as well the extrudate swell and melt fracture phenomena of the composites.

**1.6 Objectives of Study**

The primary objective of this study is:

(a)To investigate the effect of different filler loading/ratio, temperature and pressure on the melt flow system of the filler composites, which able to eliminate processing fault and defect.

(b)To study for the establishment of better processing conditions and to develop optimum morphology to maximize products performance.

(c)To observe the effect of stearic acid treated CaCO3 on the flow behaviors and melt elasticity of the PP hybrid composites.

**CHAPTER 2**

 **LITERATURE REVIEW**

**Introduction**

Filled polymer composites have continued to attract interest from researchers due to inherent benefits from working with polymers, which are ease of process ability and productivity, combined with the addition of filler, which from the reinforcement it supplies, can significantly alter the base polymer properties resulting in a low-cost composite material with potentially very useful properties (Thio *et al.* 2002; Da Silva *et al.* 2002).Nevertheless, incorporation of filler in a polymer will affect the melt rheological behavior of this filled composites systems and it is noted that this behavior will be critically important in defining the processability of the composite.

**2.1 Polypropylene Composites.**

Polypropylene (PP) composites have been used in large quantities in numerous fields of applications for many years (Pukanzsky, 1995). The success of PP composites lies in its extremely advantageous price/volume/performance relations, with the result that PP composites successfully penetrate fields traditionally occupied by other engineering plastic material such as ABS and nylon. Considerable efforts have been made to extend the application of PP composites to fields where engineering thermoplastic have been used up to now. Today, particulate filled PP composites such as talc-filled PP is still widely used.

Particularly in the automotive industry for the production of bumpers, heater housings, door pockets and trimmings, timing belt covers cladding. Other common fillers include CaCO3 kaolin, mica and carbon black, while glass fiber is still one of the most commonly used fibrous reinforcements in PP.

**2.1.1 Polypropylene**

Polypropylene (PP) is a versatile thermoplastics offering a useful balance of heat (160°C) and chemical resistance, good mechanical and electrical properties, and processing ease. Besides PE and PVC, PP is the third commodity polymer produced and applied in large quantities. Crystalline polymers of propylene were first described in the literature in 1954 by G. Natta and his associates at the Chimica Industriale del Politechico di Milano (Hanna, 1990). Earlier efforts to initiate propylene polymerization had only resulted in noncrsytalline polymers of little or no importance. With the introduction of heterogenous, stereospecific catalyst discovered by K. Ziegler for the low-pressure polymerization of ethylene, the scene suddenly changed. These reactions are products of transition metal compounds with selected organometallic compounds contained active sites for

polymerization, such that each new propylene molecule was incorporated in the polymer chain in a regular, geometric manner identical to all preceding methyl groups. Three geometric forms of the polymer chain can be obtained (Figure 2.1). Natta classified them as:

* Isotactic

All methyl groups aligned on one side of the chain

* Syndiotactic

Methyl groups alternating

* Atactic

Methyl groups randomly positioned



 Figure 2.1: Polypropylene structures (a) Isotactic, (b) Syndiotactic, (c) Atactic.

Both isotactic and syndiotactic forms will crystallize when there are cooled from molten states. Commercial injection molding and extrusion grade PP are generally 94 to 97% isotactic. Fabricated parts are typically 60% crystalline, with a range of polyhedral spherulite forms and sizes, depending on the particular mode of crystallization from the melt. Atactic PP, though is not suited to structural plastic uses, have been developed as modifiers in hot melt adhesives, roofing compounds, and communications cable- filler gels. PP can be made into a multidimensional range of products with properties and characteristics interdependent on the

• Type of polymer: homopolymer, random, or block copolymer

• Molecular weight and molecular weight distribution

• Morphology and crystalline structure.

• Additives

• Fillers and reinforcing materials

• Fabrication techniques

Homopolymer have resistance to deformation at elevated temperatures, while high stiffness, tensile strength, surface hardness and good toughness can be observed at ambient temperatures. Random ethylene-propylene copolymers are characterized by higher melt strengths. They have good clarity and resistance to impact at low temperature, gained at some sacrifice in stiffness, tensile strength and hardness. Block copolymers, preferably with ethylene, are classified as having medium, high, or extra-high impact strength with particular respect to sub-zero temperatures. Some properties that are usually considered inherent advantages of PP are (Hanna, 1990):

• Low specific gravity (density)

• Excellent chemical resistance

• High melting point (relative to volume plastics)

• Good stiffness/toughness balance

• Adaptability to many converting methods

• Great range of special purpose grades

• Excellent dielectric properties

• Low cost (per unit volume)

Properties usually considered being disadvantages of PP are:

• Flammability

• Low temperatures brittleness

• Moderate stiffness

• Difficult printing, painting and gluing

• Low UV resistance

• Reduced extruder output

• Haziness

• Low melt strength

**2.1.2 Filled Polypropylene**

Throughout the years, research has been intensively going on which various publications on the use of PP as the main matrix, coupled with various types of modified as well as unmodified fillers and reinforcements could be found in abundance in almost every polymer and composite journal. Comprehensive studies, which concentrate on a particular property of filled PP, have been done. For example, Wang & Wang (1999) have carried out some works on the shear yield behaviour of CaCO3-filled PP, which involved conducting steady shear test, a transient stress growth test, and a dynamic oscillatory shear flow. The magnitude of the yield stress of polymer melts, which is defined as any critical stress below which no flow can be observed under the condition of experimentation, was found to be closely associated with the thixotropy breakdown and recovery of the microstructure inside the compounds, thus the characteristic time of this transient process plays a part in yield stress measurement. In other words, yield behavior in highly filled (thixotropic) materials has been determined to be a function of structure and hence time.

Goel (1980) have conducted an in depth study on the effect of polymeric additives

on the rheological properties of talc filled PP. It was found that talc does not influence the viscosity of PP but decreases the elasticity of the system. Addition of polypropylene oxide as an oligomer had not only decreased the viscosity but also further decreased the elasticity of the filled polymer composite considerably. The impact properties of filled PP composites were also tackled by several workers such as Jilken (1991) and Wang and Huang (1996) they have investigated the effect of filler particle size, and had concluded that an increase in impact strength for mineral-filled PP can be obtained by decreasing the particle size of the filler particles. Wang and Huang (1996) on the other hand, had considered processing factors that could influence the state of particle dispersion and hence the impact properties of PP. The experimental results have indicated that good dispersion of filler particle would improve the impact properties of the polymer matrix, but this only occur at moderate filler loading.

Investigation conducted by Pukanszky *et al.* (1994) have suggested that orientation of anisotropic filer particle occurs even under mild shearing condition and the degree of orientation depends on the filler content of the composite. Particle orientation decreases with increasing filler content due to hindrance of rotation caused by particles interfering with each other. A comparison of nucleating ability between talc and CaCO3 has also been done, which resulted in talc being a stronger nucleator. However, it was found that the effect of nucleation on the mechanical properties of the composites is minimal as compared to the effect of filler particle orientation. In failure of particulate-filled composites, the main energy-consuming process is plastic deformation of the matrix, which is modified by the presence of fillers (Pukanszky *et al*. 1994).

**2.1.3 Fillers, Extenders and Reinforcements**

Fillers, as the name implies, have commonly been employed to cheapen or extend a product with an evident change or modification of the properties of the unfilled materials such as hardness, rigidity, viscosity or colour. Their use has grown dramatically in recent years, encouraged by the growing demand for high performance plastics and increasing polymer prices. Gradually, the realization grew that by the selective use of fillers. Certain properties of the unfilled material could be enhanced or even exceeded and reinforcement of properties was possible. Hence fillers are today employed to obtain any, poor combination of the following effects (Whission, 1971):

• Add rigidity, stiffness and hardness.

• Regulate thermal expansion and shrinkage

• Improve heat resistance.

• Improve or regulate electrical characteristics.

• Increase strength and reduce creep.

• Modify rheological properties

• Aid processability (lubrication, flow, mixing and dispersion)

• Modify appearance (opacity, colour, texture)

• Alter density and bulk

As mentioned above, the term .filler. is used to carry with it the implication of a cheap additive to an expensive material but it must be emphasized that nowadays, it is note the case with plastic materials. The filler is incorporated with the polymer to give a plastic material in which both the polymer and the filler contribute to the desirable properties of the plastic material. Often, as for example in CaCO3 filled polypropylene, the filler is much cheaper than the polymer, but fillers such as nano-particulate clays, are much more expensive than the polymer with which it is incorporated. Naturally enough, factors such as particle size will play an important role, since these influence the ease with which it will be bound in the mix together with other components. There is often a .trade off. between the uses of particular filler and its side effect on other properties. There is a movement towards development of multifunctional fillers, which will make useful contribution to performance in other directions as well as lowering cost (Murphy, 1996).

**2.2 Mineral Fillers**

Nowadays, filler play an important role in the plastic industry. In the course of time, these basic raw materials have been refined and purified and today, it becomes the source from which most of the so-called inorganic fillers are derived. The purpose of their use is not confined to cost reduction but also to improve mechanical performance such as rigidity, dimensional stability toughness and as well also change the viscosity of the filled material. The level of such improvement or changing depends significantly upon type, size and shape, content and surface treatment of the fillers (Tebtiang & Venables, 2000; Gonzalez *et al*., 2002). Gradually, it was realized that the use of different fillers could yield different properties, some of which could even reinforce the properties of the neat polymer. Mineral fillers are used extensively due to their availability, ease of processing, and cost. These fillers consist mainly of carbonates, oxides, clays, alumina silicate, silica and silicates. Examples of some of these mineral fillers commonly used are talc, calcium carbonate, kaolin, magnesium carbonate, silica, alumina, titanium dioxide and mica.

**2.2.1 Calcium Carbonate**

Among the various mineral fillers, calcium carbonate (CaCO3) is one of the most important and widely used filler for plastics in term of weight (Murphy, 1996; Pukanszky, 1995). CaCO3 is the next most abundant naturally occurring element to silicon (Wake, 1971), which is the reason why it is deemed very cost effective to be made into fillers in plastics. CaCO3 occurs naturally in two crystalline forms, calcite and aragonite, the former being the more stable and abundant form. Calcite is rhombohedral, has a specific gravity of 2.71 and a hardness of 32.0 on the Moh.s Scale. Argonite is the orthorhombic form and

occurs naturally as needle-shaped prisms or spherulites in thermal springs. It has a specific gravity of 2.93 and a hardness of 3.5 on the Moh.s Scale. When heated, aragonite transforms into calcite.

CaCO3 is sometimes coated with stearic acid to reduce particle agglomeration; hence improvements to processability, wet strength and surface finish are conferred. Commercial CaCO3 fillers come in various sizes, which range from 8 µm to nanometer sizes. The particle shape is generally isometric as displayed in Figure 2.4 hence the aspect ratio of CaCO3 is fairly low. Generally, the incorporation of rigid particulate fillers to a polymer matrix will have an embrittling effect, which results in a decrease in impact strength of the system. In fact, most of the studies on modification of semi-crystalline polymers with rigid particle reported a significant loss of toughness compared to the neat polymer. Through further investigations, however, the usage of CaCO3 as a toughening agent is a new concept, which is relatively well known to impart high impact properties and deformability, though at the cost of the strength of PP.

There are a few works that have reported regarding the ability of CaCO3 to act as toughening agent, the most recent one being Zuiderduin *et al.* (2003), which confirmed that CaCO3 has indeed the ability to not only increase the stiffness but also the toughness of PP. Furthermore, their work has also reinforced the findings of Pukanszky (1995), which claimed that CaCO3 is a very weak nucleating agent and has no influence on the melting temperature and the crystallinity of PP. Thio *et al.* (2002) had attempted to study the effect of CaCO3 filler particle size as well as varying notch depths on the fracture resistance of CaCO3 filled composites. Smaller filler particles and higher volume fractions of filler were said to induce early fractures and also lead to decreased yield stress. Impact toughness of the filled composites that uses either very large or very small particles was observed to unchange from the unfilled PP value. The failure of both particle sizes to toughen the matrix was said to arise due to the presence of agglomerates and/or individual particles with dimensions larger than the critical flaw size to initiate brittle behavior. The usage of a moderate sized particle, however, was claimed to have resulted in an increased toughness of the composite due to combined mechanisms of crack deflection toughening and local plastic deformation of interparticle-ligaments following particle matrix debonding.

CaCO3 is also considered to be inactive filler due to its nearly spherical shape and to the lack of active .OH sites on its surface (Pukanszky, 1995). The reactivity of PP is also low since its carbon chain is a polar, which does not contain reactive groups. When CaCO3 is introduced into the PP matrix, the tensile properties of the composites usually become poorer than to those of virgin PP. Mechanical properties cannot be improved by the surface treatment of the filler because of the inactivity of the two components. Nevertheless, there have been a great number of compounds such as silanes, titanates, stearic acid, elastomers, and oligomers, which are used as coupling agents to treat CaCO3 in the hope of obtaining

some degree of enhancement in strength of the composite. However, more often than not, this is seldom achieved. While some publications claimed to have observed improvement in the mechanical properties of the composites with treated fillers, the published data could hardly be interpreted and the mechanism of increased component interaction was not known.

Other interesting works using CaCO3 was done by Badran *et al.* (1982), who have used various concentrations of ethylene oxide oligomer modified CaCO3 to investigate its effect on the mechanical properties of HDPE. Impact strength as well as deformability of the polymer was found to increase tremendously, while the tensile strength and modulus values were very near to that of pure HDPE. Chacko *et al.* (1983) made use of CaCO3 to study its effect on the tensile properties of PE with differing molecular weight. It was found that the tensile stress-strain behavior was dependant upon the filler content, polymer-filler interface, and polymer molecular weight. Further investigation using scanning electron microscopy revealed the presence of a craze type deformation in the composites.

Jancar and Kucera (1990) described the performance of both CaCO3 and Mg(OH)2 as fillers for PP and observed that the tensile yield stress of PP filled with CaCO3 decreased drastically with filler loading, which was blamed on poor filler adhesion to the matrix. However, the tensile yield strength of PP filled with Mg(OH)2 either decreased slowly or remained constant up to a certain filler loading, although the filler was dubbed to have poor adhesion to the matrix as well.

Supaphol *et al*. (2004) investigated the effects of CaCO3 of varying particle size (i.e. 1.9, 2.8, and 10.5 µm), content and type of surface modification on crystallization and melting behavior, mechanical properties, and process ability of CaCO3-filled syndiotactic polypropylene (sPP). It was found that CaCO3 was a good nucleating agent for sPP. The nucleating efficiency of CaCO3 for sPP depended strongly on its purity, type of surface treatment, and average particle size. Tensile strength was found to decrease, while Young’s modulus was found to increase, with increasing CaCO3 content. Shear viscosity of CaCO3 filled sPP was found to increase with increasing CaCO3 content and decreasing particle size

Study on the melt rheology and extrudate swell of CaCO3 nanoparticle filled\ isotactic PP (iPP) had been carried out by Rapeephun *et al.* (2005) With increasing of filler content the apparent shear viscosity was noticeable increased and percentage of extrudates swell was found to decrease. Comparison between uncoated and coated CaCO3 was also conducted and found that addition of stearic acid coated had decreased the apparent shear viscosity compared to the untreated CaCO3. Decreasing in shear viscosity was due to good dispersion of the CaCO3 nano-particles and poor interfacial adhesion between coated CaCO3 surface and iPP matrix.

**2.2.2 Application of Filler Treatments.**

Particulate filled polymers must be continuously upgraded in order to meet specific requirements of various new applications. One of the possible ways to do this is by modification of filler surface to alter the filler matrix interfacial interaction (Demjen, 1997). Although changing particle size can be done to modify interaction, the range is still narrow.

 Thus, the incorporation of coupling agents onto the filler surface is an obvious way to modify interaction by either changing the size of the interface or altering the strength of the interaction.

The influence of stearic acid treated CaCO3 on the morphology and mechanical properties of PP containing ethylene-octene copolymer (EOR) were thoroughly investigated by Premhet and Horanont (1999). It was observed that there was separate dispersion between the filler and elastomer. The use of surface treatment did not improve this condition; however, it was found that stearic acid promotes hexagonal crystallization of PP and gave a composite with lower onset crystallization temperature, TC Onset, as well as crystallization temperature, TC. As a consequence, composite with treated fillers possessed greater impact strength in comparison with composites containing uncoated fillers.

Generally, distribution or dispersion of the filler will affects a great deal to the viscoelasticity of the polymer matrix. According to Thio *et al.* (2002) and Nowaczyk *et al.* (2004) the distribution of the filler within the polymer matrix can be improved by surface treatment with a dispersant, e.g. stearic acid, which helps reduce the viscosity of the matrix and to some extent, prevent the fillers from forming network.

 **Chapter 3**

**EXPERIMENTAL**

**3.1 Plan of work**

|  |
| --- |
| Step No. Action |

1. Selection of base resin/material
2. Predrying
3. Formulations
4. Dry Mixing
5. Extrusion
6. Pelletizing
7. Screening
8. Molding
9. Testing

**3.2 SELECTION OF MATERIALS**

Materials used are listed in the following table:

|  |  |
| --- | --- |
| **Materials used** | **Function** |
| PPHP | POLYMER |
| PPCP | POLYMER |
| POLYOLEFIN ELASTOMER | POLYMER (elastomer) |
| ADDITIVE | ANTIOXIDANT |
| ADDITIVE | HEAT STABILIZER |
| ADDITIVE | LUBRICANT |
| ADDITIVE | PROCESSING AIDS |
| ADDITIVE | COUPLING AGENT |

SOURCES OF THE MATERIALS:

\*PPHP-RELIANCE

\*PPCP-RELIANCE

\*ELASTOMER-DOW CHEMICAL

\*ANTIOXIDENT-CIBA SPECIALITY CHEMICALS

\*HEAT STABILISER-CIBA

\*PROCESSING AID-CIBA

\*COUPLING AGENT-DOW CHEMICAL

**3.3 MACHINE SPECIFICATION**

MACHINE NAME: Theysohn (TSK 30/40D, Twin screw extruder)

SCREW DIAMETER: 30mm

L/D RATIO: 40

MAXIMUM OUTPUT: 50 kg /hr

**3.4 COMPOUNDING**

* The base polymer and filler (different particle size) are predried at 800C for 2 hours in air circulated oven.
* All materials are mixed in a high speed mixer for 3 minutes.
* Above composition is extruded in a co-rotating twin screw extruder.
* The L/D ratio of the extruder is 40.
* The extrudates from the die are quenched in a water tank at 20-300C and then palletized.
* After pelletizing all material passed through the screen to get the O.K. material.
* Processing temperature profile of the extrusion is shown in the each zone wise for every trial.
* The rpm of the pelletizer is maintained at a speed to achieve pellets of 3mm x 3mm size.

**3.5 Formulation of Trial-1 with Talc Coated 20µ**

Run Size- 5 kg

Batch Size- 5 kg

|  |  |  |
| --- | --- | --- |
| **Ingredients** | **Percentage** | **Kilograms** |
| PPCP High Impact | 20% | 1.0000 |
| PPCP High MFI | 13% | 0.6500 |
| PPHP | 30% | 1.5000 |
| Elastomer | 12% | 0.6000 |
| Talc Coated(20µ) | 24% | 1.2000 |
| Primary antioxidant | 0.05% | 0.0025 |
| Heat stabilizer | 0.1% | 0.0050 |
| Lubricant | 0.2% | 0.0100 |
| Processing aids | 0.15% | 0.0075 |
| Coupling agent | 0.5% | 0.0250 |

Bulk density of pp for feeder-1;

 (Weight of beaker +weight of material)-weight of beaker

 1000

 (250+530)-250 = 0.530

 1000

Similarly as for feeder -2 (Talc coated-20 µ) the bulk density:

 Bulk density =0.470 ;Feeder 1- Set point- 15.200kg/h; Feeder2- Set point- 4.80kg/h

**3.5.1 Process parameter of TSK- 30 for Trial -1 with Coated 20µ:**

|  |  |  |  |
| --- | --- | --- | --- |
| **Control Name** | **Unit** | **Set Point** | **Actual Temp**. |
| Control Barrel 1: | 0C | --- | ---- |
| Control Barrel 2: | 0C | 190 | 186 |
| Control Barrel 3: | 0C | 200 | 197 |
| Control Barrel 4: | 0C | 210 | 210 |
| Control Barrel 5: | 0C | 215 | 217 |
| Control Barrel 6 : | 0C | 215 | 217 |
| Control Barrel 7: | 0C | 220 | 236 |
| Control Barrel 8: | 0C | 220 | 237 |
| Control Barrel 9: | 0C | 225 | 235 |
| Control Barrel 10: | 0C | 225 | 233 |
| Die Temp.: | 0C | 230 | 231 |
| Adapter/Cont. | 0C | 230 | 232 |
| Speed Side Seeder1: | Rpm | 131 | ----- |
| Speed Side Seeder2: | Rpm | ----- | ----- |
| Speed Main Drive: | Rpm | 523 | ----- |
| Vacuum Pressure | Inch-hg | -28 | ----- |
| Torque Main Drive | Amp | 32 | ----- |
| Feeder RPM | Rpm | ---- | ----- |
| Cutter Speed | ----- | 3 | ----- |
| Production Rate | Kg/hr | 20 | ----- |

For the melt blending the temperature profile of the extrusion are shown of each zone for every trial the extrudates of the compositions shown in the table above were palletized in Scheer palletizing Machine(Germany).

**3.6 Formulation of trial-2 with Coated 10µ:**

Machine name- TSK- 30

Run size- 5 kg

Batch size- 5 kg

|  |  |  |
| --- | --- | --- |
| **Ingredients** | **Percentage** | **Kilograms** |
| PPCP High Impact | 20% | 1.0000 |
| PPCP High MFI | 13% | 0.6500 |
| PPHP | 30% | 1.5000 |
| Elastomer | 12% | 0.6000 |
| Talc coated( 10µ) | 24% | 1.2000 |
| Primary antioxidant | 0.05% | 0.0025 |
| Heat stabilizer | 0.1% | 0.0050 |
| Lubricant | 0.2% | 0.0100 |
| Processing aids | 0.15% | 0.0075 |
| Coupling agent | 0.5% | 0.0250 |

Bulk density of feeder-1 (PP) = 0.530

Bulk density of feeder-2 (GSS-10) = 0.460

Feeder 2- Set point- 4.800 kg/hr

Feeder1- Set point- 15.200 kg/hr

**3.6.1 Process parameter of TSK- 30 for Trial -2 with Coated -10µ**

|  |  |  |  |
| --- | --- | --- | --- |
| **Control Name** | **Unit** | **Set Point** | **Actual Temp**. |
| Control Barrel 1: | 0C | --- | ---- |
| Control Barrel 2: | 0C | 190 | 186 |
| Control Barrel 3: | 0C | 200 | 197 |
| Control Barrel 4: | 0C | 210 | 210 |
| Control Barrel 5: | 0C | 215 | 217 |
| Control Barrel 6 : | 0C | 215 | 217 |
| Control Barrel 7: | 0C | 220 | 236 |
| Control Barrel 8: | 0C | 220 | 237 |
| Control Barrel 9: | 0C | 225 | 235 |
| Control Barrel 10: | 0C | 225 | 233 |
| Die Temp.: | 0C | 230 | 231 |
| Adapter/Cont. | 0C | 230 | 232 |
| Speed Side Seeder1: | Rpm | 131 | ---- |
| Speed Side Seeder2: | Rpm | ----- | ---- |
| Speed Main Drive: | Rpm | 523 | ---- |
| Vacuum Pressure | Inch-hg | -28 | ---- |
| Torque Main Drive | Amp | 32 | ---- |
| Feeder RPM | Rpm | ---- | ---- |
| Cutter Speed | ----- | 3 | ---- |
| Production Rate | Kg/hr | 20 | ---- |

**3.7 Formulation of Trial-3 with Coated 7µ:**

Machine name- TSK- 30

Run size- 5 kg3

Batch size- 5 kg

|  |  |  |
| --- | --- | --- |
| **Ingredients** | **Percentage** | **Kilograms** |
| PPCP High Impact | 20% | 1.0000 |
| PPCP High MFI | 13% | 0.6500 |
| PPHP | 30% | 1.5000 |
| Elastomer | 12% | 0.6000 |
| Coated Talc (7µ) | 24% | 1.2000 |
| Primary antioxidant | 0.05% | 0.0025 |
| Heat stabilizer | 0.1% | 0.0050 |
| Lubricant | 0.2% | 0.0100 |
| Processing aids | 0.15% | 0.0075 |
| Coupling agent | 0.5% | 0.0250 |

Bulk density of feeder-1 (PP) = 0.530

Bulk density of feeder-2 (GSS-7) = 0.330

**3.7.1Process parameter of TSK- 30 for Trial -3 with Coated-7µ:**

|  |  |  |  |
| --- | --- | --- | --- |
| **Control Name** | **Unit** | **Set Point** | **Actual Temp**. |
| Control Barrel 1: | 0C | --- | ---- |
| Control Barrel 2: | 0C | 190 | 186 |
| Control Barrel 3: | 0C | 200 | 197 |
| Control Barrel 4: | 0C | 210 | 210 |
| Control Barrel 5: | 0C | 215 | 217 |
| Control Barrel 6 : | 0C | 215 | 217 |
| Control Barrel 7: | 0C | 220 | 236 |
| Control Barrel 8: | 0C | 220 | 237 |
| Control Barrel 9: | 0C | 225 | 235 |
| Control Barrel 10: | 0C | 225 | 233 |
| Die Temp.: | 0C | 230 | 231 |
| Adapter/Cont. | 0C | 230 | 232 |
| Speed Side Seeder1: | Rpm | 131 | ---- |
| Speed Side Seeder2: | Rpm | ----- | ---- |
| Speed Main Drive: | Rpm | 523 | ---- |
| Vacuum Pressure | Inch-hg | -28 | ---- |
| Torque Main Drive | Amp | 32 | ---- |
| Feeder RPM | Rpm | ---- | ---- |
| Cutter Speed | ----- | 3 | ---- |
| Production Rate | Kg/hr | 20 | ---- |

**3.8 Formulation of Trial -4 withTalc Coated -3µ**

Machine name- TSK- 30

Run size- 5 kg

Batch size- 5 kg

|  |  |  |
| --- | --- | --- |
| **Ingredients** | **Percentage** | **Kilograms** |
| PPCP High Impact | 20% | 1.0000 |
| PPCP High MFI | 13% | 0.6500 |
| PPHP | 30% | 1.5000 |
|  Elastomer | 12% | 0.6000 |
| Coated Talc (3µ) | 24% | 1.2000 |
| Primary antioxidant | 0.05% | 0.0025 |
| Heat stabilizer | 0.1% | 0.0050 |
| Lubricant | 0.2% | 0.0100 |
| Processing aids | 0.15% | 0.0075 |
| Coupling agent | 0.5% | 0.0250 |

Bulk density of feeder-1 (PP) = 0.530

Bulk density of feeder-2 (Talc coated 3µ) = 0.450

**3.8.1 Process parameter of TSK- 30 for Trial -4 with Talc Coated 3µ**

|  |  |  |  |
| --- | --- | --- | --- |
| **Control Name** | **Unit** | **Set Point** | **Actual Temp**. |
| Control Barrel 1: | 0C | --- | ---- |
| Control Barrel 2: | 0C | 190 | 186 |
| Control Barrel 3: | 0C | 200 | 197 |
| Control Barrel 4: | 0C | 210 | 210 |
| Control Barrel 5: | 0C | 215 | 217 |
| Control Barrel 6 : | 0C | 215 | 217 |
| Control Barrel 7: | 0C | 220 | 236 |
| Control Barrel 8: | 0C | 220 | 237 |
| Control Barrel 9: | 0C | 225 | 235 |
| Control Barrel 10: | 0C | 225 | 233 |
| Die Temp.: | 0C | 230 | 231 |
| Adapter/Cont. | 0C | 230 | 232 |
| Speed Side Seeder1: | Rpm | 131 | ---- |
| Speed Side Seeder2: | Rpm | ----- | ---- |
| Speed Main Drive: | Rpm | 523 | ---- |
| Vacuum Pressure | Inch-hg | -28 | ---- |
| Torque Main Drive | Amp | 32 | ---- |
| Feeder RPM | Rpm | ---- | ---- |
| Cutter Speed | ----- | 3 | ---- |
| Production Rate | Kg/hr | 20 | ---- |

**3.9 SPECIFICATION OF INJECTION MOULDING FERROMATIC MILACRON-80W**

|  |  |  |
| --- | --- | --- |
| INTERNATIONAL DIMENSIONS |   | 800-217 |
| INJECTION UNIT SPECIFICATIONS | UNITS | 36 MM FRAME |
|   |   | A  | B  | C |
| SCREW DIAMETER | Mm | 32 | 36 | 42 |
| INJECTION CAPACITY MAX. (GPPS) | Gm | 99 | 125 | 171 |
| THEROTICAL DISPLACEMENT | Cc | 104 | 132 | 180 |
| INJECTION PRESSURE MAX. | Bar | 2074 | 1639 | 1204 |
| INJECTION RATE | cc/sec | 105 | 133 | 180 |
| INJECTION SCREW STROKE | Mm | 130 | 130 | 130 |
| SCREW L/D RATIO |   | 22.6 | 20 | 17 |
| SCREW SPEED | Rpm | 393 | 312 | 312 |
| PLASTICIZING CAPACITY | gm/sec | 15 | 17 | 26 |
| TOTAL HEAT CAPACITY | Kw | 7.3 | 7.3 | 7.3 |
| CLAMP UNIT SPECIFICATIONS |   |
| CLAMP FORCE | Tons | 80 |
| CLAMP STROKE | Mm | 500 |
| MAXIMUM DAYLIGHT | Mm | 700 |
| MINIUM MOULD HEIGHT | Mm | 200 |
| PLATEN SIZE | Mm | 610 x 570 |
| DISTANCE BETWEEN TIE ROD ( HxV) | Mm | 410 x 370 |
| TIE ROD DIAMETER | Mm | 70 |
| EJECTOR STROKE | Mm | 100 |
| EJECTOR FORCE | Kn | 27 |
| GENERAL DATA |   |
| ELECTRICAL MOTOR | kW (hp) | 15(20) |
|  |  |  |

|  |
| --- |
| **F:\Documents and Settings\Quality\My Documents\My Pictures\omega-tn.jpg** |
| Figure 3.1 Ferromatic Milacron InjectionMolding Machine(80 Ton; SIGMA –SERIES) |

**3.9.1 PROCESS PARAMETER OF INJECTION MOULDING**

The granules of the extrudates are predried in an air circulated oven at 900C for 2 hours and injection molded in a microprocessor based FERROMATIC MILACRON injection moulding machine fitted with a master mould containing the cavity for tensile strength, flexural and impact specimens. After its ejection from the mould, specimens were conditioned at 230C at 50 %RH for 48 hours. Processing parameters are shown in the table-

**3.9.1 CLAMP SET UP**

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| CLOSE | POSITION | SPEED | PRESSURE | TIME |
| Fast close(1) |  | 25 |  |  |
| (2) | 150.0 | 30 | ------ | 2.26 |
| (3) |  | 40 | ------ |  |
| Mold safety(1) | 30 | 30 | 35 | 5.00 |
| (2) | 23 | ---- | 30 | 0.20 |
| Tonnage | 15.50 | ----- | 50 | 0.64 |

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| OPEN | POSITION | SPEED | PRESSURE | TIME |
| Break away (1) |  | 30 | ----- | 0.94 |
| Fast open(1) | 150.0 | 30 | ------ | ------- |
| (2) | 35.0 | 30 | ------ | ------- |
| (3) | 35.0 | 25 |  | ------- |
| Slow open(1) | 250.0 | ---- | 30 | 0.20 |
| (2) | 300 | ----- | ------ | 0.91 |

3.9.1.2 EJECTER SET UP

|  |  |
| --- | --- |
| Ejector start delay | 0.00 Sec. |
| Dwell time | 0.00 Sec. |
| Ejector pulse | 1 Sec. |
| Ejector forward speed  | 40% |
| Ejector ret. Speed | 40% |
| Ejector forward pressure | 30% |
| Ejector retract pressure | 35% |

3.9.1.3TEMPERATURE SET UP

|  |  |  |  |
| --- | --- | --- | --- |
| Control Name | Unit | Set Point | Actual Temp. |
| Zone 1: | 0C | 180 | 180 |
| Zone 2: | 0C | 190 | 190 |
| Zone 3: | 0C | 200 | 200 |
| Zone 4: | 0C | 210 | 208 |
| Zone 5: | 0C | 30 | 41 |
| Zone 6: | 0C | 30 | 41 |
| Feed throat | 0C | 60 | 63 |
| Oil temp. | 0C | 50 | 37 |
| Cabinet temp. | 0C | ----- | 40 |
| Alarm band | 0C | ----- | 20% |

3.9.1.4 INJECTION SET UP

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
|  | Position(mm) | Velocity (%) | Pressure (%) | Time(sec.) |
| Inj. (1) | ---- | 35 | 35 | 5.0 |
| (2) | 8.0 | 35 | ---- |  |
| (3) | 0.0 | 35 | ---- | 0.80 |
| Pack(1) | 5.0 | 5.0 | 35 | 8.0 |
| (2) | ---- | ---- | 35 | 8.0 |
| Hold(1) | ---- | ---- | 30 | 8.0 |
| (2) | ---- | ---- | 30 | 8.0 |

3.9.1.5 EXTRUDER SET UP

|  |  |
| --- | --- |
| Decompose before shot size | 0.0 mm |
| Extruder RPM # 1 | 60 |
| Back Pressure# 1 | 2 |
| Extruder RPM # 2 | 60 |
| Back Pressure# 2 | 2 |
| Shot Size | 32 |
| Sled forward Pres. | 40 |
| Sled Retract Pres. | 10 |
| Actual Ext.Pres. | 0 rpm |
| Act. Screw Pos. | 32.7 |

**3.10. OBSERVATION**

**FOR TRIAL -1**

Tensile Specimen-- Shot Size: 29.5 mm

 Cooling Time: 15 sec.

Flexural Strength-- Shot Size: 22.0 mm

 Cooling Time: 20 sec.

Izod-- Shot Size: 16.2 mm

 **FOR TRIAL -2**

Tensile Specimen-- Shot Size: 30.0 mm

 Cooling Time: 20 sec.

Flexural Strength-- Shot Size: 22.0 mm

 Cooling Time: 25 sec.

Izod-- Shot Size: 16.2 mm

 Cooling Time: 20 sec.

 **FOR TRIAL -3**

Tensile Specimen-- Shot Size: 32.0mm

 Cooling Time: 15 sec.

Flexural Strength-- Shot Size: 22.0 mm

 Cooling Time: 15 sec.

Izod-- Shot Size: 16.2 mm

 CoolingTime: 15sec.

 **FOR TRIAL -4**

Tensile Specimen-- Shot Size: 32.0 mm

 CoolingTime: 15 sec.

Flexural Strength-- Shot Size: 22.0 mm

 CoolingTime: 15.0 sec.

Izod-- Shot Size: 17mm

 CoolingTime: 15 sec.

**Chapter 4**

 **Result and Discussion**

**4. Mechanical properties**

Tensile strength as per ASTM D 638 is evaluated using universal tensile testing machine LR 10k from Lloyd instruments Ltd. U.K at a crosshead speed of 50 mm/min. Flexural properties according to ASTMD790 are tested using LR 10K from Lloyd instruments Ltd. U.K. Izod impact test are carried out using an HEM impact tester (ASTM D 256). A 2.00J energy hammer is used and the striking velocity was 3.46m/sec. For Izod impact test specimens the notch is cut using a motorized notch-cutting machine (Hemtek). The unit of expression is kJ/m2.



**Fig: 4.1 UNIVERSAL TESTING MACHINES**

**LLOYD (10K) INSTRUMENT (UK)**

 (**Source: industry lab)**



***RESULTS AND DISCUSSION***

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| Sr.no. | Properties | Method | Unit | Trial-1 COATED 20µ | Trial-2 COATED 10µ | Trial-3 COATED7µ | Trial-4 COATED3µ |
| 1 | MFI@ 2300C/2.16kg | ASTM D-1238 | Gms/10Min | 13.5 | 14.2 | 10.2 | 10.2 |
| 2 | SPECIFIC GRAVITY | ASTM D-792 | - | 1.05 | 1.06 | 1.06 | 1.06 |
| 3 | FILLER CONTENT | ASTM d-5630 | % | 22.8 | 23.3 | 24.9 | 24.9 |
| 4 | TENSILE STRENGTH | ASTM D-638 | Kgf/CM2 | 185 | 185 | 188 | 202 |
| 5 | ELONGATION@ BREAK | ASTM D-638 | % | 90 | 50 | 50 | 44 |
| 6 | FLEXURAL STRENGTH | ASTM D-790 | Kgf/cm2 | 282 | 292 | 338 | 352 |
| 7 | FLEXURAL MODULAS | ASTM D-790 | Kgf/cm2 | 15280 | 15370 | 20540 | 22760 |
| 8 | IZOD IMPACT (NOTCH) | ASTM D-256 | KJ/m2 | 12.5 | 11.5 | 14.5 | 18.0 |

**4.1Tensile properties**

Above data shows the variation of tensile strength with filler concentration of Talc and the size of the filler particle. There is a significant increment in the strength as the filler loading increased lesser talc particle sizes showed higher increments. The increment may be due to the fine structure of the talc filler providing good reinforcement. Elongation properties as seen from abovetable, decreased with the presence of small filler that indicates interference by the filler in the mobility or deformability of the matrix. This interference was created through the physical interaction and immobilization of the polymer matrix by imposing mechanical restraints.

**4.2Flexural properties**

Above datadepicts the variation in flexural modulus with varying the size of the talc and the concentration of the filler. The flexural modulus increased with the decrease in filler size of the talc. The rate of increase of flexural modulus is comparable to the increase in the concentration and decrease in particle size of talc. Thus it is confirmed that the total area available to deformation stress played an important role. Above data presents the variation in flexural strength. It is seen that flexural strength increased with the decrease in particle size of the filler. Similarly the flexural strength is also varying as the size of the filler talc size is decreased and concentration increased.

**4.3 Impact properties**

Above table illustrates the variation of impact (Izod) strength with filler by weight percentage and the size of the talc particle. It is clear from the table that the strength increment at high weight percentage of filler may be attributed to the formation of small sized crystallites, i.e. spherulites, as well as the capacity to absorb more energy by increased portion of matrix. A further increase in weight percentage reduced the deformability of the matrix, and, in turn, reducing the ductility in the skin area so that the tended to form a weak structure. Above table shows the Variation of the Impact Strength of polypropylene with Filler Content as well as the size of the talc particle.



**FIG -4.2 IZOD IMPACT TESTER**

**(Source: industry lab)**

Chapter 5

**Conclusion and suggestion for future work**

 **5.1 CONCLUSIONS**

* There is a significant increment in the flexural properties with an increase in the fillerloading a decrement in the size of filler (talc) particle size.
* The toughness and elongation at break decreased as particle size and agglomeration concentration increased in the all cases of fillers.
* Inorganic fillers, such as talc, added to the polymer improved impact property, tensile Property and dimension stability.
* The mechanical properties of the polypropylene are found to be a function of the particle size, aspect ratio, the dispersion, the particle orientation and the interfacial interaction between the minerals and the polymer matrix. Fine filler particle such as talc gave significant improvement in the mechanical properties.

**5.2 SUGGESTION FOR FUTURE WORK**

Some of the future work to answer these questions is as follows:

• A model that accurately predicts the stress growth and stress relaxation of flow induced stresses generated during mold filling and subsequent cooling after cessation of flow for fiber reinforced thermoplastics must be developed.

• There needs to be more extensive studies on the surface chemistry, i.e. the interactions that occur between that of the filler and the matrix.

• Develop a method to enhance the dispersion of the nanoparticles in order to maximize the modulus enhancement of the composites.

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