

**FINDING THE BEST LEVEL OF FILLER MASTER  
BATCH TO BE ACCOMMODATED IN THIN GAUGED  
PRIMARY PACKAGING PRODUCTS**

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Degree of Master of Science

Department of Chemical Engineering

University of Moratuwa

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Thesis/Dissertation submitted in partial fulfilment of the requirements for the degree  
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Thank you.

**Abstract:**

***“Finding the best level of filler master batch to be accommodated in thin gauged primary packaging products”***

The main aim of this dissertation was “Finding the best level of filler master batch to be accommodated in thin gauged primary packaging products”, as currently there is only limited information available in the printed media especially for Thermoforming made thin gauged food contact plastic products.

Here only the thermoformed thin gauged primary packaging products made out of Polystyrene and Polypropylene materials were studied. To find out best filler level in plastic compounds 2 series of formulations such as Series N1 for Polystyrene based materials and Series N2 for Polypropylene based materials were prepared, where virgin filler load level varied from 0% to 25% were prepared and experimented. And these formulations were based on incremental addition of heavy filler such as Calcium Carbonate in virgin form as Filler Master Batch. Each and every Raw Material batches were tested for MFI (Melt flow Index) against the specification. Thin gauged sheets were extruded according to the decided formulation. Products were turned out via thermoforming process to test for consumer acceptance. Further in order to find out the best level of filler master batch to be accommodated to these products, thermoformed primary packaging was examined for Appearance Test, Specific Gravity test, Drop Test or Impact test, Oil Test and Actual Filler Master Batch test. Those tests were carried out to find out whether physical and aesthetic properties were compromised or not. It was observed during the trials that 25% Virgin FMB based formulated sheets were not suitable for thermoforming process as it tended to crack and torn like a paper. It was concluded through Actual FMB level test, that the accumulated Calcium Carbonate Filler level in the regrind introduced in these formulation had an impact on overall level of fillers in the extruded sheets had caused these cracks and defects. Based on the test results it was concluded that 26% of Filler in Styrene based materials and 40% of Fillers in polypropylene based materials were the optimum level of Filler Master Batch for these thin gauged primary packaging products.

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## **LIST OF ABBREVIATIONS**

Abbreviation	Description
GPPS	General Purpose Polystyrene
HIPS	High Impact Polystyrene
PS	Polystyrene
PP	Polypropylene
FMB	Filler Master Batch
WMB	White Master Batch
RP	Recycled Plastics (Crush)



## CHAPTER 01

### INTRODUCTION

As per the information released by Transparency Market Research published in a new market report titled “**Plastic Packaging Market- Global Industry Analysis, Size, Share, Growth, Trends and Forecast 2014~ 2020**”, the Global Plastic Packaging Market was valued at US \$ 260 Billion in the year 2013 and is expected to reach US \$ 370 Billion by year 2020 with growth rate of 5.2% over the forecast period between 2014~ 2020. In terms of volume, the plastic packing market was worth 78.4 Mega Tons in the year 2013 and expected to reach 108.9 Mega Ton by the end of 2020[5]

Expansion planning on FMCG (Fast Moving Consumer Goods) product Industries were the key driver for this growth in the packaging industries. The Global Packaging industry can be divided in two category of product segments. They were:

- Rigid Packaging
- Flexible Packaging

Application of these packaging are segmented in to six category based on their applications. They are;

- Food & Beverages
- Industrial
- Household Products
- Personal Care
- Medical
- Others ( Including various components in Automotives)

Rigid Packaging market is also segmented into two category as

- Disposable
- Permanent.

This research was concerned with the Disposable Rigid Plastic Thermoformed product which was used in Food & Beverage industries such as Hotels, Restaurants and Catering (HORECA- Market). The rising demand for these primary packaging products providing meals for Office and construction workers “on-the-go”. This boosted the demand for disposable packaging products for food.

Disposable Rigid Plastic Thermoformed Packaging consists of thin gauged and comes in all shapes and sizes of Plastic Cups, Plates, Tubs, Bowls, portions cups, clamshells etc. and also with different types of materials such as PP (Polypropylene), PS (Polystyrene), APET (Amorphous Polyethylene Terephthalate), CPET (Crystallized Polyethylene Terephthalate), RPET (Recycled Polyethylene Terephthalate) etc. The increase in demand for these consumer plastic products was due to its aesthetically pleasing, protection, performance and competitive cost. Further, the growth of Rigid Plastic Packaging products was expected to continue in the coming decade due to lightweight and user-friendly because they have increasingly replaced the traditional packaging materials.

The main raw material for these disposable packaging products were generated from petrochemicals such as Crude Oil. With high demand for Crude Oil and limited supply the price of Crude Oil for the past few years has been increased, which had led to the increase in cost of manufacturing of these disposable plastic products. Therefore, Packaging Industries were looking towards alternative raw material substitutions to reduce the cost of production. The introduction of filler was commonly used by plastic processors and number of studies had been carried out. However information related to study of the effect of the filler on properties of thermoformed products especially of thin gauge one was very limited.

One of the popular fillers used in Rigid Plastic Packaging industry is Calcium Carbonate and the choice for this inorganic mineral filler is accounted for two main reasons such as Low Cost and Bright white color.

Therefore, the topic selected for this research was “Finding the best level of filler master batch to be accommodated in thin gauged primary packaging products”. Since the

information and research of this filler master batch for thin gauged thermoformed primary packaging products were limited, So I had to do a research to find out the limitation and level of extent that we can add Calcium Carbonate based Filler Master Batch in the formulation in order to reduce the cost. Further, the price of FMB against the synthetic resin was around 30~ 40%. Which means every single kilogram of Calcium Carbonate based Filler would save 2/3 value of the Synthetic Resin. Therefore, the level of additional increase means the more cost saving. The aim of research was to find out the optimum level of dosage through continuous addition of recycle plastics in the formulation to provide cumulative effect in the end to find best possible FMB level without compromising the property and quality of the end product.

In the Thermoforming industries trim or skeletal scrap was depend on mould design distribution on the sheet. There were instances where skeletal scrap was around 65% depend on the product. So it was not feasible to throw out these skeletal scrap, therefore it was reground and re-used as a Recycle Plastic (RP) material. Some extruders were capable to handle direct regrind having a size of 6~8mm in their formulation, but there were other types of extruders requires only palletized re-grind in such situation these regrinds were palletized via Palletizers and used as RP materials. Since FMB – Filler Master Batch was added in main formulation there will be Calcium Carbonate in the RP Materials. Most companies ignores the level of FMB in the RP material and add FMB freshly for each and every formulation in order to minimize the cost of production. Therefore, actual content of Calcium Carbonate was much higher than that of indicated level of FMB in the formulation. This was due to accumulation of FMB or Calcium Carbonate in the RP materials when it passed through several number of re-grind process. Therefore, level of FMB may reached up to 57% when several number of re-grind passes occurred. Therefore, the product may fail, if there is no proper tracking of the no of passes. In the Thermoforming industry it was not possible to track as mass production was carried out and the volume of products were too high as well as the volume of generation of re-grind were too high. Therefore, alternate method of identifying the level of FMB or Calcium Carbonate was necessary to decide the best

possible level of FMB to accommodate in the Finished Good products so that it may not fail during consumer usage.

There was no research done so far on the best level of FMB to be accommodated in the FMCG thin gauged primary packaging product. Therefore, the research was based on to find the best FMB accommodation level without compromising the physical and aesthetic properties of the product.

Physical and aesthetic properties include the appearance of the product such as free from deformation, impurities, surface gloss, fitment if any and cracks. There shouldn't be any fail when subject to moderate impact such as when closing a lid to the cup by applying pressure on the edge of the lids. Further, it should not fail during usage such as when subjected to cold or oil foods it should be leak proof.

## **CHAPTER 02**

### **OBJECTIVES**

The objectives of the present research were formulated below:

1. Study of effect of filler load in Polystyrene based composition on flow ability of Plastic Composition. In order to achieve this objectives series of formulation of Polystyrene based composition with increase of filler loading level was to be designed and to study the flow ability through the Appearance test.
2. Study of effect of filler load in Polypropylene based composition on flow ability of Plastic composition. In order to achieve this objectives series of formulation of Polypropylene based composition with increase of filler loading level was to be designed and to study the flow ability through the Appearance test.
3. Development of Plastic compound formulation in order to find the best level of filler to be accommodated while maintaining the consistent end product weight and without compromising the physical and aesthetic properties. In order to achieve this objectives series of formulation of Polystyrene and Polypropylene based composition with increased filler loading level were to be designed against



the reference sample with the 0% filler loading level. Further to maintain the consistent end product weight specific gravity and actual FMB level test were to be carried out. Further, Appearance Test, Drop test as well as Oil test to be carried out in order to ensure that there were no deviation of Physical and aesthetic properties in the end product.

## **CHAPTER 03**

### **LITERATURE REVIEW**

#### **3.1 Properties of Polypropylene and Polystyrene**

##### **3.1.1 Preparation of Polypropylene**

Polypropylene is a linear hydrocarbon polymer containing almost no unsaturation. It has almost similar properties to polyethylene, such as swelling and the behavior of the solution and the electrical properties. The presence of a methyl group attached to alternative carbon atoms on the skeleton of the chain can alter the properties of the polymer in several ways, for example, interfere with the molecular symmetry and the tightening of the chain. This causes the increase of the crystalline melting point. As a result, most of the regular polypropylene with 50<sup>0</sup>C melting point higher than most regular polyethylene. Side methyl groups also influence some of the chemical behavior. For example, the tertiary carbon atom provides a site for the oxidation so that the polymer is less stable than the polyethylene to the influence of the oxygen. In addition, heat treatment and high energy led to the split of the chain rather than the Crosslinking. Preparation of polypropylene is almost similar to the preparation of polyethylene using catalysts such as Ziegler type. In both situations, the monomers are produced by the cracking of petroleum products such as natural gas or light oils. For the preparation of Polypropylene, the fraction C3 (i.e. Propylene or Propane) is the basic intermediate which is separated by fractional distillation. In the preparation of the polymer, such as water and the methyl acetylene are eliminated. A typical catalyst system can be prepared by reacting the titanium trichloride with that of the triethyl of aluminum, the tributyl of aluminum or the aluminum diethyl monochloride in naphtha under nitrogen to form a

suspension consisting of 10% catalyst and 90% of naphtha. The properties of the polymer was highly dependent on the composition of the catalyst and the shape and size of the particles<sup>[10]</sup>.

In the current market we can find different grades of Polypropylene. And these grades of Polypropylene are classified to five main classes such as:

1. Homopolymers produced by Ziegler –Natta Catalysis;
2. Block Copolymers produced by Ziegler –Natta Catalysis
3. Random Copolymers produced by Ziegler- Natta Catalysis
4. Rubber-Modified blends of the above;
5. Homopolymers and copolymers produced by metallocene catalysis.

Based on the influence of the methyl group Polypropylene is categorized into three types. They are:

1. Isotactic Polypropylene
2. Syndiotactic Polypropylene;
3. Atactic Polypropylene:

The isotactic form is more regular, because the methyl groups are all placed on one side of the molecule. Therefore, such molecules cannot crystallize in a zigzag form like those of the polyethylene due to the steric hindrance methyl hangovers, but crystallize in a helix, with three molecules required for a turn of the helix. The helices which occur on right and left, but both forms may enter the same crystal structure. Commercial polymers of polypropylene are usually 90 to 95% isotactic. In these products, the syndiotactics and atactics structures may be present in the form of complete molecules or as variable-length blocks in molecules isotactic chains otherwise. Polymers of stereo block can also be trained in which a block of residue monomers with a right helix is succeeded by a left-handed helix block. The frequency with which these changes in direction of the helix can have an important influence on the crystallization and so on properties in bulk of the polymer. Many manufacturers simply claim that their products are highly

isotactic, others cite the crystallinity of polymer obtained after a specified annealing treatment, while others cite the 'isotactic index', the percentage of insoluble in n-heptane polymer.

Commercial providers of polypropylene indicate that their molecular weight of material lie in the range of minutes = 38 000 ~ 60 000 and  $M_w = 220\ 000 \sim 700\ 000$ , with values of  $M_w / M_n$  to about 5.6 to 11.9<sup>[1]</sup>. The influence of molecular weight on the bulk properties of polypropylene is often contrary to that experienced with most other well known polymers. Although an increase in molecular weight increases the viscosity in the molten state and the resistance to shocks/impact, in agreement with most of the other polymers, it causes also a lower resistance elasticity, low hardness, low stiffness and a softening point. We think that this effect is due to the fact that the high molecular weight polymer crystallizes not so easily as a material of lower molecular weight and what are the differences in the degree of crystallization that affect the bulk properties. We can also mention that an increase in molecular weight led to a reduction of the brittle point.[10] See Table 3.1.1 below.

Table 3.1.1 Some Mechanical and Thermal Properties of Commercial Polypropylene

Property	Test Method	Homo-polymers			Copolymers	
Melt Flow Index	2.16kg @230 <sup>0</sup> C	3.0	0.7	0.2	3.0	0.2
Tensile Strength (MPa)	Straining Rate 45cm/min	34	30	29	29	25
Elongation at Break (%)	“	350	115	175	40	240
Brittleness Temp ( <sup>0</sup> C)	ASTM D.476	+15	0	0	-15	-20
Vicat Softening Point ( <sup>0</sup> C)	BS 2782	145~150	148	148	148	147
Rockwell Hardness	R- Scale	95	90	90	95	88.5
Impact Strength (J)	13.5	34	46	46	57.5	

[Source: Plastic Materials by Brydson, J.A]

The isotactic polypropylene has a lower density of 0.90 g / cm<sup>3</sup> and presents a higher softening point and therefore a higher maximum operating temperature. Items can withstand boiling and be subjected to many steam sterilization operations. For example, the mouldings were sterilized in hospitals for more than 1000 hours at 135 ° C in wet and dry conditions without any critical damage. In addition, polypropylene seems to be

free of problems of environmental stress cracking. The only exception are to be with concentrated sulfuric and chromic acids. And it has a higher brittle point and more likely to be oxidized.

As stated above, the mechanical and thermal polypropylene properties depend on the isotacticity and the molecular weight and the other features of the structure. The properties of five commercial materials of almost the same content isotactic, but which differ in molecular weight and being either a homo polymer or of block copolymers are compared in the table (3.1.1) above. In addition, it clearly shows that the increase in molecular weight - decrease melt flow index that causes a reduction in resistance to tensile strength, rigidity, hardness and the brittle point, but an increase in resistance to shocks/impact. The General effects of the isotactic index and melt flow index on some mechanical and thermal properties are also represented graphically in the figures 3.1~3.4 [10].

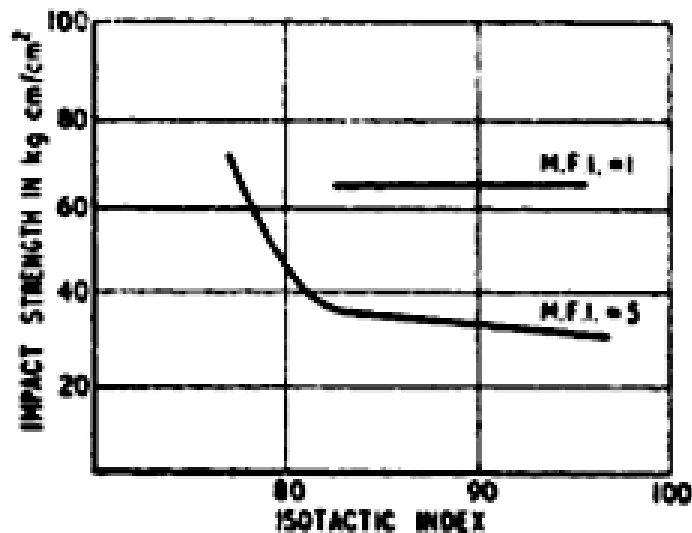


Fig 3.1 Variation of Impact Strength with Melt Flow Index (10kg@190°C) and isotactic index. (Source: Plastic Materials by Brydson, J.A)

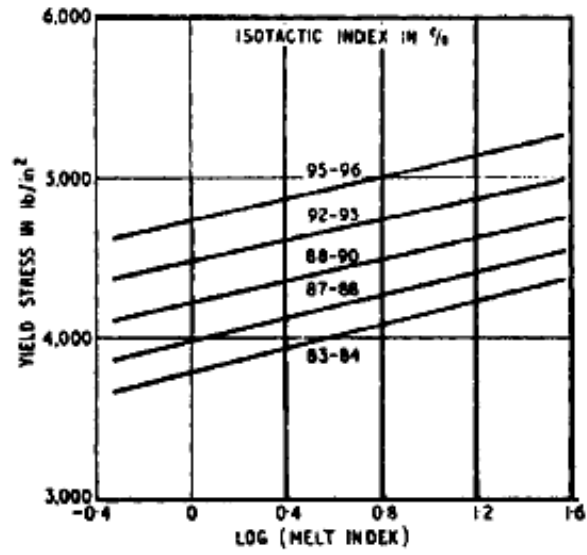


Fig 3.2 Variation of Tensile Yield Stress with Melt Flow Index (10kg@190<sup>0</sup>C) and isotactic index. (Source: Plastic Materials by Brydson, J.A)

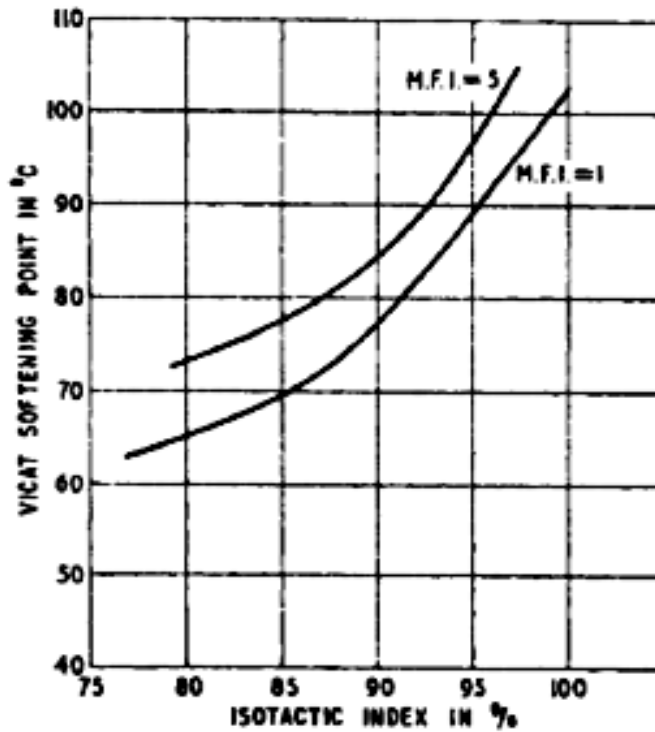


Fig 3.3 Variation of Vicat Softening point (5Kg Load) with MFI and Isotactic Index. (Source: Plastic Materials by Brydson, J.A)

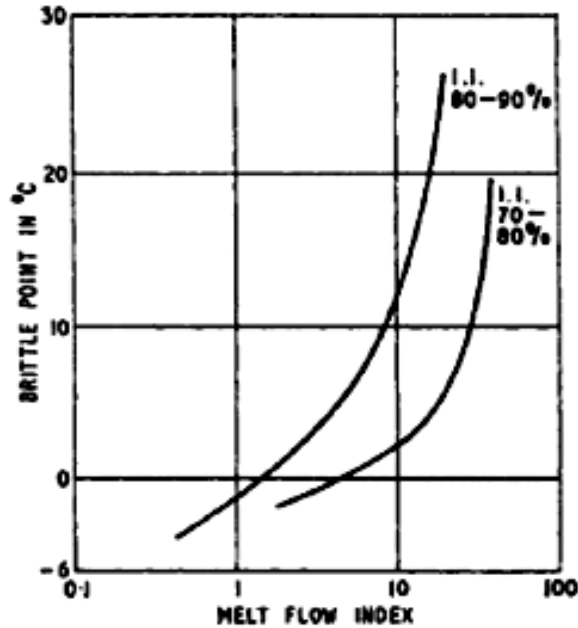


Fig 3.4 Variation of ASTM D 746 Brittle point with MFI and Isotactic Index.

(Source: Plastic Materials by Brydson, J.A)

Atactic polypropylene can be obtained as a by-product during the production of isotactic polypropylene process. Complete Atactic polypropylene is an amorphous material with a very small amount of crystallinity, having a specific gravity of 0.86 g / cm<sup>3</sup> and the average molecular weight of viscosity in the range of 20,000 ~ 80,000. The appearance of the Atactic polypropylene is between a wax and a rubber, and it swells substantially in aliphatic and aromatic hydrocarbons at room temperature. Now, Atactic polypropylene is used as compounds of coating for roofing materials, road construction, etc.

Syndiotactic typed polypropylene was introduced to the market by year 1990 and it was produced by a new metallocene system and polypropylene grafted with styrene and /or maleic anhydride. Currently syndiotactic polypropylene polymer is more expensive than other types of polypropylenes both because of the costs of catalyst and the small scale of production.

Syndiotactic Polypropylene are generally softer, tougher and more transparent than isotactic materials but exhibit similar fusion points. Please see the table 3.1.2 below[10].

Table 3.2 Comparison of some properties of Syndiotactic and Isotactic Polypropylene

	<b>Syndiotactic PP</b>	<b>Isotactic PP</b>
Density (g/cm <sup>3</sup> )	0.9	0.9
Elastic Modulus (MPa)	61	16.5
Impact Strength (kJ/m <sup>2</sup> )	80	16
Opacity (%)	1.7	85
Crystallinity	30~40	40~60
T <sub>m</sub> ( <sup>0</sup> C)	168	163

(Source: Plastic Materials by Brydson, J.A)

### 3.1.2 Preparation of Polystyrene

Most of the commercial styrene is prepared by the Dow process or a similar system. The process involves the reaction of benzene with ethylene into the ethylbenzene, its dehydrogenation to styrene. Therefore, it is useful to look at this process in each of the three stages.

#### Preparation of the ethylbenzene

The ethylbenzene is prepared by reaction of ethylene with benzene in the presence of a Friedel-Crafts such as aluminum chloride at about 95 ° C.



Fig: 3.5 Reaction of Benzene with Ethylene<sup>[10]</sup>

To improve the efficiency of the catalyst, ethyl chloride is added to which produces hydrochloric acid to the reaction temperatures.

The purity of ethylene is not critical provided that acetylene is not present. Normal purity of ethylene used is about 95%. The purity of the benzene is slightly higher than 99% and it is important here that the sulfur as an impurity, is less than 0.10%. To reduce the amount of side reaction and minimize the production of polyethylbenzenes, molar ratios of the raw material and the products are approximately as shown in the following equation (Fig 3.6).

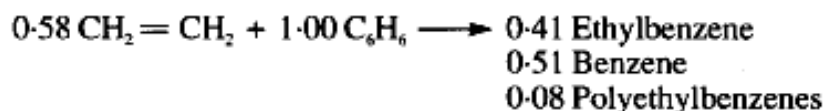


Fig. 3.6 Molar Ratios of the raw Material and the products in the production of polyethylbenzene<sup>[10]</sup>.

The products are cooled when passing through the reaction chamber, which comes in the form of a complex with hydrocarbons and settles. The ethylbenzene, benzene and the polyethylbenzenes are separated through fractional distillation, the ethylbenzene has a greater than 99% purity. The polyethylbenzenes are dealkylated by heating to 200 ° C in the presence of aluminum chloride and these products with the same benzene are recycled.

The plants were installed by some manufacturers to produce ethylbenzene via a catalytic reforming process that converts aliphatic hydrocarbons into mixture of aromatic hydrocarbons. This can be later split to give benzene, toluene, and a 'fraction of xylene' from which one can obtain the ethylbenzene.

### Dehydrogenation

Styrene is produced from the ethylbenzene via dehydrogenation process

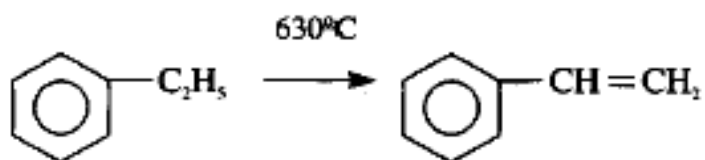


Fig 3.7 Dehydrogenation Process of ethylbenzene<sup>[10]</sup>



It is an endothermic reaction in which an increase in volume accompanies the dehydrogenation. The reaction is therefore favored by a reduced pressure operation. In practice, the steam is by passed through with the ethylbenzene to reduce the partial pressure of the latter instead of performing a reaction at high temperature under partial vacuum. Using catalysts chosen such as magnesium oxide and iron oxide, one can get a conversion of 35 to 40% per pass, with yields of 90 to 92 percent.

### **Purification of Styrene**

Dehydrogenation reaction produces a "crude styrene", which consists of approximately 37.0% of styrene, 61% of ethylbenzene and about 2% aromatic hydrocarbons such as benzene and toluene. The purification of styrene is made quite difficult by the fact that the boiling point of styrene (145.2<sup>0</sup>C) is only 9<sup>0</sup> C higher than that of the ethylbenzene and because of the strong tendency of styrene to polymerize at high temperatures. To get a successful distillation, it is so necessary to provide inhibitors suitable for styrene to distill under partial vacuum and use specially designed distillation columns.

In the same process, the crude styrene is first passed through a pot containing elemental sulphur which dissolves enough to become an inhibitor of polymerization. Toluene and Benzene are then removed by distillation. The ethylbenzene is then separated from the styrene and tar through two distillation columns, each with temperatures above 50 ° C and below temperatures of 90 ° C under vacuum for about 35 mmHg. The tar and sulphur are eliminated by a final distillation column and styrene is inhibited at all times by the addition of 10 ppm of t-butylcatechol, who less harmful effects on the final polymer than sulfur. Styrene is a colorless liquid with a pleasant smell when pure, but with an unpleasant smell due to traces of aldehydes and ketones, if allowed to oxidize by air exposure. It is a solvent for polystyrene and many synthetic rubbers, including SBR, but there is a mutual solubility in water is very limited. The Principal properties of pure styrene is given below in Table 3.1.3.

Table 3.1.3 Principal Properties of pure styrene<sup>[10]</sup>

Molecular Weight	104.14
Density at 25 <sup>0</sup> C	0.9010 g/cm <sup>3</sup>
Refractive Index at 25 <sup>0</sup> C	1.5439
Boiling Point	145.2 <sup>0</sup> C
Vol. Shrinkage on Polymerisation	17%

[Source: Plastic Materials by Brydson, J.A]

Styrene participates in numerous number of chemical reactions. In particular, it has a strong tendency to polymerize when exposure to ultraviolet light or on heating.

### **General properties Polystyrene**

Polystyrene is a rigid and rigid transparent thermoplastic that emits a characteristic metallic ring when dropped. It is free of odour and taste, burns with a sooty flame and has a low specific gravity of 1,054. Because of its low cost, its moldability, its low moisture absorption, and good dimensional stability, good electrical insulation properties, its color and its reasonable chemical resistance, it is widely used in injection moulding and vacuum forming. In addition, the low thermal conductivity has been used in polystyrene foam which used for insulation. The main limitations of the polymer are the brittleness, the inability to withstand the temperature of boiling water and its mild resistance to oil.

The mechanical properties of polystyrene depend to some extent on the nature of the polymer (e.g. molecular weight), on the process of preparation of the sample to be tested and the test process, as is the case for all plastic materials.

#### **3.1.2.1 Forms of Polystyrene**

There are 3 different forms of polystyrene such as

1. Expanded Polystyrene Foam (EPS)
2. Extruded Polystyrene Foam (XPS)

### 3. Extruded Polystyrene

First of all, each of these forms of polystyrene has its own variety of applications. Similarly, polystyrene can be used in explosives linked to the polymer. Expanded polystyrene density is an important factor, because it can go from 25 kg / m<sup>3</sup> to 200kg/m<sup>3</sup> and depends on the amount of gas used to create foam.

Extruded polystyrene foam (XPS) is a rigid insulation in high performance with a closed-cell structure that makes it impermeable to moisture. It is an insulator which is available in different grades for specific applications requiring high loads or a resistance to excessive moisture levels and superior thermal performance. The insulating panels in extruded polystyrene are made from General Purpose Polystyrene (raw GPPS material), which comes in the form of pellets typically of 3 to 5 mm in diameter.

Packaging is one of the main uses of polystyrene, there are also different types of packaging. It is important to know the difference between some of these polystyrenes, namely General-purpose Poly styrene (GPPS) and High Impact Polystyrene (HIPS).

#### **3.1.2.1 General Purpose Polystyrene – GPPS**

General Purpose Polystyrene, also known under the name of crystal clear polystyrene, is entirely transparent and rigid, and is widely used in food packaging applications or the jewelry boxes. Long ago, the most common application of GPPS were observed in cases where the CD included. In addition to being consistent with FDA standard, it is also low cost, x-rays resistant, without smell or taste and easy to deal with. GPPS is better suited for storage and packaging because of its transparency. The GPPS can be molded in exciting shapes like that of a treasure chest or a heart to serve boxes of sweets for gifts. Transparent and rigid boxes are perfect for storage, allowing the consumer to see the contents of the box. As GPPS complies with the FDA, it is completely safe to use for the storage of food.

Here are other properties that emphasize the advantage of GPPS:

- FDA Compliant – non toxic
- Good dimensional stability
- Low cost compared to other materials
- Easy to paint, glue and print on
- Excellent aesthetic qualities

### **3.1.2.2 High Impact polystyrene (HIPS)**

High Impact Polystyrene also called TPS - Toughened Polystyrene. For many applications, the GPPS might be considered too brittle. Therefore, the polystyrene mixed with polybutadiene rubber would give HIPS in solution polymerization. Today, the practice is first to dissolve the rubber in the styrene monomer, then polymerize styrene in the usual way. Thanks to this process, the resulting mixture will contain not only the rubber and polystyrene, but also a grafted polymer where small side chains of styrene were attached to the rubber molecules. This gives a marked improvement of impact forces that can be obtained.

Polystyrene and rubber can be mixed in several ways. Originally, the ingredients were compounded in a two-rolled mill, in an internal mixer grinder or in an extruder. The impact strength of the products, however, was little better than the unmodified polymer. The mixture of SBR latex and latex of polystyrene, followed by the co-coagulation and drying, has also been used in the past, but once again, the improvement is marginal.

It has been shown that with mixtures of polystyrene and SBR, rubber should exist in discrete droplets, of less than 50 microns in length, where a good finish is required, in the matrix of polystyrene. We think that, in one such form, the rubber can reduce the spread of cracks or crack propagation and the fracture in various ways.

A high-impact polystyrene can have seven times the ordinary polystyrene impact resistance, but about half of the tensile strength, a lower hardness and a softening point

about 15 ° C lower. Due to the rubber content, there may be a reduction in stability to light and heat and stabilizers are normally incorporated.

A tough transparent sheet can be produced by mixing the standard polystyrene with a copolymer sequenced in an extruder in ratios from 80: 20 to 20:80, depending on the application of the product subsequently thermoformed from the sheets. For example, the sheet for thermoforming of egg tray will not require the same level of impact strength than that required for the jars of jam.

### **3.1.3 Properties of PP and PS.**

#### **Polypropylene:**

**Acronym;** PP

**Class** Poly ( $\alpha$ -olefins)

**Structure:**  $-\text{CH}_2-\text{CH}(\text{CH}_3)-$

**Molecular weight of repeat unit:** 42.07 g/mol

**Major Applications:** Fiber, Slit tape, Cast and biaxially oriented film, Containers and closures, automotive interiors trim, appliance housings and components, component in elastomeric blends with polyethylene and olefinic rubbers.

**Properties of Special Interest:** Low Cost; easily processed by injection molding, extrusion, and spinning; can be oriented; excellent resistance to chemicals; low color; can be stabilized to provide good thermal aging stability; moderate strength and stiffness; good toughness when impact modified either in the reactor or by compounding; excellent fatigue resistance; modest clarity.

**Preparative Techniques:** Ziegler-Natta polymerization with titanium halide/aluminum alkyl catalyst and, optionally, ether, ester, or silane activator. Catalyst may be deposited on a magnesium chloride support. Slurry and gas phase processes are used. Catalyst systems based on metallocenes are under development. Typical comonomers are ethylene and 1-butene.

Table 3.1.4 Melt flow Index vs Molecular Weight Mw of Polypropylene

Melt Flow Index	Molecular Weight Mw (g/mol)
0.63	646,000
2.9	412,000
11.9	297,000

(Source: Plastic Materials by Brydson, J.A)

**Shrinkage factor:** Total Shrinkage 1.5 – 1.8 % (PP Semi crystalline plastics are having very high shrinkage than amorphous plastic such as Poly Styrene).

Table 3.1.5 Properties of Polypropylene

Property	Units	Conditions	Value
Density	g/cm <sup>3</sup>	298K, Crystalline Phase	0.936~ 0.946
		298K, Amorphous Phase	0.850~ 0.855
		298K, Commercial Material	0.90 ~ 0.91
Melt Flow Index	g(10min) <sup>-1</sup>	ASTM D 1238	0.2~500
Ignition Temp	K	Calculated from Critical heat flux data	736
Thermal Conductivity	W/mK	293K	0.12 ~ 0.22
Decomposition Temp	K	TGA in helium	623
Degree of Crystallinity	%	DSC	50~70
Tg	K	DMA (30Hz)	283.7
Tm	K	100% Crystalline	~459

(Source: Polypropylene Handbook by Moore, E.P.Jr)

**Polystyrene:**

**Acronym:** PS

**Class:** Vinyl Polymers

**Structure :**  $-\text{CH}_2-\text{CH}(\text{C}_6\text{H}_5)-$

**Major Applications:** One of the most widely used Plastics, having applications in industries of packaging, appliances, construction, automobiles, electronics, furniture, toys, housewares, and luggage.

**Properties of Special Interest:** Crystal Clear thermoplastic, hard, rigid, free of odor and taste, ease of heat fabrication, thermal stability, low specific gravity, excellent thermal and electrical properties for insulating purpose, and low cost.

**Shrinkage factor:** Total Shrinkage 0.4 – 0.7% [12]. (Poly Styrene is amorphous plastic therefore, less shrinkage than crystalline plastics such as PP).

Table 3.1.6 Properties of Polystyrene

Property	Units	Conditions	Value
Tg	K	-	373
Tm	K	-	513
Tensile Modulus	MPa	Unoriented Oriented monofilament	3200~3400 4200
Compressive Modulus	MPa	-	3000
Shear Modulus G	MPa	-	1200
Bulk Modulus	MPa	-	3000
Flexural Modulus	MPa	-	3100
Tensile strength at Break	MPa	-	30~60
Elongation at Break	%	-	1~4
Flexural Strength	MPa	-	95
Compressive Strength	MPa	-	95
Impact Strength (Izod)	J/m	ASTM D256	19.7
Ignition Temperature	K	-	675

(Source: Plastic Materials by Brydson, J.A)

## **3.2 Effect of Fillers on Properties of Plastics & Process**

### **3.2.1 Calcium Carbonate as a Filler**

Calcium Carbonate can be found as marble or limestone. Calcium Carbonate is the popular filler in thermoforming industry. Calcium carbonate serves as a functional additive in thermoforming. One of its major applications is for manufacturing polypropylene trays, which are widely used in the packaging of food and disposable items. Other applications include plant – propagation trays, automotive parts, construction material, household items, and plant pots, which are described above. Many advantages can be obtained by using calcium carbonate in thermoforming applications.

#### **Advantages of Calcium Carbonate**

Higher thermal diffusivity and improved mechanical properties are the most important advantages of calcium carbonate in thermoforming applications. Higher thermal conductivity results in higher output. Since the end product has better mechanical properties, it can be manufactured with lower wall thickness, which allows raw material costs to be reduced.

#### **Effect of Calcium Carbonate on Sheet Extrusion Process**

Calcium carbonate modifies the properties of polymer melts. The rate of all heating and cooling processes is determined by the material's thermal diffusivity. Calcium carbonate has a thermal diffusivity that is many times greater than that of most polymers. It is therefore an effective additive for increasing the thermal conductivity of polypropylene. This results in faster melting in the extruder, faster cooling on the cooling roll, and a more homogeneous melt temperature. The faster cooling permits faster machine speeds as calcium carbonate reduces cooling capacity. Another benefit of calcium carbonate is the low coefficient of thermal expansion, which remains constant over a wide temperature range. In contrast to semi-crystalline polymers, such as PP, thermal expansion is greater at the melting point.



Calcium carbonate – modified polymers therefore have a lower melt volume than the virgin polymers. This fact, together with decreased adhesion to barrel wall, has found to reduce torque and pressure during extrusion. Overall, modification of thermoplastics with calcium carbonate offers the opportunity to significantly increase output— to an extent depending on machine equipment and processing conditions.

The introduction of calcium carbonate into the polymer matrix reduces the amount of viscoelastic material in the formulation. Thus it reduces typical viscoelastic phenomena, such as die swell, neck - in, and shark skin. The mineral particles increase the internal shear forces, and therefore the melt viscosity. This improves the dispersion and distribution of other additives.

### **Effect of Calcium Carbonate on Thermoforming Process**

The extruded sheets can be thermoformed from the roll, either directly or after storage. In the latter case, semicrystalline polymers, such as PP, are often heated in two stages, first preheated in circulating air oven and then by a main heater. In general, only a thin surface layer is heated directly. The heat is transported by thermal conduction into the core of the film. The higher thermal diffusivity of calcium carbonate therefore improves the heating process. On roll - fed machines equipped with film preheating, the cycle time is determined by the cooling time. The higher the cooling rate, as made possible by calcium carbonate, the more significantly the output is increased. Productivity can be increased by 12%, in some cases even by 35% or higher.

Semi-crystalline polymers such as PP have a very narrow processing window, which is the result of the sharp transition from a rubbery elastic solid to a relatively low viscosity melt. The addition of calcium carbonate modifies the melt rheology, imparting a pronounced pseudoplastic behavior. That means that the melt has a high viscosity at low shear rates. Consequently, there is a broader temperature range in which the material can be successfully shaped. Moreover, the pseudoplasticity, together with the lower thermal

expansion, reduces the sagging of the film. Stretching during film transportation, which is necessary for most semicrystalline plastics, can be reduced accordingly.

The deformation behavior of the molten polymer changes from viscoelastic to plastic. Correspondingly, the definition (shaping fidelity) of the parts is visibly improved. This leads to more clearly pronounced surface details and sharper edges. The lower thermal expansion, together with the lack of volume expansion at the melting point, reduces the processing shrinkage of the formulation. Correspondingly, the mold can be designed with less allowance for shrinkage.

The calcium carbonate particles embedded in the plastic surface reduce the forces of adhesion between the material and the mold, which facilitates demolding. Punching out is also made easier in many cases.

### **3.2.2 Property Improvements**

In thermoforming applications, between 10% and 40% calcium carbonate is added. The mineral additive increases the stiffness of polymers. While the yield stress of identical parts is reduced, the cross-machine direction of breaking elongation is significantly increased, resulting in more durable final products. The improved impact strength and reduced directional dependency of the breaking stress are principally the result of the increased isotropy of the material. The aspect ratio of calcium carbonate, defined as the ratio of particle length to thickness, is approximately 1:1. That makes it impossible for the particles to align themselves in a preferred direction. In addition, the better homogenization by minerals reduces the molecular orientation of the polymer. This effect can be demonstrated by infrared studies. A further advantage of the lower orientation, synonymous with less frozen tension, is significant reduction of the warpage of the thermoformed parts.

In many cases the maximum load (top load) that the thermoformed parts can withstand is increased, especially in applications with a low forming ratio. The plant pot has a high deep draw ratio and requires certain precautions in processing, especially because of the

greater amount of calcium carbonate in the polymer matrix (more than the described 35%). The improvement of mechanical properties often allows the film thickness to be reduced while maintaining stability requirements.

Film containing calcium carbonate is gauged down in thickness to the extent that the thermoformed parts are just as heavy as the virgin PP regardless of the higher density of the calcium carbonate in the film. The results show that the top load of the thickness-reduced film with 20% calcium carbonate is even higher than that of the virgin PP. Molecular orientation and crystallinity have an influence on all mechanical properties. Both parameters are affected by the processing conditions and the thermoformed part's design. The heating and cooling conditions, in particular, as well as machine speeds during extrusion and thermoforming, offer the possibility of optimizing the mechanical properties. In Europe, if a film must be in direct contact with acidic foods ( $\text{pH} < 4.5$ ), use of multilayer films is recommended with calcium carbonate in the core layer to meet EU limits of migration.

The more that the polymer is replaced with the calcium carbonate, the more tortuous the diffusion path becomes, but the barrier properties of the parts produced are better. The polymer is more permeable than calcium carbonate (impermeable).

Permeability to water vapor and oxygen fall with increasing calcium carbonate content. In the absence of pigments, calcium carbonate produces an opaque white color in transparent plastics. Black, dark-colored, cream, or white pigmented formulations do not require an additional color to the masterbatch. If fluorescent colors are required, especially when pigment systems with relatively low opacity such as red and blue are used, it may be advisable to use multilayer sheets.

Polyolefins have very low surface energy. Anchorage of printing inks is therefore restricted. The addition of calcium carbonate improves printability, since it has a high surface energy and a certain micro roughness. A matting effect may occur if very high amounts are added.

### **Cost Reduction**

While polymer prices have risen greatly in recent years, the price of calcium carbonate has stayed relatively stable. This opens up the possibility for significant cost savings, besides the improvement in mechanical properties that often results and the reduction of film thickness. The increased stiffness achieved with calcium carbonate has interested many researchers.

The use of calcium carbonate provides several advantages in thermoforming applications. The improved thermal efficiency throughout the entire processing chain pays off in higher output. The improved mechanical properties result in reduced wall thicknesses. Overall, the introduction of calcium carbonate is a very economically attractive solution for film manufacturers and thermoformers.

### **3.2.3 Effect of Filler on Extrusion Process**

The addition of fillers can significantly change the processing properties of a polymer. Viscous dissipation is increased with increasing viscosity, density, and thermal conductivity, and by decreasing specific heat.

Adding fillers to the polymer mix of PP or PS affects all of these parameters. The generation of heat in the polymer itself depends on its thermal properties, viscosity, and the conductive heat transfer to or from the extruder. By adding most fillers, the amount of energy required for viscous dissipation is greatly reduced in relation to the mass output.

Polymers are poor conductors of heat compared with Calcium Carbonate fillers. Moreover, polymers require more energy to raise their temperature (specific heat). Additionally, most fillers have a significantly greater density than the common polymers, giving them more “thermal bulk.” Finally, addition of fillers usually increases the compound’s viscosity as compared with the neat polymer.

**MORE VISCOSITY=MORE SHEAR STRESS**

Increasing viscosity increases the shear stress in the extruder, which is the basis of the mechanical-to-thermal energy conversion in a single-screw extruder. Stated simply, much of the effect of fillers in a single screw extruder can be described by a combination thermal property called diffusivity.

Diffusivity is a good indicator of the ability to raise the temperature and the ability to transfer heat.

$$\text{Diffusivity} = \text{Thermal Conductivity} / (\text{Specific Heat} \times \text{Density})^{[4]}$$

The units in the SI system are:

$$m^2/sec = W/(m \text{ } ^\circ K) \div J/(kg \text{ } ^\circ K) \times (kg/m^3)$$

Information on materials' thermal properties can be found on the internet and a relatively accurate average of each thermal property for the polymer and fillers can be determined by simple proportioning of the thermal properties to the mix percentage.

As an example, a polypropylene filled with 40% calcium carbonate would have a diffusivity approximately 3.5 times greater than that of neat PP. This means it will take much less energy from the extruder drive and will transfer energy much faster than the neat PP in relation to the mass of the output. This means lower motor load and quicker, more uniform melting than neat PP at the same output (kg/hr).

Higher diffusivity has implications throughout the whole process. As noted, the extruder drive load will be lower at the same output and the melt temperature will be more uniform. Cooling will be easier for the same reason. Heat will go in faster and easier and will come out faster and easier.

### 3.2.4 Extruder and Mold Wear Caused by High Filler load in Plastic Compound

Calcium carbonate-filled plastic materials processing equipment will be worn barrel, screw, die, die, etc., which will use the benefits of cheap fillers were discarded equipment damage caused by early offset, or even more harm than good. On the one hand we have the answer is wear the problem did exist, we must face it and take the necessary measures to deal with, on the other hand we must be clear that the calcium carbonate-filled plastic on steel in contact with the wear is imminent.

In the commonly used inorganic mineral powder materials, calcium carbonate hardness is relatively high, but compared with the plastic processing equipment mainly used for metal parts for the nitrided steel is a lot of difference, by wear and tear theory metal hardness 1.25 times higher than the abrasive hardness above, is a low wear.



Fig 3.8 Worn out cavities (Source: Dana Tool Room WLL)



Fig 3.9 Worn out screws(Source: Dawanco Ind. WLL)

### **3.2.5 Effect of Calcium Carbonate on plastic aging properties**

As polymers, in the light, heat and other environmental conditions of molecular chain rupture occurs, and may produce graft or cross-linking reaction, the macro performance of the mechanical properties decrease, a phenomenon known as aging.

Experiments show that calcium carbonate containing polypropylene film and pure polypropylene film in the sun exposure found that pure polypropylene film, indicating that the presence of calcium carbonate aging of polyethylene films have some promote the role,

### **3.2.6 Forming the problem size shrinkage**

Better use of cost-effective in the modification of plastic instead of the traditional plastic materials (such as the PP filled with mineral powder instead of ABS), in addition to performance and appearance should meet expectations, the molding shrinkage size cannot be ignored important problem.

Molding shrinkage rate of change in size will affect the molding products, geometry and size. For pure PP is based on material injection molding mold shrinkage of 1.5 to 2.0% in the design of the (above-mentioned study, the same conditions, size of pure PP, molding shrinkage rate 1.7%), calcium carbonate, if filled, the molding Processing shrinkage of smaller size, so for those who depend on the cooling shrinkage mold release products, locking occurs, surface scratches and deformation problems.

### **3.3 Thermoforming Process**

Thermoforming Process categorized into two sects. Thin Gauged thermoforming (0.005”~ 0.030”) which is fed with roll feed process system for continuous processing specially in Packaging Industries. And sheet thickness above 0.031”~0.310” are called heavy gauged thermoforming which cut sheets are used for forming during the process. Though the Global packaging industries revenue has increased to \$530Billion in the year 2014, yet the research papers released based on packaging industries are much less. Further the technology and technical studies are limited and confined only to the manufacturing companies. Therefore, the information regarding thermoforming process and procedures are very limited.

Thermoforming or Thermoformed products – The word explained the process itself. Which means form through heat. That is semi-finished goods are produced with heat and forming to a shape. When thermoplastic sheets such as PS or PP Sheet is heated to required temperature it becomes soft and sags like rubber sheets and it can be stretched to the required shapes. The integrated process chain in Thermoforming is given below.



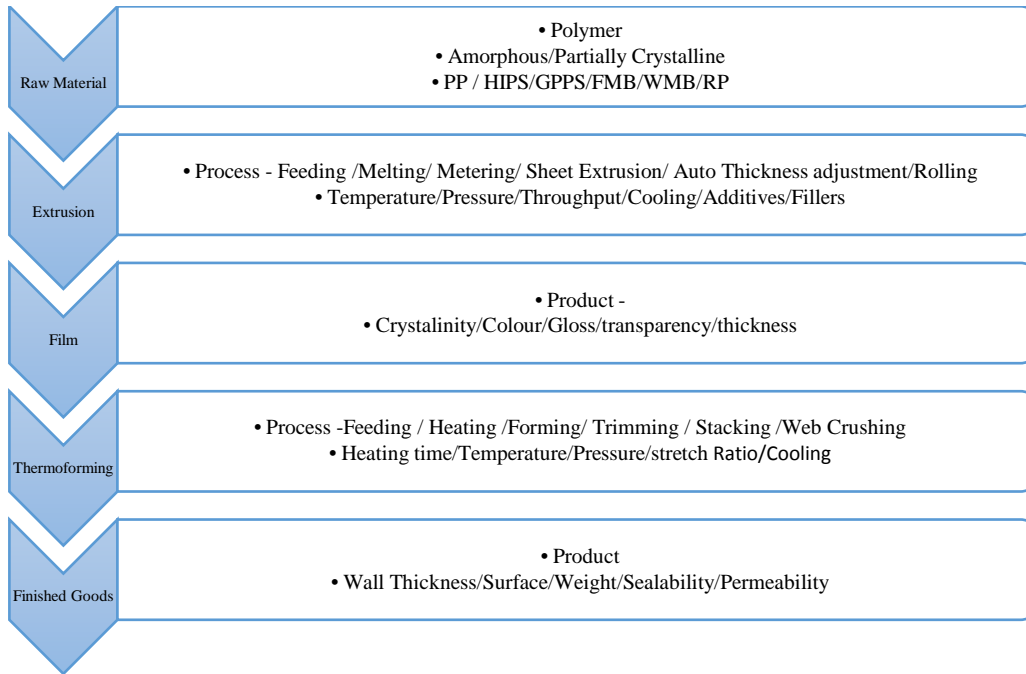


Fig. 3.10 Flow chart of Thermoforming Process

As explained above only thin gauged or Roll fed thermoforming process is discussed in this report.

The Configuration of Thermoforming Machine is given below.

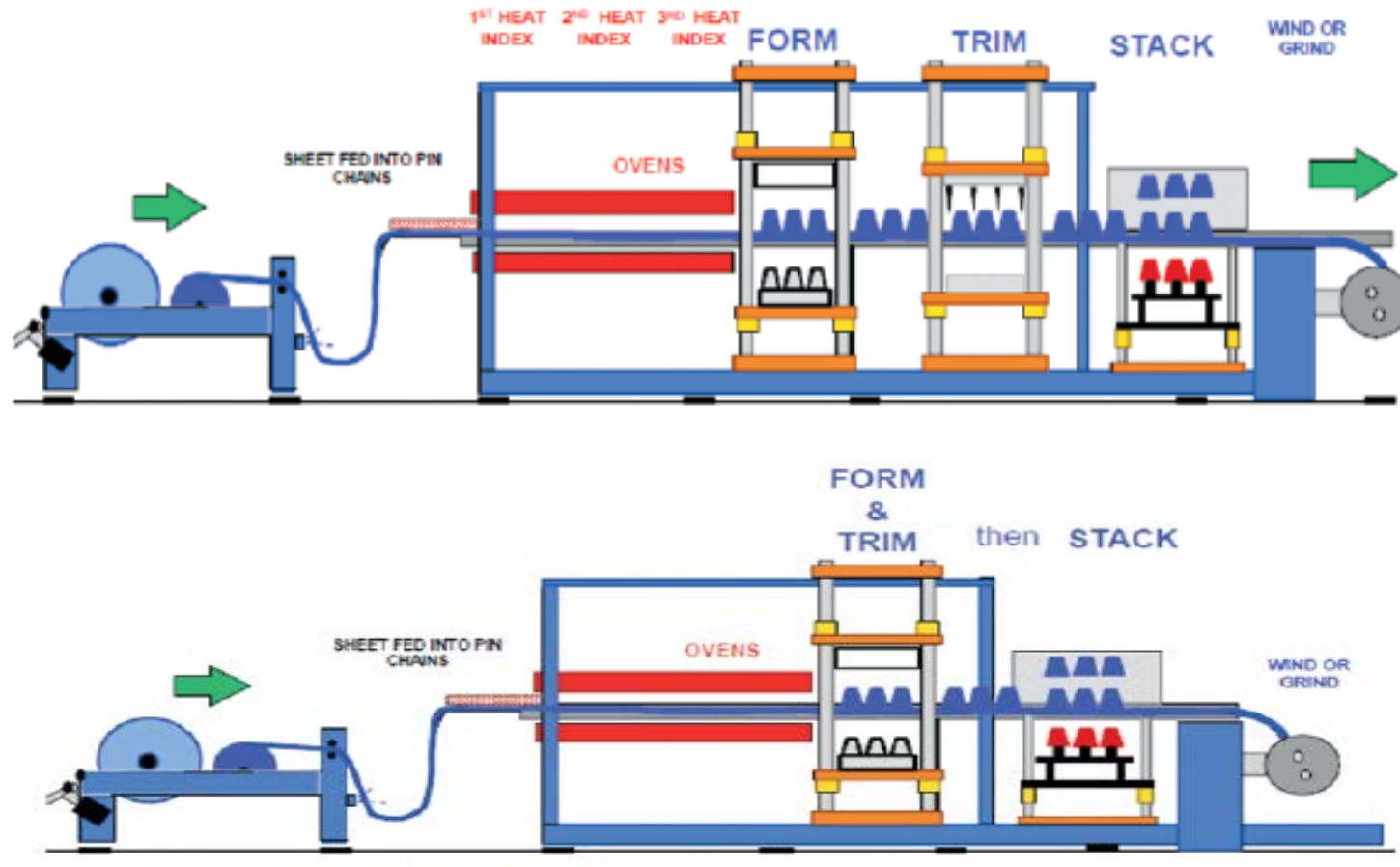


Fig: 3.11 Configuration of Thermoforming Machines (Source: Report on Dusseldorf K2013)

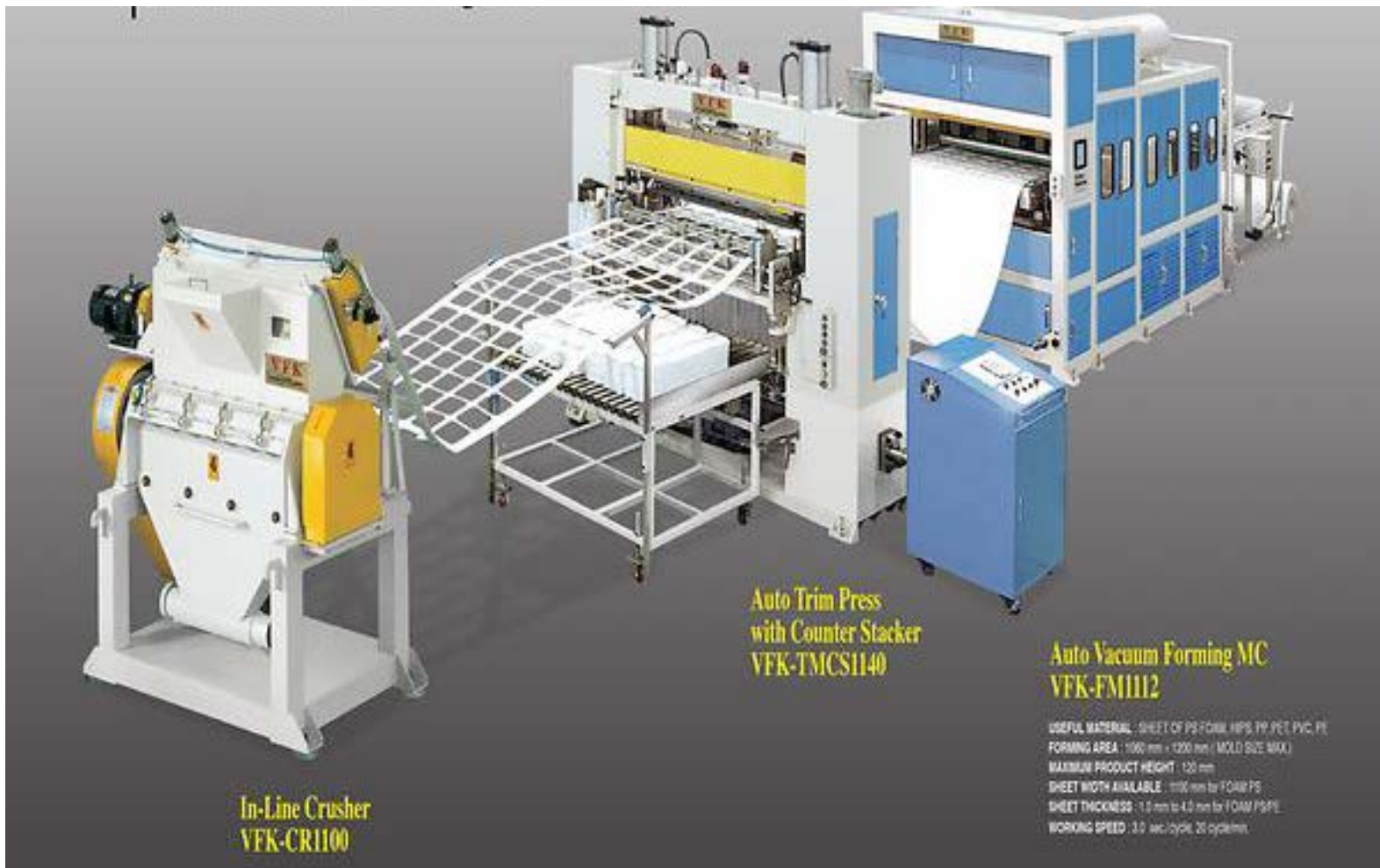
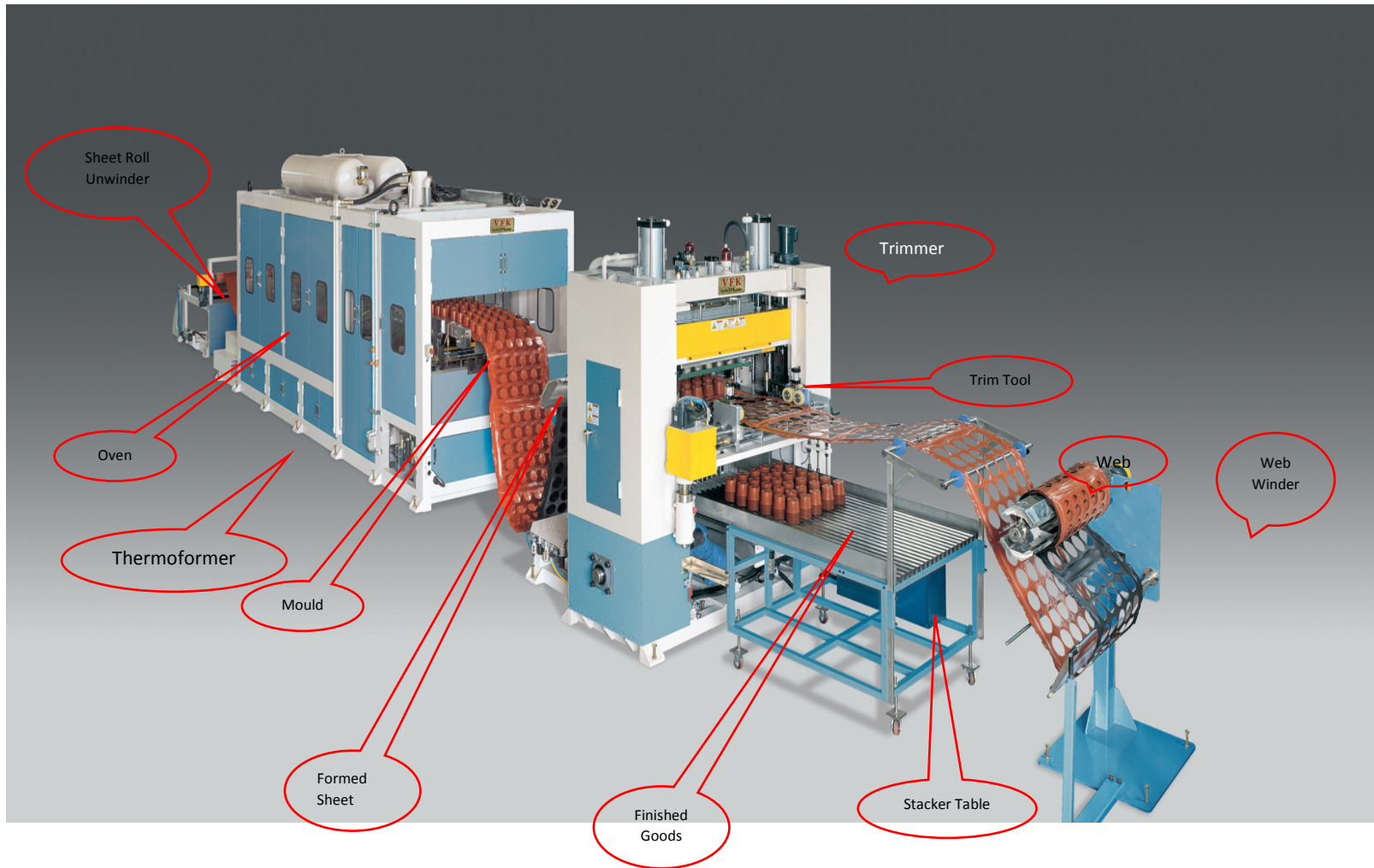


Fig. 3.11.1. VFK Corporation Auto Vacuum Forming Machine with in line crusher (Source: VFK Corp.)

**VFK Corp – from Korea. – Pressure Thermoforming Machine.**



**Fig 3.12 VFK Corporation Pressure forming Machine (Source: VFK Corp)**

In the field of Thermoforming there are three types of Thermoforming techniques are available. They are:

1. Pressure forming.
2. Vacuum Forming
3. Mechanical forming.

### 3.3.1 Pressure Forming

In the process of Pressure Forming[12] – plastic sheet is clamped between pinned chain and then heated till it becomes soft and sagged then the sheet will be subjected to the special mould in which Rim edge are beaded to prevent air leak. With around 6 ~ 8 Bar pressure product is formed. Pressure forming techniques can provide low cost and consistent parts with precise details continuous products. Earlier the extruded sheets were subject to embossing roller to get the textures on the sheet. With the advancement of the thermoforming industries now textures are added on to the cavity in order to get the textures on pressure formed products such as cups and plates.

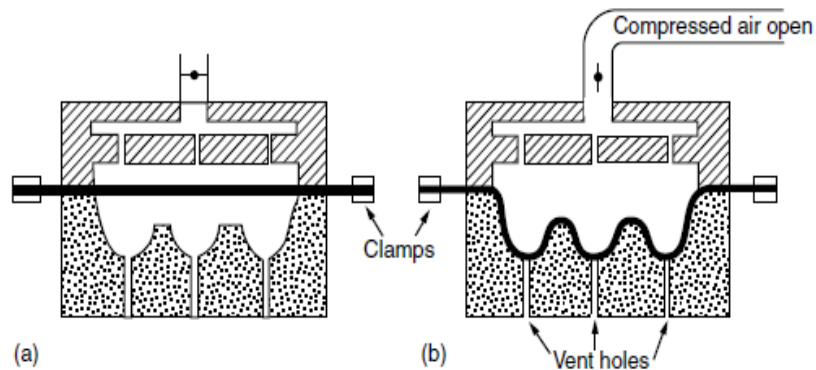


Fig 3.13 Pressure forming (Source: Advance Thermoforming, Sven Engelmann)

- (a) Heated sheet is clamped over the mould cavity.
- (b) Compress air is opened to force the sheet towards cavity to form.
- (c) Vent holes are given to exit the air between cavity and sheet to get better impression to the product.

(d) Water cooling lines are given to mould in order to cool down the cavity to make product rigid.

(e) Once pressure is released the part is move forward to trimmer unit.



Fig: 3.14 Pressure Forming Mold Cavities (Source: Dana Tool Room WLL)



Fig 3.15 Vacuum Forming Mold Cavities with Vent holes (Source: Dana Tool Room Wll)

### **3.3.2 Vacuum Forming**

In the process of vacuum forming - vacuum is applied through vent holes in the mould cavities which sucks air between the heated sheet and the cavity using suction pump. RIM will clamp the heated sheet with cavity. The vacuum is maintained till the part is cooled and becomes rigid. In vacuum forming we get very good intricate shapes and designs due to suction. Further, vacuum forming is much more user friendly than the pressure forming. Unlike in the pressure forming the beading gets damaged during continuous production and need frequent replacement to prevent air leakage. In case of vacuum forming we do not get such issues.

Further, vacuum forming is of two types :

1. Plug Assisted [12] and
2. Straight Cavity forming[12] ( None Plug)

Straight Cavity forming is suitable for mostly flat type of product such as plates and low height tubs etc. and it is not suitable for Cups and bowls as it solidifies prior to reach the base of the cavity and more suction pressure is necessary and there will be uneven wall thickness during deep draw sucking. Therefore, such type of products are produced using plug assisted system. By this way the heated sheet will be pushed towards close to the base of the cavity so that less vacuum pressure is applied as well as uniform distribution of the wall of the product can be achieved.

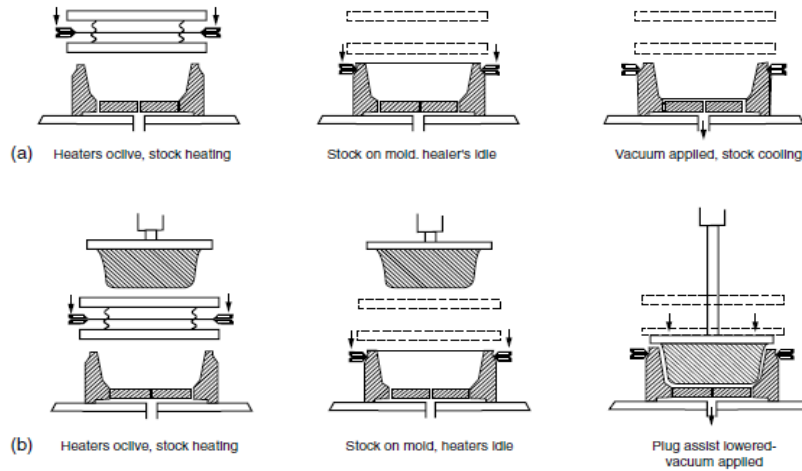


Fig: 3.16 (a) Straight vacuum forming mold. (b) Plug assisted vacuum forming mold (Source: Advance Thermoforming, Sven Engelmann)

Plug assisted system is also two types fixed plug forming and moving plug forming details are described below.

### Fixed Plug Forming

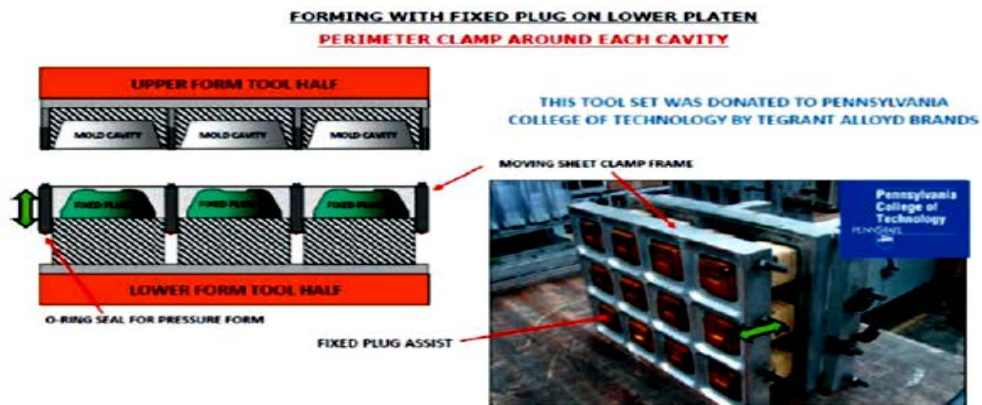


Fig: 3.17 Forming with fixed plugs and sheet clamps

(Source: Report from Dusseldorf K2013)

- (a) Sheet is heated and moved between mould platens
- (b) Female cavities are moved toward sheet line
- (c) Lower mold platen moves up to sheet line



- (d) Sheet clamp rail holds the sheet firmly between moving platens.
- (e) Air tight is formed with the complete closure of the mould and vacuum is turned on.

In fixed plug system product wall thickness manipulation is limited only to the platen closure speed.

### Moving Plug Forming

Moving plug forming is also called third motion plug assist forming technique. In this system platens are closed together very quickly and plug is moved as per the requirement of product wall thickness. In this method wall thickness of the deep drawn product is easily manipulated as well as uniform wall thickness can be achieved throughout the deep drawn products. This will ensure fast production cycle times and possibility of reduction of the product weight and obtain consistent wall distribution.

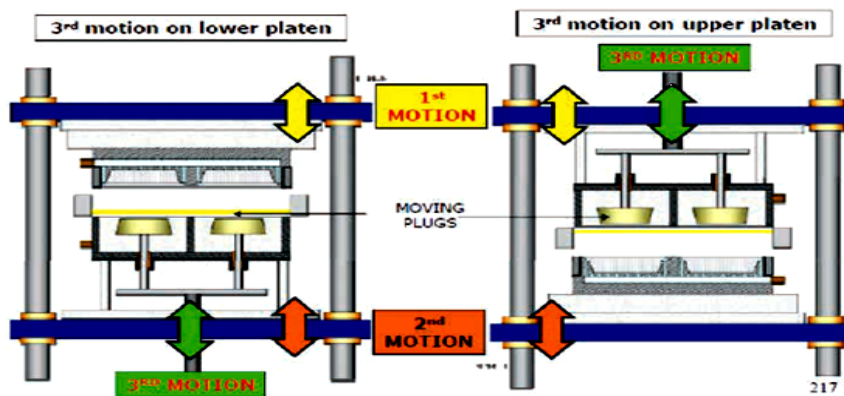


Fig: 3.18 Moving plug assisted forming (Source: Report from Dusseldorf K2013)

### 3.3.3 Mechanical Forming

Mechanical forming techniques are developed neither use vacuum nor pressure in thermoforming. In this case matching male and female mould combination is used to form product. In this process heated sheet is clamped between male and female mold and mechanical force is applied till the part cooled and becomes rigid. There will very good dimensional accuracy in this type of techniques [12].

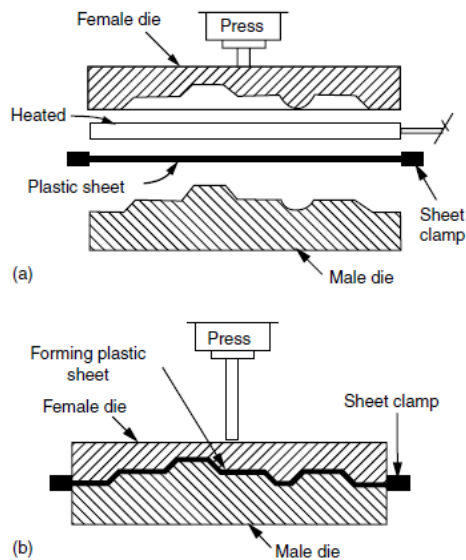


Fig: 3.19 Mechanical forming(Source: Advance Thermoforming, Sven Engelmann)

- (a) Sheet is heated and kept between male and female mould
- (b) Part is formed using mechanical force.

### 3.3.4 Heating and Cooling the Sheet

Heating the Thermoplastic sheet is the first step in the process of Thermoforming. PP / PS sheet is heated to forming temperature depending on the sheet thickness and then formed against the mould, then the formed sheet is cooled to a temperature where the part retains the shape of the mould.

Depending on the material type and the thickness of the sheet, appropriate method of heating is adopted. There are three modes off heat transfer is applied for heating and cooling of sheets.

1. Conductive heat transfer technique.

In GN Plastics Machineries from Canada use Conductive heat transfer techniques in their GN Thermoforming Machines specially for PET sheets. In these machines PET sheets are heated by Heated Platen then it is vacuum formed and trimmed on the same platen. Here thermal conductivity / thermal diffusivity of the polymer material place

vital roll. When Thermal diffusivity is high means more energy dissipate through the polymer sheet more uniformly.

## 2. Convective heat transfer technique.

In Sunwel Machineries from Taiwan uses Convective heat transfer techniques in PP Thermoforming Machines. As the PP material should be preheated prior to heat the sheet for forming. In these machineries PP Sheets are subject to heating chamber with hot air prior to heat to thermoforming temperature. Here heat transfer is depend on the flow rate of heated air. Higher the flow rate of the air greater the energy transfer. Proportionality is called heat transfer coefficient.

Convective heat transfer is applied during the process of cooling of parts. As mould cavity surface is cooled through coolant such as water running through channels in the mould.

## 3. Radiation heat transfer technique

In VFK Machineries from Taiwan uses Radiation heat transfer techniques in VFK Thermoforming machines. In the primary oven they use IR ceramic typed heating elements in the oven to heat the sheet, specially Poly Styrene sheet to thermoforming temperatures.

Final steps in product forming in cooling of parts rapidly to prevent any warps and surface defects. For this smooth and steady flow of water through the mould channels should be circulated.

### **The Forming Temperature of Sheet**

The forming temperature of sheet is depend on the shapes of the part and the experience of the operator. Currently in the industry the operator determines the forming temperature based on sagging of sheet and depth of the parts. If the part is deeply drawn

then more hotter sheet required to form. Which means greater sag or more loss in gloss is required.

Technically Polystyrene has lower forming temperature of  $125^{\circ}\text{C}^{[13]}$  and upper forming temperature of  $180^{\circ}\text{C}^{[13]}$  by comparing this with the glass transition temperature of the  $100^{\circ}\text{C}$  and its normal injection molding temperature of  $220^{\circ}\text{C}$ . Though it has a window of  $55^{\circ}\text{C}$  for forming temperature, in actual practice it is around  $5\sim 7^{\circ}\text{C}^{[13]}$  of window due to the shape of the mould and stretching ratio. In the case PP forming window is much less than PS material. Therefore, more depends on the experience of Operator is vital in Thermoforming industries.

### **3.3.5 Trimming Process**

So far we have discussed the heating and forming of the product. Next step of process of Thermoforming is Trimming Process which also called finished good stage of Thermoforming process. In this stage product is separated from the Thermoplastic sheet and result would be trim scrap or web or skeleton which will be crushed or regrind to reuse in the process.

This mechanical separation of product from the sheet is called trimming process. In thin gauged thermoforming techniques Steel-Rule die cutting is commonly used. Which is forcing a sharpened steel blade perpendicularly into the sheet, cutting or fracturing the sheet into two pieces. In addition to above method Match Metal Punch and dies method also used to trim in certain types of products such as portion cups and drinking cups.

There are two types of trimming applied in the Thermoforming industries [14].

- (a) Form and Trim in Place Method.
- (b) Post Trim Method.

Form and Trim in Place Method.

In this method Trim in process done as soon as Product is formed in the same station.

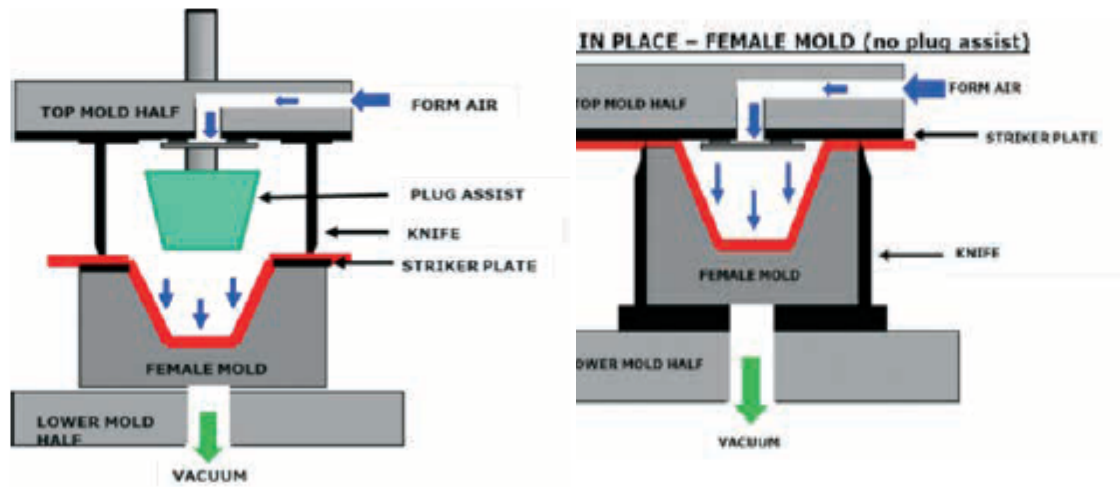


Fig: 3.20 Form and Trim in Place Method (Source: Report from Dusseldorf K2013)

Form and Trim in Place method excellent trim to part accuracy and since the sheet is still in hot condition when the cut is affected burr and powder formation minimizes. Further, advantage of this method will reduce the space requirement for thermoforming.

**Post Trim Method.**

Heat and formed sheet is transferred to a stand-alone trim station to trim and stack separately. This method is utilized in Large bed Thermoformers with very high outputs. These thermoforming beds consists of large no of cavities to form at once. In post Trim method, the trim in speed should be matched the forming speed. Otherwise the forming presses will be idle for portion of trimming time.

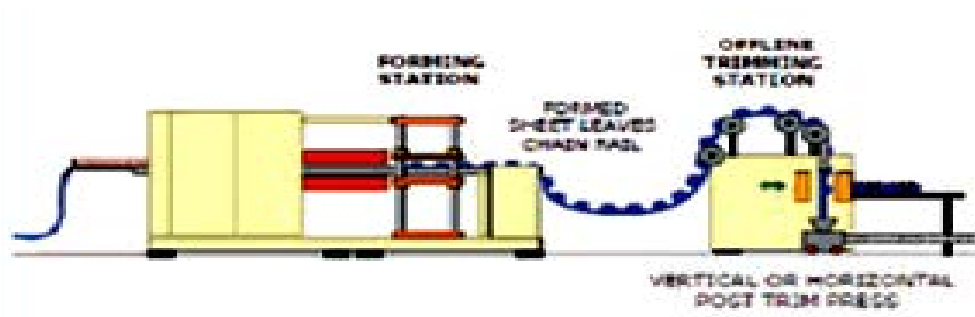


Fig: 3.21 Post Trim Method (Source: Report from Dusseldorf K2013)

### 3.3.6 Stacking or Collection Process of Thin Gauged Products.

Thin Gauged Thermoforming is high volume process. Therefore, counting, Stacking or Collection of these products are vital in these industries. Based on the volume manual collection is not productive. Therefore, automated collecting process is introduced in Thermoforming industries. One part is trimmed from web it is collected on a stacker or robotic picker is used to pick the part from the web. Now it is common to use Robotic picker systems these days. These robotic pickers are having fingers with vacuum tips to pick trimmed parts from the web and collected on the conveyer belt.

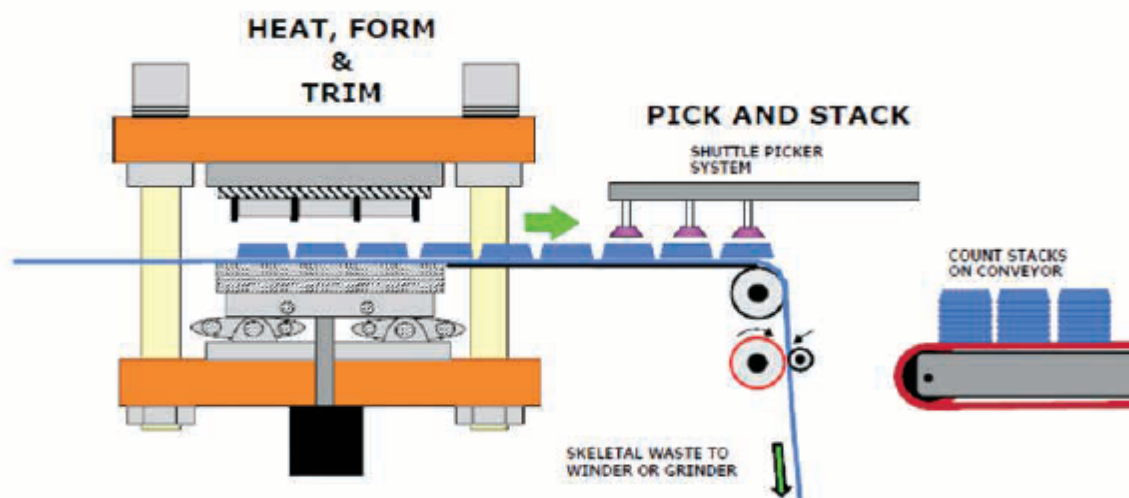


Fig: 3.22 Stacking Process(Source: Report from Dusseldorf K2013)

### 3.3.7 Web Regrinding Process – Scrap Crushing Process

Thermoforming is the secondary process in the Packaging Industries after the primary process of extrusion. Once product is removed from the sheet via trim in process, the balance web or trim scrap or skeleton is subjected to crushing or grinding or granulation process. As the almost 40~50% of the roll sheet is subjected to crushing depending on the type of product. And these scraps are grounded and subjected to cycle separation process in order to separated suitable particle size of 8~10mm to re-use as Raw Material in the extrusion process.

In these granulators the web subjected to rotary blades and cut or shered into small pieces and these pieces are sieved via 6~8 mm perforated curved plate and transferred to cyclone separator via blower pump. And via cyclone separator heavy particles are collected to the separated hoppers or Bags. And fine powder particles are collected to the Jute bags as indicated in the pictures below.

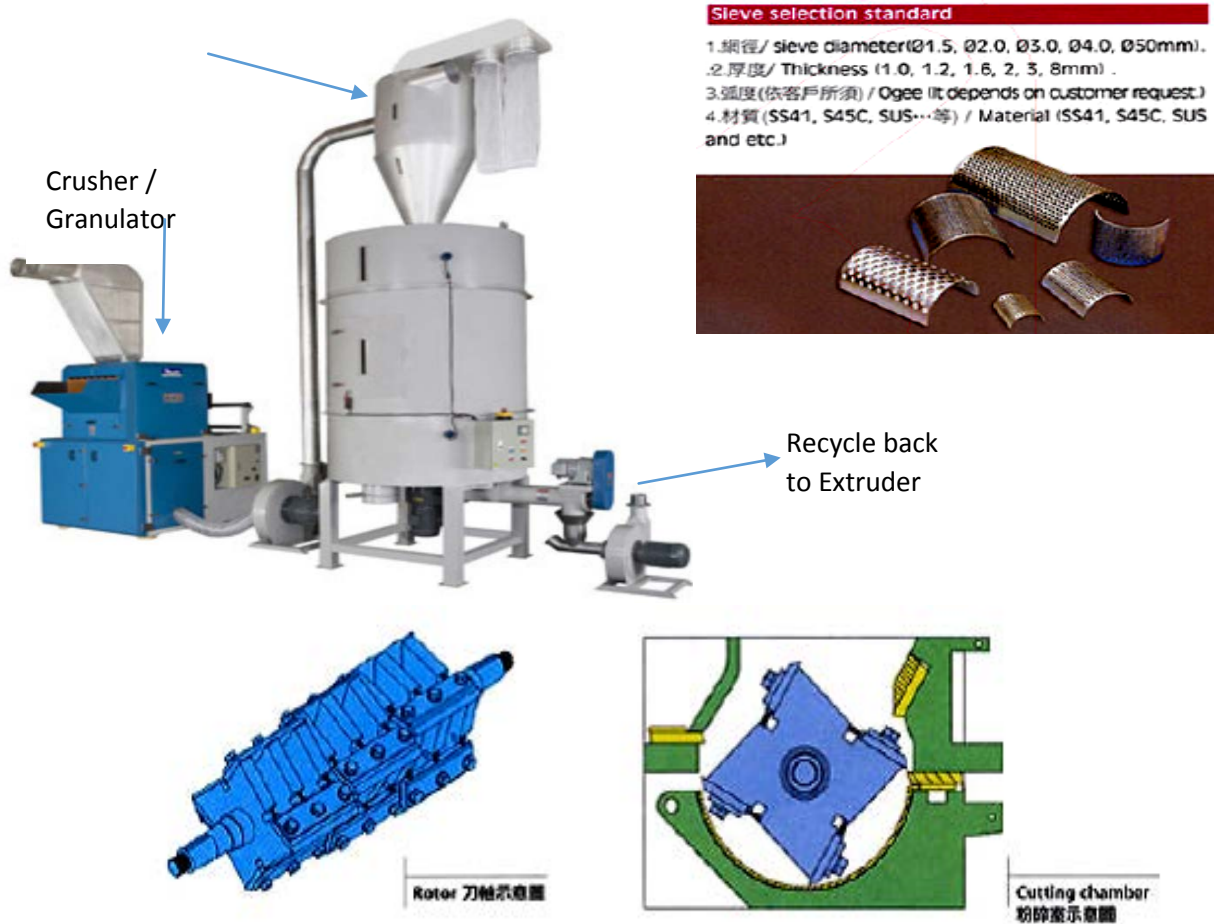


Fig 3.23 Web Regrinding Process (Source: VFK Corp)



Fig 3.24 After Crushing Recycles collected to Bags via Cyclone Separator  
(Source: Dawanco Ind WLL)

### 3.3.8 Closed Loop System of Thermoforming

This is a new concept in Thermoforming Industries – Specially Food packaging Industries. As it is necessary to ensure that the raw material as well as the product should be free from contamination, and with the advent to lean manufacturing the industries has started to introduce closed loop system of operation to prevent any human contact on the raw material such as Recycle during extrusion process.

Process flow of closed loop system is given below.

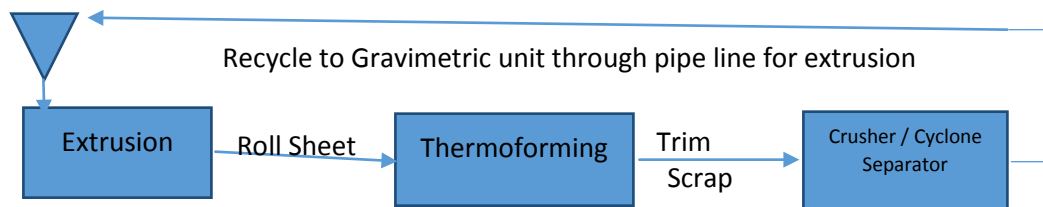


Fig 3:25 Closed Loop System



## CHAPTER 4 - EXPERIMENTAL

### 4.1 Materials Used

1. GPPS – General Purpose Polystyrene PS -125 Grade was used for this experiment in the co-extruder as a lamination layer of the extruded sheet. It was brought from Sabic – Saudi Arabia. This product was made from continuous mass polymerization of styrene monomer. It was crystal –like hard and brittle polymer. PS- 125 was designed for extrusion and thermoforming applications and it could be used for food packaging, jewelry and gift boxes, medical supplies such as petri dishes, test tubes and specimen jars etc. Material Specification and MSDS is given in Appendix 1.
2. HIPS - (High Impact Polystyrene), PS 330 Grade was used for this experiment as the main composition material in the main extruder in combination with Filler Master Batch, White Master Batch and Regrind. It was brought from Sabic ,Saudi Arabia. This product was manufactured by continuous mass polymerization of styrene monomer with addition of an elastomer incorporated during polymerization to achieve impact resistance property. It was opaque in colour. PS 330 was primarily designed for extrusion and thermomforming applications and it can be used for food packaging and dairy products. Material Specification and MSDS- Material Safety Data Sheet is given in Appendix 2.
3. FMB – Filler Master batch - EF-PE-80 Grade was used for this experimentin combination with HIPS ,White Master Batch (WMB) and Regrind. It was brought from EuroPlast-European Plastic Joint stock Company from Vietnam. It was manufactured by twin screw extruder machine. This product contains Calcium Carbonate( $\text{CaCO}_3$ ) as filler, Linear Low Density Polyethylene (LLDPE) as Carrier resin and additives such a dispersion agent, processing aids. The product was suitable for extrusion, blown film, threading yarn, injection molding and thermoforming, etc.. It was white in colour and had good dispersion and wasfreefrom agglomerations. Material specifications and Material Safety Data Sheet (MSDS) is given in Appendix 3.
4. WMB – White Master Batch – VC-W 30S Grade was used for this experiment in combination with HIPS, FMB and Regrind. It was brought from Vina Colour Co.,Ltd. Vietnam. It containedhighly dispersive Rutile Titanium Dioxide ( $\text{TiO}_2$ ) as whitening agent,

in addition to that Polyethylene (PE) as carrier resins, Calcium Carbonate ( $\text{CaCO}_3$ ) and Coupling agents. This product was recommended for thermoforming and is of Food grade. It was of white in Colour and having good dispersion. It was compatible with PS and PP. Material Specification and MSDS is given in Appendix 4.

5. PP – Poly Propylene – HC 205TF Grade was used for this experiment in combination with FMB, WMB and Re grind. It was brought from Borealis AG, Vienna, Austria. HC 205TF was a specially nucleated polypropylene homopolymer grade designed for thermoforming packaging applications. Its increased crystallization temperature allows reduced cycle time and increased output. Material specification and Material Safety Data Sheet (MSDS) is given in Appendix 5.

#### 4.2 Testing of RawMaterial Properties

Melt flow Indices of each of above mentioned materials were tested for MFI as per ASTM D 1238 & ISO 1133. For that Computerized Melt flow Index tester – Model KAYJAY/2006/AC from Manufacturers and Exporters of Material, Film Testing Equipment Manufacturers, Mumbai, India were used.



Fig: 4.1 MFI Tester (Source: Manf. & Exp. Material..India)

The procedure for determining MFI is as follows:

- I. A small amount of the polymer sample (around 4 to 5 grams) is taken in the specially designed MFI apparatus. A die with an opening of typically around 2 mm diameter is inserted into the apparatus.
- II. The material is packed properly inside the barrel to avoid formation of air pockets.
- III. A piston is introduced which acts as the medium that causes extrusion of the molten polymer.
- IV. The sample is preheated for a specified amount of time: 5 min at 190 °C for polyethylene and 6 min at 230 °C for polypropylene.
- V. After the preheating a specified weight is introduced onto the piston. Examples of standard weights are 2.16 kg, 5 kg, etc.
- VI. The weight exerts a force on the molten polymer and it immediately starts flowing through the die.
- VII. A sample of the melt is taken after the desired period of time and is weighed accurately.
- VIII. MFI is expressed in grams of polymer per 10 minutes of duration of the test.

### **4.3 Preparation of Plastic Compounds and Extrusion of Sheet**

#### **4.3.1. Compound Preparation for extrusion of PS Sheet.**

HIPS, FMB, WMB and Regrind Materials were compounded in the hopper of extruder fitted with Model WSB 440 -McGuire Gravimetric Blender ( Fig.4.2) . It allowed to feed material continuously to the hopper of the main extruder. HIPS, FMB, WMB and Regrind Materials were used in Main Extruder. GPPS virgin material was used in co extruder. The purpose of GPPS to add to the co-extruder in order to get a lamination layer on the sheet. This layer would be the contact layer for foods in the final product. GPPS materials were fed to the co-extruder through vacuum loader.

Blender consisted of four compartments which were capable to mix material homogeneously prior to feed to the hopper of main extruder. In the Gravimetric Blenders – Each material was dosed by a stainless



Fig: No. 4.2 Mcguire Gravimetric Blender (Source: Mcguire Inc. USA)

steel slide gate. The slide gate was capable of dosing a wide range of settings from as little as 1% to 100% based on an average density free flowing material. (0.65 BD)

The hoppers were mounted above the Weigh Bin with a default 4kg batch size.

In normal operation the system tares the weight of the weigh bin and then each material was dispensed Regrind, virgin HIPS, WMB, and FMB in sequence, weighing each material after each dispense confirming material required before moving on to the next material.

The complete batch was released into the mixing chamber, and mixed with the previous batch to ensure homogenous mix.

Material blending was controlled by a level sensor mounted in the upper part of the mix chamber.

When the sensor was covered the blender will not dispense any further batches. The level of material in the mix chamber was critical to good mixing.

As the material in the mix chamber was consumed the sensor was uncovered and then the blender then commence the next cycle.

This Mcquire Gravimetric Unit is capable of auto calibration which ensures accurate dispense of every material with in the blend.

The PLC Controller updates every dispense materials in grams/second flow rate of each material, ensuring consistent accuracy.

Formulation settings were fed to the Controller unit via keypad. Further, audible alarm and strobe light with silence function, should a batch be incomplete, for example running out of Virgin or Masterbatches.

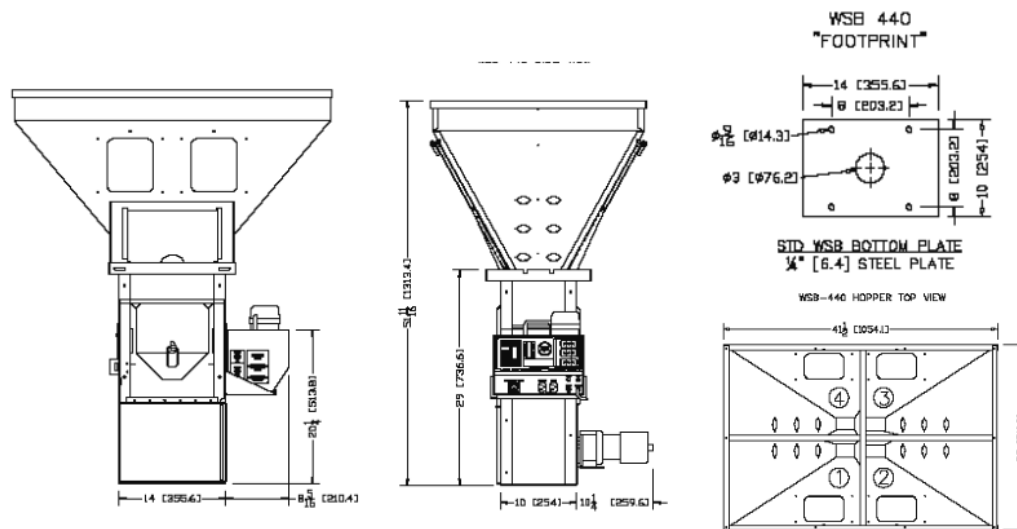


Fig 4.3 Schematic diagram of Mcquire Blender (Source: Mcquire Inc. USA)

#### 4.3.2 Compound Preparation of PP Sheet

In case of PP Sheet- PP virgin material, FMB, WMB and Re grind were fed to the main extruder via above mentioned McGuire Gravimetric Blender for consistent homogenous mixture.

#### 4.4 Compounding formulations

Two types of compounds of various formulations were prepared for each type of product material. Formulas of series N1 were based on styrene plastics and Formulas of series N2 were based on PP plastics.

Series N1 formulas included following Materials.

GPPS, HIPS, FMB, WMB and PS Regrind.

Series N2 formulas included following Materials.

PP, FMB, WMB and PP Regrind.

The formulations of Series N1 were prepared and given in table No. 4.1

Table. No 4.1 Formulations of compounds of series No1

<b>Item</b>	<b>Extruder</b>	<b>% Reference</b>	<b>%</b>	<b>%</b>	<b>%</b>	<b>%</b>
GPPS	Co- Ex	2	2	2	2	2
HIPS	Main	32	22	17	12	7
FMB	Main	0	10	15	20	25
WMB	Main	1	1	1	1	1
Regrind	Main	65	65	65	65	65

Justification for above formulations.

1. Regrind with FMB

This material brings down cost of formulation, however as any recycled material it creates number of problems for processor. In order to estimate optimum quantity of FMB in compounds, the compositions containing varying quantities of FMB (table No 4.1) were prepared, plastic sheets were extruded and product was made by thermoforming. Problems experienced during extrusion and thermoforming processes, including final product appearance were observed and recorded. Appearance of thermoformed products were observed.

Table. 4.2 Effect of Regrind with FMB on extrusion of PS plastic sheet and product appearance.

No	Regrind with FMB, %	Fresh polystyrene, Virgin FMB, Virgin WMB%	Compound bulk density	Extrusion problems	Product appearance
1	100	0	0.207	Interruption in feeding and cannot extrude sheet even after installation of vibrator to the hopper.	Thin gauge product, brittle, easily breaks
2	70	30	0.485	Feeding improved with reduction of Speed. Non-uniformity reduced	Slightly brittle. Output Reduced.
3	60	40	0.690	Continuous Feeding observed without interruption,	Product was uniform and quality improved.
4	50	50		The same as previous.	Costly product.

If 100% regrind is utilized there will be a reduction of output by 40% and if 70% regrind is used there will a reduction of output by 20%. Further, higher level of regrind is not capable to feed the hopper as the regrind flakes thickness was much less as the product was thin gauged. Since the bulk density of the regrind was around 0.207 and continuous feeding was not possible. Therefore, it was decided to utilize 65% regrind considering the average bulk density of feed materials to the hopper of main extruder.

Further, during the extrusion of sheet, it was observed that when the virgin FMB level was above 25% in PS materials as well as when virgin FMB level of above 25% in PP materials – Extruded sheets tends to crack and easily torn like a paper. And It was difficult to fed to the thermoforming machine as the sheet tends to crack and torn while feeding the roll to the Thermoformer. Further, WMB- White Master Batch was retained at minimum dosage level of 1% to provide required bright whiteness to the end

product. Further, the finished product was compared against the reference 0% Virgin FMB product.

2. White Master Batch –

WMB was maintained at lowest level of 1% in order to provide bright white to the product.

3. Filler Master Batch –

Virgin FMB level was varied from 0% to 25% with increment of 5%. Each increment of 5% was compensated with HIPS virgin, this was due to the reduction of usage of virgin material and to maintain the maximum possible level of regrind in the mixing formulation.

4. General Purpose Polystyrene

GPPS was introduced to the co-extruder as a lamination layer. Therefore it was maintained only at 2% as further reduction had observed uneven lamination on the sheet.

The Formulations of Series N2 were prepared and given in table No. 4.3

Table. No 4.3 Formulations of compounds of series No2

Item	% Reference	%	%
PP	34	14	9
FMB	0	20	25
WMB	1.0	1.0	1.0
Regrind with FMB	65	65	65

Justification for above formulations.

1. Regrind with FMB

This material brings down cost of formulation, however as any recycled material it creates number of problems for processor. In order to estimate optimum quantity of FMB in compounds, the compositions containing varying quantities of FMB (table No 4.3) were prepared, plastic sheets were extruded and product was made by



thermoforming. Problems experienced during extrusion and thermoforming processes, including final product appearance were observed and recorded. Appearance of thermoformed products were observed.

Table. 4.4 Effect of Regrind with FMB on extrusion of PP plastic sheet and product appearance.

No	Regrind with FMB, %	Fresh polypropylene, Virgin FMB, Virgin WMB%	Compound bulk density	Extrusion problems	Product appearance
1	100	0	0.386	Interruption in feeding and cannot extrude sheet even after installation of vibrator to the hopper.	Thin gauge product, brittle, easily breaks
2	70	30	0.573	Feeding improved with reduction of Speed. Non-uniformity reduced	Slightly brittle. Output Reduced.
3	60	40	0.715	Continuous Feeding observed without interruption,	Product was uniform and quality improved.
4	50	50		The same as previous.	Costly product.

If 100% regrind was utilized there will be a reduction of output by 40% and if 70% regrind is used there will a reduction of output by 20%. Further, higher level of regrind is not capable to feed the hopper as the regrind flakes thickness was much less as the product was thin gauged. Since the bulk density of the regrind was around 0.386 and continuous feeding was not possible. Therefore, it was decided to utilize 65% regrind considering the average bulk density of feed materials to the hopper of main extruder.

## 2. White Master Batch –

WMB was maintained at lowest level of 1% in order to provide bright white to the product.

## 3. Filler Master Batch –

Virgin FMB level was varied from 0%, 20% & 25%. In each formulation increase in FMB level was compensated with PP virgin, this was due to the reduction of usage

of virgin material and to maintain the maximum possible level of regrind in the mixing formulation.

#### **4.5 Extrusion of Plastic Sheets.**

##### **4.5.1 Machine Used.**

**Extruder** – Two Layer PS/PP Sheet Extruder Plant -Model VFK- 130/70 Co-Ex with an output of 700-800kg/hr of PS/PP. capable of extruding sheet thickness of between 0.15mm ~ 2mm and sheet width of 1000mm was used in experiment. It was brought From VFK Corp. South Korea. It was fitted with Nordson –typed Metering geared pump for primary extruder. Side scrap winder, Mcguire Gravimetric Blender Unit for measured material flow to the hopper. It was capable to produce sheet with 100% regrind with 40% reduction in output. The Machine specification is given in Appendix 6.



Fig 4.4 VFK PP/PS Extruder (Source; VFK Corp.)

#### 4.5.2 Extruder Setting Parameters for PS Materials

Table 4.5.1 Extrusion data for Polystyrene Materials

<b>a. Main Extruder</b>								
Zone	Zone 01	Zone 02	Zone 03	Zone 04	Zone 05	Zone 06	Zone 07	Zone 08
Temp C	185	190	195	200	205	210	215	220
Zone	Screen Changer		Gear Pump			Feed Block		
Temp C	220		225			220		
<b>b. T-Die</b>								
Zone	Zone 01		Zone 02		Zone 03		Zone 04	
Temp C	213		217		226		217	
<b>c. Co Extruder</b>								
Zone	Zone 01		Zone 02		Zone 03		Screen Changer	
Temp C	210		215		215		218	
<b>d. Speed and Output</b>								
	Main Extruder	Gear Pump	Co Extruder	Line Speed		Main Master	Out put (Kg/hr)	
Meter (rpm)	42.5	81	31	20.85		81	610	
<b>e. Pressure Control</b>								
SP				38				
PV				37				

#### Material Feeding Sequence.

Main Extruder - HIPS, FMB, WMB, PS Regrind via Mcguire Gravimetric Blender

Co – Extruder - GPPS via Vacuum Loader

Extruded sheets were checked for Appearance such as free from any crack, smooth shining lamination of GPPS layer, and wounded to 350kg batch of rolls for thermoforming process. Edge trims were grounded via trim re grinder unit and collected to Jute bags to re-use as regrind.

### 4.5.3 Extruder Setting Parameters for PP Materials

Table 4.5.2 Extrusion Data for Polypropylene Materials

<b>a. Main Extruder</b>								
Zone	Zone 01	Zone 02	Zone 03	Zone 04	Zone 05	Zone 06	Zone 07	Zone 08
Temp °C	215	225	235	240	250	260	265	245
Zone	Screen Changer		Gear Pump			Feed Block		
Temp °C	265		255			255		
<b>b. T- Die</b>								
Zone	Zone 01	Zone 02	Zone 3	Zone 04	Zone 05			
Temp °C	225	225	225	225	225			
<b>c. Co Extruder</b>								
Zone	Zone 01	Zone 02	Zone 03	Screen Changer	Adaptor			
Temp °C	NA	NA	NA	NA	265			
<b>d. Speed and Output</b>								
	Main Extruder	Gear Pump	Co Extruder	Line Speed	Main Master	Out put (Kg/hr)		
Meter (rpm)	75	53	5	NA	81	604		
<b>e. Pressure Control</b>								
SP				25				
PV				91				

#### Material Feeding Sequence.

Main Extruder - PP Virgin, FMB, WMB, PP Re grind via Mcguire Gravimetric Blender

Co – Extruder - Empty

Extruded sheets were checked for Appearance such as free from any crack, smooth shining surface. And sheet were wound to 350kg batch of rolls for thermoforming process. Edge trims were grounded via trim re grinder unit and collected to Jute bags to re-use as re grind.

#### **4.5.4 Thermoforming Machine Used.**

Product was made with Auto Vacuum forming Machine – Model VFK- 800/1200 Bed Sized Thermoforming machine from VFK Corp. South Korea. Which was fitted with Auto trim press with counter stacker and Inline crusher.

#### **Thermoforming Machine Used – From VFK Corp. 800/1200 Bed Size Model.**



Fig 4.5 VFK 800/1200 Thermoforming Machine (Source: Dawanco Ind WLL)

#### 4.5.5. Thermoforming Setting Parameters for PS Materials.

Table 4.5.3 Thermoforming Machine Setting Data for Polystyrene Materials.

<b>a. Heating Temp (°C) Upper Side</b>							
1	2	3	4	5	6	7	8
384	289	374	249	399	314	419	289
9	10	11	12	13	14	15	-
349	199	379	244	249	104	114	
<b>b. Heating Temp (°C) Lower Side</b>							
1	2	3	4	5	6	7	8
369	314	259	254	109	219	230	109
9	10	11	12	13	14	15	-
139	261	139	134	104	134	124	
Upper Forming Mould			Closing Distance			283mm	
			Opening Distance			185mm	
Lower forming Mould			Closing Distance			207mm	
			Opening Distance			123mm	
<b>c. Auto hydraulic Trimming Machine</b>							
Feed # 01			104.9				
Feed # 02			134				

#### 4.5.6. Thermoforming Setting Parameters for PP Materials.

Table 4.5.4 Thermoforming Machine Setting Data for polypropylene

<b>a. Heating Temperature (<sup>0</sup>C) upper side</b>														
SV1	SV2	SV3	SV4	SV5	SV6	SV7	SV8	SV9	SV10	SV11	SV12	SV13	SV14	SV15
261	261	161	161	166	162	261	237	167	162	158	201	237	230	230
SV16	SV17	SV18	SV19	SV20	SV21	SV22	SV23	SV24	SV25	SV26	SV27	SV28	SV29	SV30
230	230	230	230	242	230	242	230	227	273	273	207	303	199	199
SV31	SV32	SV33	SV34	SV35	SV36	SV37	SV38	SV39	SV40	SV41	SV42	SV43	SV44	SV45
299	299	280	280	212	207	190	280	202	192	192	195	195	273	273
<b>b. Heating Temperature (<sup>0</sup>C) Lower Side</b>														
SV1	SV2	SV3	SV4	SV5	SV6	SV7	SV8	SV9	SV10	SV11	SV12	SV13	SV14	SV15
257	257	257	257	257	255	200	224	221	206	206	210	314	309	303
SV16	SV17	SV18	SV19	SV20	SV21	SV22	SV23	SV24	SV25	SV26	SV27	SV28	SV29	SV30
298	298	293	289	289	287	293	284	319	318	294	290	289	288	284
SV31	SV32	SV33	SV34	SV35	SV36	SV37	SV38	SV39	SV40	SV41	SV42	SV43	SV44	SV45
283	279	318	348	298	280	270	348	298	281	295	289	289	324	343

(SV- Setting value of Infra-red ceramic typed heating elements)

Following Products were Made for Testing (Fig. 4.6).

They were: 250g Tub, 500g Tub, 6oz Cup, 7oz cup, 6oz bowl, 13oz bowl, Plate 1,2,3,4,5.



Fig 4.6 Thin gauged product samples

#### 4.6 Testing of Finished Products.

Finished Products were subjected to following testing for any quality deviations.

- (a) Appearance
- (b) Specific Gravity
- (c) Drop Test
- (d) Oil Test
- (e) Actual FMB Level Test

##### 4.6.1 Appearance

Each and every samples produced **w**as subjected to visual appearance test such as following

- |                   |                  |
|-------------------|------------------|
| 1. Forming        | 4. Impurities    |
| 2. Fitment If any | 5. Surface Gloss |
| 3. Cracks         |                  |



## 4.6.2 Specific Gravity

For this Electronic Densimeter : MD-300S, which is combined high precision electronic balance and a patented container was used to measure specific gravity of the product. The machine adopts “ Archimedes’ principle and the determination of (relative) density at 4C: 1g/cm<sup>3</sup>.



Fig 4.7 Densimeter MD 300 Series.

Test was done as per ISO 1183 Standards.

A standard measurement mode is selected for normal specific gravity measurement.

Procedure:

- I. Turn on the power and check the display and Press “Re-Zero” key if it does not show on the display.
- II. Place a sample on the sensor gently and weight will be displayed.
- III. Press Enter key after the stable mark “O” appears where it takes the average of aerial gravity.
- IV. Average mark “-“was vanishing one by one and the stable mark “O” starts blinking.
- V. The aerial gravity appears again when memorized. “L” appears on the lower left corner of the display when the sample is taken away from the sensor.

- VI. Hold sample by using tweezers and place it on the central part of the measuring tray in water gently. In doing so, get rid of bubbles sticking on the samples by shaking it slightly in water. The bubbles may interfere with the accuracy of measurement.
- VII. Place sample on the measuring tray in water to measure the underwater gravity.
- VIII. The average mark “- “ is vanishing one by one and the stable mark “O” starts blinking.
- IX. Measurement results are displayed. The results would be specific Gravity.

#### **4.6.3 Drop Test / Falling Test**

The Drop test was in reference to the standard IQS 1169/1988 – Quality Test Method for single use plastic cups, plates, and containers. For this Dried Sand of ( $\rho \geq 1.75\text{g/cm}^3$ ) is used to fill the container.

Test Procedure:

- I. Fill the cup or tub container with dried sand up to the brimful.
- II. Close the cup or tub container with the suitable lid.
- III. From a height of 100cm (1m), drop the cup or tub, ensuring that the bottom edge of the cup or tub will hit the ground diagonally.
- IV. Observe if there is a presence of crack or breakage on the cup or tub
- V. Record the results as “Passed” or “Failed”

**ILLUSTRATION:**

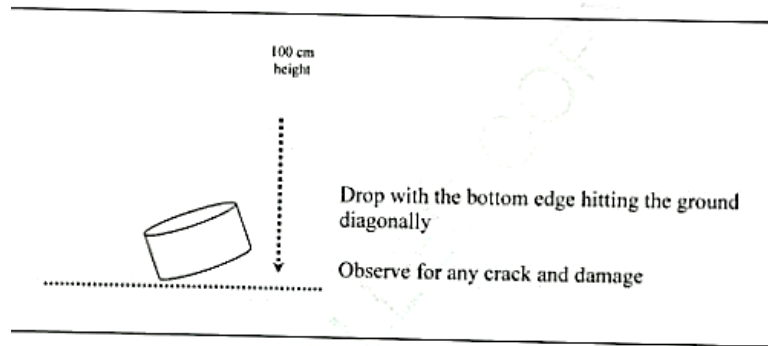


Fig. 4.8 Schematic Diagram of Drop test

#### **4.6.4 Oil Proof Test**

This test is carried out to check the resistance of plastic products to physical change, crack and leakage when exposed to oily food products. This also highlights the level of impermeability of the product. For this test commercially available cooking oil is used.

Procedure:

##### **At Room Temperature:**

- I. Prepare the test samples and place them on the top the white absorbent paper.
- II. Measure the require volume of the cooking oil in the graduated cylinder (at least up the brimful of the test container if the lid is being tested)
- III. Fill the container, making sure not to splash out oil comes in contact with the lid,
- IV. Let the test specimen stand for 48hours. Check for any physical change (i.e. Crack/deformation/dicolouration and record the time of observation)

- V. Check for any leakage mark on the white absorbent paper board.
- VI. Record the results.

#### **At Cold Temperature:**

Repeat the above test, only this time, the test specimens were subjected to cold temperature, inside the refrigerator ( $\leq 0\text{C}$ ).

There should be no deformation / discoloration / cracks or leaks to be observed with in 8hours of testing. If so Record the results as “Passed” or “Failed”. 48hrs testing time: simulating the extreme condition.

#### **4.6.5. Actual FMB Level Test / Muffle Furnace Test**

Following Muffle Furnace Test was developed by the Author in order to find out actual FMB level in the test pieces.

The test pieces are subjected to externally heated chamber, the walls of which radiantly heat the content of the chamber, so that the material being heated has not contact with the flame.

Procedure;

- I. Switch on the unit from the main switch
- II. Set the required temperature ambient to 500 C
- III. After the desired temperature is attained, allow it to become stable for half an hour
- IV. Prepare the 3 grams of sample, weigh the empty crucible in grams and the weight of the crucible with sample.
- V. Place the crucible in the muffle furnace for 1hr /80 minutes
- VI. Before removing the crucible, switch off the furnace and remove the top plug from the socket. Wait for 50~60 minutes for cooling.
- VII. Remove the crucible. Allow to cool slightly and re-weigh.

Actual FMB % = (Weight of Sample remaining/ weight of original sample) x 100

## CHAPTER 5

### Results & Discussion

Following Results were obtained for Thin Gauged Product made out of PS and PP Material

#### 5.1 Melt Flow Index

Test Results of Melt Flow Index of the Raw Material used for Formulation of Series N1 and N2 were given below.

Table 5.1 Test Results of Melt flow Index of Raw Materials used in Formulation Series N1 and N2

Material	Standards	Results (g/min)
GPPS – PS 125	200C , 5 Kg	6.9
HIPS – PS 330	200C, 5 Kg	3.0
FMB – EF –PE 80	190C, 5 Kg	23.2
WMB – VC- W 30S	230C, 2.16kg	11.5
PP - HC 205TF	230C, 2.16kg	4.0

As per the test results of Melt Flow Index of Raw Materials were in accordance to the specification sheet given in Appendix 1~5.

#### 5.2. Appearance

Test Results of Appearance of the product made out of formulation of series No.1 given below

Table No. 5.2.1 Test Results of Appearances of PS Mixing (Formulation series N1)

	<b>Appearance</b>				
<b>PS Mixing</b>	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
0% FMB	Passed	Passed	Passed	Passed	Passed
10% FMB	Passed	Passed	Passed	Passed	Passed
15% FMB	Passed	Passed	Passed	Passed	Passed
20% FMB	Passed	Passed	Passed	Passed	Passed
25% FMB	Failed at Sheet stage				

It was noted that in each formulation the product passed in Appearance test except the 25% Virgin FMB formulation. Samples made out of 0% ~20% Virgin FMB based were free from impurities, cracks and good forming was observed as well as even surface gloss were observed. In the case of 25% Virgin FMB formulated sheet -it was noted that the sheet cracked while extrusion and it was not possible to feed to Thermoforming machine to turnout product.

Test Results of Appearance of the product made out of formulation of series No.2 given below

Table No. 5.2.2. Test Results of Appearances of PP Mixing (formulation series N2)

	<b>Appearance</b>				
<b>PP Mixing</b>	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
0% FMB	Passed	Passed	Passed	Passed	Passed
20% FMB	Passed	Passed	Passed	Passed	Passed
25% FMB	Failed at Sheet Stage				

It was noted that in each formulation the product passed in Appearance test except the 25% Virgin FMB formulation. Samples made out of 0% Virgin FMB formulation was more flexible as it is of Polypropylene material and 20% Virgin FMB based were free from impurities, cracks and good forming was observed as well as even surface gloss were observed and less flexible than 0% Virgin FMB based product. In the case of 25%

Virgin FMB formulated sheets - it was noted that the sheet cracks while extrusion and it was not possible to feed to Thermoforming machine to turnout product.

### 5.3 Specific Gravity

Test Results of Specific Gravity of the product made out of formulation of series No.1 given below:

Table 5.3.1 Test results of the specific gravity of the formulation series N1

	Specific Gravity				
PS Mixing	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
0% FMB	1.058	1.06	1.061	1.059	1.062
10% FMB	1.17	1.17	1.187	1.192	1.164
15% FMB	1.203	1.197	1.214	1.206	1.191
20% FMB	1.23	1.244	1.225	1.206	1.213
25% FMB	Sheet cracked during extrusion and tearing like a paper. Cannot be used in Thermoforming Machine				

Based on above results the specific gravity of the extruded sheet increased with the increase of FMB in the formulation. Further, this highlights the product increase in product weight. By studying this we should be able to reduce the sheet thickness in order to maintain the constant end product weight. i.e.

$$\rho = m / V \quad (\text{where } \rho - \text{Density, } m - \text{Mass, } V - \text{Volume})$$

$$\rho = m / (A \times t) \quad (\text{where } A - \text{Surface area, } t - \text{thickness of the sheet})$$

Since the mass of the product  $m$  was constant, and surface area of the extruded sheet  $A$  was constant as the sheet width was fixed by T- die width.

Therefore,  $\rho \propto 1/t$  (density was inversely proportional to thickness)

This means - in order to maintain the constant weight of end product with the addition of FMB into the formulation, the thickness of the sheet had to be decreased. Therefore, study of specific gravity provide necessary information on decision on thickness of the sheet.

Test Results of Specific Gravity of the product made out of formulation of series No.2 given below:

Table 5.3.2 Test results of Specific Gravity of formulation series N2

	Specific Gravity				
PP Mixing	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
0% FMB	0.915	0.921	0.912	0.918	0.91
20% FMB	1.265	1.196	1.022	1.142	1.171
25% FMB	Sheet cracked during extrusion. Cannot be used in Thermoforming Machine.				

Based on above results the specific gravity of the extruded sheet increased with the increase of FMB in the formulation. Further, this highlights the product increase in product weight. By studying this we should be able to reduce the sheet thickness in order to maintain the constant end product weight. i.e.

$$\rho = m / V \quad (\text{where } \rho - \text{Density, } m - \text{Mass, } V - \text{Volume})$$

$$\rho = m / (A \times t) \quad (\text{where } A - \text{Surface area, } t - \text{thickness of the sheet})$$

since the mass of the product  $m$  was constant, and surface area of the extruded sheet  $A$  was constant as the sheet width was fixed by T- die width.

Therefore,  $\rho \propto 1/t$  ( density was inversely proportional to thickness)

This means - in order to maintain the constant weight of end product with the addition of FMB into the formulation, the thickness of the sheet had to be decreased. Therefore, study of specific gravity provide necessary information on decision on thickness of the sheet.

Further, in both cases when 25% of Virgin FMB was introduced to the formulations cracks appeared on the sheet. And sheet was torn like a paper. Therefore, sheet made out of 25% Virgin FMB formulation was not able to fed to the thermoforming machine to turn out product.

Since the above test we were able to reduce the thickness of the sheet to maintain the constant end product weight. But the stability of the product was questionable.



Therefore, Drop test was necessary to study the impact test of the product in order to understand the usability of the product in day to day life.

#### 5.4 Drop Test

Test Results of Drop Test of the product made out of formulation of series No.1 given below:

Table 5.4.1 Test Results of Drop Test of formulation series N1

	Drop Test				
PS Mixing	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
0% FMB	Passed	Passed	Passed	Passed	Passed
10% FMB	Passed	Passed	Passed	Passed	Passed
15% FMB	Passed	Passed	Passed	Passed	Passed
20% FMB	Failed	Failed	Failed	Passed	Passed
25% FMB	Failed at Sheet Stage				

It was observed due to addition of FMB the specific gravity of the sheet increased. Therefore, in order to maintain constant end product weight sheet thickness was reduced with the increase in FMB.

The reduction of thickness of the sheet may impact on the product stability and usability. To study the usability impact test or drop test was carried out on finished product. From the test result we can conclude that in the formulation of series N1 - Virgin FMB level 0% ~ 15% were passed in Drop Test. And major portion of the 20% Virgin FMB formulation failed. This was due to the actual FMB level was higher than the FMB level in the formulation. This was explained earlier due to the accumulated FMB in the regrind had caused this failure.

Test Results of Drop Test of the product made out of formulation of series No.2 given below:

Table 5.4.2 Test Results of Drop Test of Formulation Series N2

	Drop Test
--	-----------

PP Mixing	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
0% FMB	Passed	Passed	Passed	Passed	Passed
20% FMB	Passed	Passed	Passed	Passed	Passed
25% FMB	Failed at sheet Stage				

From above results we can conclude that 0% ~ 20% Virgin FMB level in formulation of Series N2 has passed the impact test and 25% Virgin FMB formulation of Series N1 as well as N2 failed at sheet stage as the sheet tends to crack and easily torn. Therefore, as explained above in PS mixing formulation the actual FMB level was much higher than that of indicated percentage level of FMB in the formulation of Series N1 and N2. Therefore, in order to study the actual FMB level in the finished product I had designed a test method called Muffle Furnace test. Test procedure was explained in earlier chapter.

### 5.5 Actual FMB Level Test / Muffle Furnace Test

Test Results of Actual FMB Level Test of the product made out of formulation of series No.1 given below:

Table 5.5.1 Test Results of Actual FMB level of formulation series N1

	Actual FMB				
PS Mixing	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
0% FMB	0.21%	0.11%	0.24%	0.29%	0.31%
10% FMB	18.60%	18.74%	19.30%	19.89%	18.08%
15% FMB	21.06%	20.72%	22.31%	21.54%	20.16%
20% FMB	27.59%	28.52%	27.54%	24.51%	26.16%
25% FMB	Failed at sheet stage				

According to above results it was obvious that the actual FMB level was higher than the formulation. This was due to FMB accumulation in the Re grind (Refer Table 5.5.3). In

the above results of 0% Virgin FMB contained almost upto 0.3% of Fillers, this is due to the WMB contains FMB which contributed to this value. Further, 20% Virgin FMB formulation contained actual FMB level of 24.5% ~ 28.5% . Further, the 28.5% Sample had failed in the drop test due to the higher level of Fillers. Further 25% Virgin FMB Formulated sheet had failed during extrusion of sheet as the sheet got cracked and easily torn due to higher level of filler.

Test Results of Actual FMB Level Test of the product made out of formulation of series No.2 given below:

Table 5.5.2 Test Results of Actual FMB level of formulation series N2

	Actual FMB				
PP Mixing	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
0% FMB	0.2%	0.3%	0.2%	0.2%	0.3%
20% FMB	40%	35.90%	21.82%	29.56%	32.63%
25% FMB	Failed at sheet stage				

According to above results it was obvious that the actual FMB level was higher than the formulation. This was due to FMB accumulation in the Regrind (Refer Table 5.5.3). In the above results of 0% virgin FMB contained almost up to 0.3% of Fillers, this is due to the WMB contains FMB which contributed to this value. Further, 20% virgin FMB formulation contained actual FMB level of 21.8% ~ 40%. Further, all the Samples of 20%FMB virgin Formulation had passed in the drop test even though higher level of Fillers of 40%. Further 25% Virgin FMB Formulated sheet had failed during extrusion of sheet as the sheet got cracked and easily torn due to too much higher level of filler.

Actual FMB level in Regrind Materials given in Table 5.5.3.

Table 5.5.3 Actual FMB level of Regrind used in Formulation Series N1 and N2

Crush Material	Actual FMB Level
0% FMB PS Regrind	0.16%

10% FMB PS Regrind	12.84%
15% FMB PS Regrind	16.99%
20% FMB PS Regrind	21.70%
0% FMB PP Regrind	0.14%
20% FMB PP Regrind	23.54%
25% FMB PP Regrind	37.2%

According to the above results of Actual FMB level in Regrind –shows very high amount of heavy fillers but due to the thin gauged product the material bulk density very low and it was discussed in Section 4.4 above. Since the bulk density of the regrind was around 0.207 and continuous feeding was not possible. Therefore, it was decided to utilize 65% regrind considering the average bulk density of feed materials to the hopper of main extruder. Since the objective of this research was to find the best level of filler master batch for the thin gauged thermoformed product, only FMB was increased by reducing the virgin materials as an intention to minimize the cost of end product as well as to maintain the average bulk density around 0.65.

### 5.6 Oil Test

Test Results of Oil Test of the product made out of formulation of series No.1 given in Table 5.6.1:

Table 5.6.1 Test Results of Oil Test of Formulation Series N1

	<b>Oil Test</b>				
<b>PS Mixing</b>	<b>Sample 1</b>	<b>Sample 2</b>	<b>Sample 3</b>	<b>Sample 4</b>	<b>Sample 5</b>
0% FMB	Passed	Passed	Passed	Passed	Passed
10% FMB	Passed	Passed	Passed	Passed	Passed
15% FMB	Passed	Passed	Passed	Passed	Passed
20% FMB	Passed	Passed	Passed	Passed	Passed
25% FMB	Failed at sheet stage				

Oil Test was carried out as these thin gauged product were subjected to storage of oiled foods. Test was carried out to find out if there was any impact on the product when adding of heavy Fillers to the formulation. According to the results product has passed almost all samples made up to 20% Virgin FMB formulation. Since the 25% virgin FMB formulation had failed at the sheet level. It was not possible to carry out the oil test for 25% virgin FMB formulation sheet.

Test Results of Oil Test of the product made out of formulation of series No.2 given in Table 5.6.2:

Table 5.6.2 Test Results of Oil Test of Formulation Series N2

	Oil Test				
PP Mixing	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
0% FMB	Passed	Passed	Passed	Passed	Passed
20% FMB	Passed	Passed	Passed	Passed	Passed
25% FMB	Failed at Sheet stage				

Oil Test was carried out as these thin gauged product were subjected to storage of oiled foods. Test was carried out to find out if there was any impact on the product when adding of heavy Fillers to the formulation. According to the results product has passed almost all samples made up to 20% Virgin FMB formulation. Since the 25% virgin FMB formulation had failed at the sheet level. It was not possible to carry out the oil test for 25% virgin FMB formulation sheet.

## **CHAPTER- 6:**

### **Conclusions**

The study result showed.

1. That increase in filler load in plastic composition based on polystyrene resulted in reduction of flow ability that come through uneven melt distribution during thermoforming stage of thin gauged primary packaging product.

2. That increase in filler load in plastic composition based on polypropylene resulted in reduction of flow ability that come through uneven melt distribution during thermoforming stage of thin gauged primary packaging product.
3. It was found that max concentration of total filler content of 26% in Polystyrene based composition gave satisfactory appearance and customer acceptable product. Further, it was found that max concentration of total filler content of 40% in Polypropylene based composition gave satisfactory appearance and customer acceptable product.

Further, the study results showed that thin gauged thermoform products made out of Polystyrene and Polypropylene materials were developed with best level of loading of Filler Master Batch. In Polystyrene based thin gauged products when actual FMB level increased above 26% it tends to fail in drop test as it increases the brittleness in Food Containers which tends to break or crack during drop test. And it was proven through Actual Filler Master Batch Level Test. Therefore, it was important to manage the Filler based regrind to ensure mixing will not exceed the maximum Filler level of 26% in Polystyrene based thin gauged thermoformed primary packaging food containers in order to get customer acceptance similar to the reference sample of 0% virgin Filler Master Batch product. In order to ensure that, suitable gravimetric blenders should be installed to ensure required mixing ratio to be followed in the extruder.

Further, in the case of Polypropylene a based thin gauged thermoformed product with the actual Filler level up to 40% has passed the drop test as well as the Oil test. Therefore, it was important to maintain maximum level of 40% actual FMB level in the Polypropylene based thin gauged thermoformed primary packaging food containers in order to get customer acceptance similar to the reference sample of 0% virgin Filler Master batch product. Currently the prices of Fast Moving Consumer Goods (FMCG) primary plastic packaging products are more competitive in the market and there are no of new companies are emerging to the market. Therefore, more research need to be conducted in following areas.

- (a) Increase the level of Filler Master Batch beyond 26% for Polystyrene and 40% for Polypropylene by using twin screw extruders to provide better mixing ratios.
- (b) Introduction of nano particle sized Filler Master batch to improve the loading level.

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**Appendix I.**

**Characteristics**

General purpose polystyrene PS 125 is manufactured by continuous mass polymerization of styrene monomer. It is a crystal-like, hard and brittle polymer.

- Medium flow with excellent clarity.
- Higher Vicat and heat distortion temperatures allow its use in many different applications.

**Applications**

- It is recommended for the manufacture of a variety of packaging items, namely jewelry and gift boxes; medical supplies such as petri dishes, test tubes and specimen jars, etc.
- Another important use is in capping the high impact polystyrene coextruded sheet for high surface gloss.
- It could be blended with impact modifier resin for clear packaging articles.

Typical Properties <sup>a</sup>	Unit	Value <sup>b</sup>	ASTM Method
MELT INDEX (200°C, 5 KG)	g/10 min.	7.0	D-1238
DENSITY (23°C)	g/cm <sup>3</sup>	1.05	D-792
BULK DENSITY (METHOD B)	g/cm <sup>3</sup>	0.60	D-1895
VICAT SOFTENING POINT (Rate A, 1 Kg/50°C)	°C	95	D-1525
HEAT DISTORTION (METHOD B, 455 KPA), ANNEALED	°C	90	D-648
ULTIMATE TENSILE STRENGTH (5mm/min)	(MPa)	43	D-638
ULTIMATE TENSILE ELONGATION	%	2	D-638
TENSILE MODULUS	(MPa)	2598	D-638
FLEXURAL STRENGTH	(MPa)	82	D-790

**Appendix II**  
Material Specification of HIPS



**Characteristics**

High impact polystyrene PS 330 is manufactured by continuous mass polymerization of styrene monomer. An elastomer is incorporated during polymerization to achieve impact resistance property. It is generally opaque in color.

- High impact strength polystyrene with high heat distortion temperature and good physical properties.

**Applications**

- It is primarily designed for extrusion and thermoforming applications.
- It can be used for food packaging and dairy products.

Typical Properties:1	Unit	Value	ASTM Method
MELT INDEX (200°C, 5 KG)	g/10 min.	3.0	D-1238
DENSITY (23°C)	g/cm <sup>3</sup>	1.04	D-792
BULK DENSITY (METHOD B)	g/cm <sup>3</sup>	0.60	D-1895
VICAT SOFTENING POINT (Rate A, 1 Kg/50°C)	°C	99	D-1525
HEAT DISTORTION (METHOD B, 455 KPA), ANNEALED	°C	97	D-648
ULTIMATE TENSILE STRENGTH (5mm/min)	(MPa)	29	D-638
ULTIMATE TENSILE ELONGATION	%	50	D-638
TENSILE MODULUS	(MPa)	2353	D-638
FLEXURAL STRENGTH	(MPa)	44	D-790
FLEXURAL MODULUS	(MPa)	2353	D-790

**Appendix III.**

## PRODUCT INFORMATION AND DATA SHEET

PRODUCTION NAME: FILLER MASTERBATCH  
PRODUCT GRADE: EFPE 80

### About Product

This product is one of kinds filler masterbatch. It is manufactured by twin crew extruder machine a most modern technology represented in Vietnam.

### General Description

This product contains: CaCO<sub>3</sub>, resin and some additives. It's suitable for extrusion, blowing threading yarn, injection molding, etc. This product has very good dispersion, free of agglomeration bring for the end product more whiteness, transparent, glossier, smooth's surface.

### Technical Analysis

#### Physical Property

Items	Method of experiment	Unit	Standard
Calcium Carbonate (CaCO <sub>3</sub> ) content	ASTM D 5630	wt %	80
Melt Mass-Flow Rate (190°C/5kgs)	ASTM D 1238	g/10 mins	23
Melt Temperature	DSC	°C	110
Bulk density	ASTM D1895	g/cm <sup>3</sup>	1.15
Particle size of CaCO <sub>3</sub> Powder	Malvern	µm	3
Water Content	ASTM D 644	wt %	<0.15

#### Properties:

Appearance (Surface color)	: White
Additives	: Dispersion agent, processing aid
Processing temperature	: 140 – 280°C
Usage rate	: 5 -40 %
Pellet size	: 3x3 (±0.3) mm
Carrier resin	: LLDPE
Compatibility	: LLDPE, LDPE, HDPE, PP, PS..
Packing	: 25 kgs/bag
Storage	: Keep at dry condition

#### Caution:

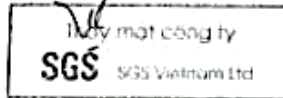
**EUROPEAN PLASTIC JOINT STOCK COMPANY**  
DONG VAN INDUSTRIAL ZONE, DUY TIEN DISTRICT, HANAM PROVINCE  
VIETNAM

The following sample was submitted and identified on behalf of the client as below:

Sample : EFPE 80  
Characteristic : WHITE FILM  
Country of Destination : NA  
Sample Receiving Date : March 27, 2015  
Testing Period : March 27, 2015 to April 15, 2015  
Test Requested : Please refer to the result summary.  
Test Method & Results : Please refer to next page(s).  
Result Summary :

Test Requested	Conclu
Commission Regulation (EU) No 10/2011 and hence Article 3 of European Regulation No. 1935/2004.	
a) Plastic – Overall Migration	PAS
b) Plastic – Specific Migration of Heavy Metals	PAS

Signed for and on behalf of  
SGS Vietnam LTD



Tran Thi Lien  
Hardline and E&E Lab Manager

**Test Report**

No. 441229/HLL15/0878

Date: April 15, 2015

Page 2 of 3

**Test Results:**
**Sample Description:** 1. EFPE 80

**a) Overall migration**

Method: With reference to Commission Regulation (EU) No 10/2011 of 14 January 2011 Annex III and Annex V for selection of condition and EN 1186-1:2002 for selection of test methods; EN 1186-3:2002 aqueous food simulants by total immersion method.

Simulant Used	Test Condition	Result (mg/dm <sup>2</sup> )	Reporting Limit (mg/dm <sup>2</sup> )	Permissible Limit (mg/dm <sup>2</sup> )
		1		
50% Ethanol (V/V) Aqueous Solution	10 days at 40°C	ND	3	10
3% Acetic Acid (W/V) Aqueous Solution	10 days at 40°C	4.45	3	10
Deionized Water	10 days at 40°C	ND	3	10
<b>Comment</b>	--	<b>PASS</b>	--	--

 Note : 1. mg/dm<sup>2</sup> = milligram per square decimeter

2. °C = degree Celsius

3. ND = Not Detected

4. Permissible Limit is according to Commission Regulation (EU) No 10/2011 of 14 January 2011 with amendments.

**b) Specific Migration of Heavy Metals**

Method: With reference to Commission Regulation (EU) No 10/2011 of 14 January 2011 Annex III and Annex V for selection of condition and EN 13130-1:2004 for selection of test method. Analysis was performed by ICP-MS.

Simulant Used: 3% acetic acid (w/v) in aqueous solution

Test Condition: 40°C for 10days

Test Item	Result (mg/kg)	Reporting Limit (mg/kg)	Permissible Limit (mg/kg)
	1		
Specific Migration of Barium	ND	0.25	1
Specific Migration of Cobalt	ND	0.01	0.05
Specific Migration of Copper	ND	0.5	5
Specific Migration of Iron	ND	0.5	48
Specific Migration of Lithium	ND	0.5	0.6
Specific Migration of Manganese	ND	0.25	0.6
Specific Migration of Zinc	ND	0.25	25
<b>Comment</b>	<b>PASS</b>	--	--

Note : 1. mg/kg = milligram per kilogram of foodstuff in contact with

2. °C = degree Celsius

3. ND = Not Detected

4. Permissible Limit is according to Commission Regulation (EU) No 10/2011 of 14 January 2011 with amendments.

5. Test condition &amp; simulant were specified by client.

**Remark:** These tests were performed by SGS Vietnam's Chemical lab.

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## Appendix IV

### Material Specification of WMB



## SPECIFICATION

**Name: White Masterbatch**

**Grade: VC – W 30S**

### About product

VC –W 30S is a poly propylene based mastebatch containing highly dispersive TiO<sub>2</sub> Rutile which is recommended for PS thermoforming. It ensure better dispersion and .

### Physical Properties

Index	Test method	Unit	Typical Value
Carrier	---	---	PE + coupling agent
TiO <sub>2</sub> content	---	%wt	30
MI @2.16kg/230°C	ASTM D1238	g/10 mins	11 ± 1
Moisture	ASTM D644	%	0.10 max
Shape	----	granule	2~3 mm <sup>φ</sup>

Additives : Dispersion agent, processing aid

Processing temperature : 150 – 330°C

Uses percent : 2- 8 %

Compatibility : PE, PS

Packing : 25 kg/bag

Storage : Keep at dry condition

### Medical norm

Attain the Safety Norm for Health and Foodstuff issued by Vietnam Directorate for Standards and Quality allow the decision 3339/2001/QĐ-BYT promulgated by Vietnam Ministry of Health.

Safety apply for handling process. No healthy damage infected.

*Caution: This product information is based on our general experience and does not constitute a specification. Since many factors affect the use of our products, no warranty is given or implied with respect to this information or patent infringement. We do not accept liability for any loss or damage arising from the use of this information. All sales are subject to our Standard Terms and Conditions of Sale.*

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## Appendix V

### Material Specification of Polypropylene

#### HC205TF

##### DESCRIPTION

HC205TF is Homopolymer grade produced by the proprietary **Borstar®** process, intended for sheet and thermoformed packaging applications.

This grade is produced using the **Borealis Nucleation Technology™ (BNT)**. Its increased crystallisation temperature allows reduced cycle time and increased output. Products originating from this grade have excellent transparency, good organoleptic properties, good balance of stiffness and impact strength at ambient temperatures.

##### APPLICATIONS

In-line & Off line thermoforming

High transparency trays, cups and containers

Margarine tubs and dairy containers

Blending with copolymers for lids

##### SPECIAL FEATURES

Good stiffness-impact balance

Excellent processability

Excellent clarity

Broad application window

Consistent shrinkage (post forming)

Excellent product consistency

Reduced cycle time / higher output

##### PHYSICAL PROPERTIES

Property	Typical Value	Test Method
Density	900 -910 kg/m <sup>3</sup>	ISO 1183
Melt Flow Rate (230 °C/2.16 kg)	4 g/10min	ISO 1133
Flexural Modulus (5 mm/min)	1700 MPa	ISO 178
Tensile Modulus (1 mm/min)	1750 MPa	ISO 527-2
Tensile Strain at Yield (50 mm/min)	8 %	ISO 527-2
Tensile Stress at Yield (50 mm/min)	35.5 MPa	ISO 527-2
Melting Temperature (DSC)	164 - 168 °C	ISO 3146
Heat Deflection Temperature (0.45 MPa)	106 °C	ISO 75-2
Charpy Impact Strength, notched (23°C) <sup>1</sup>	5 kJ/m <sup>2</sup>	ISO 179/1eA

<sup>1</sup>Data should not be used for specification work

<sup>1</sup> Measured on injection moulded specimens according to ISO 1873-2

## Appendix VI

### Extruder Machine Specification



**PURPOSE AND SPECIFICATIONS:**  
**SHEET EXTRUDER, 1000mm SHEET WIDTH, 700-800kg/hr for PS**  
**Including metering gear pump for primary Extruder, Nordson-USA**

**I. Machine Specifications**

- 1) Material: PS / PP (Thermoforming Grade)  
 Main Extruder PS / PP Pellet/Flakes  
 Sub Extruder PS / PP Pellet/Flakes  
 Layers: Two Layers  
 Thickness: 0.15 mm to 2.0mm (Thickness variation around  $\pm$  20microns)  
 Sheet Width: 1000mm finish on winder (After Trim)  
 Sheet output: 700 kg/hr PS (130mm $\varnothing$  Extruder with 50% flakes and 50% virgin)  
 65kg/hr PS (70mm $\varnothing$  Extruder with 50% flakes and 50% virgin)  
 (at 70% regrind, output rates will decrease by a maximum 20%)  
 (at 100% regrind, output rates will decrease by a maximum 40%)

Sheet Thickness (microns)	Output ( kg/hour)
150 – 200	350 – 450
250 – 300	450 – 500
350 – 350	450 – 500
400 – 450	500 – 550
500 – 550	550 – 650
600 – 650	650 – 700

- 2) Line Speed 5m-60m/min  
 3) Machine Color  
 Main Body (White B48880)  
 Safety Cover (NS/V Blue)  
 Control Panel (NS/V Blue)  
 Heat Cover Black  
 Electric Duct Orange

**II. Machine Parts**

- 1) Main Extruder  
 (a) Main Driving Motor 200kw x 4P, AC Inverter Motor  
 (b) Power Transmittal V-Belt (5V)  
 (c) Hopper

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