Purging Mixture for Extruder

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Degree Thesis
Plastics Technology
2015

DEGREE THESIS	
Arcada	
Degree Programme:	Plastics Technology
Identification number:	14224
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Title:	Purging mixture for extruder
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Examiner (Arcada)	Mirja Anderson

Abstract:

This thesis work focuses on compounding a mechanical purge mixture for extruders. The base resin for making the purge mixture is recycled High Density Polyethylene chosen for its high density and good processing temperature. The additives are mainly clay and silicon dioxide added as filler and scrubbing materials respectively. The purge mixture was produced by mixing the base resin and additives in percentage ratios into five places labeled A, B, C, D, and E. the mixtures were extruded and tested for purging effect in comparison to a commercial purge compound, it was observed that the performance of specimen E which contained 87% recycled HDPE, 8.7% clay and 4.3% silicon dioxide had performance near that of the commercial purge compound. The synthesized purge mixture can be applied in extruders for removing resins like Acetal, ASA blends, Fluoro-polymers, Nylon, Polybutylene terephthalate, polycarbonates, Acrylonitrile butadiene styrene, PEI, PET, polyester alloys, polyolefins, PPS, PVC and TPE as well as effect color changes in these resins. Although the purge mixture can be used in extruders for purging resins and colors, its effect in other molding machines have not been tested.

Keywords:	Purge mixture, compounding, Polyethylene, Contami-nanats, Extrusion, Additives,
Number of pages:	53
Language:	English
Date of acceptance:	

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ABREVIATIONS AND ACRONYMS

HDPE	High Density Polyethylene
LDPE	Low Density Polyethylene
UHDPE	Ultra-High Density Polyethylene
PE	PolyEthylene
PP	PolySpropylene
CPC	Commercial Purge Compound
PEI	Polyetherimiden
PVC	PolyVinylChloride
PET	PolyEthylene Terephthalate
PPS	PolyPhenylene Sulfide
TPE	Thermoplastic Elastomers
PC	Polycarbonate
PS	PolyStyrene
ASA	Acrylonitrile Styrene Acrylate
UV	Ultra Violet
Tg	Glass Transition Temperature
FPA	Flouropolymer Based Processing Aid
LMF	Low Melt Flow
HCL	HydroChloric acid
RPM	Revolutions Per Minute

Foreword

My immense gratitude to my supervisor Valeria Poliakova for her support, technical advice and guidance. A big thank you to my Examinar Mirja Anderson for her encouragement and counselling when I decided to start my thesis work. Finally my gratitutde to all my friends especially Bogdan Molchanov for the advice and inspiration.

1 INTRODUCTION

1.1 Background

Though thermoplastic extrusion has been used for decades to mold plastics materials into continuous profile of desirable forms and quality, it has also been a very difficult task for molders to change materials without opening the extruder or using so much plastic material to remove deposits of resins and other contaminants from the extruder's barrel after extrusion.
Before the introduction of purge compounds, molders had options of opening and cleaning the extruder manually or flushing the contaminants with higher molecular weight resins which could push the contaminants away from the extruder. The limitations of these techniques are in the time wasted to open and clean the machine manually, and the inability of more viscous resins to remove certain contaminants like carbon deposits and degraded residue from the barrel of the machine. It is for these reasons that purge mixtures were formulated to clean different forms of contaminants that may be formed during extrusion.

There is growing need for cheap purge compounds in plastic productions as molders need to minimize the overall cost of production in order to maximize profit. Commercial purge compounds however play significant role in quick and safe removal of contaminants from extruders, thereby reducing the machines down time and improving quality in production. The commercial purge compounds are plastic resin compounds with additives that are produced primarily to clean plastic machines when changing color, resin or removing different types of contaminants.

In Arcada's plastic laboratory, different kinds of polymer materials are extruded while running experiments with the extruder, the common method of removing contaminants from the barrel while changing material or color has been to flush the contaminants with regrinds of high density polyethylene (HDPE). Although this method has served the laboratory for years, it is however not effective in shut downs and removing tough contaminants in the barrel. Another problem is the time taken to purge high density plastics from the extruder. Since this is a teaching laboratory serving many students, there is need to produce a cheap purge mixture that can quickly remove contaminants from the barrel while scoring and scrubbing the extruder.

The purge mixture for the extruder is a cheap purge compound that contains a base resin with additives of various types compounded into a melt and grinded into pelet capable of removing different kinds of contaminants from the barrel of the machine.

1.2 The aim and objectives

The Aim and objectives of this thesis work are:

1. To identify the various forms of contaminants in an extruder,
2. To compound a cheap purge mixture that can be used to remove these contaminants from the extruder,
3. To compare the cleaning effects of the purge mixture with commercial purge compounds,
4. To recycle plastic scraps by converting them into purge mixtures,

1.3 Literature Sources

The literature sources for this study combines qualitative research approach with series of laboratory experiments. The qualitative sources reviewed books, articles and different internet sources for information concerning commercial purge compounds, base resins for the formulation of purge compounds, possible contaminants in an extruder and plastic additives capable of cleaning extruders without dame. The laboratory experiments however, provided information on the melt flow index and dispersion of certain additives in base resins.

1.4 Scope of the study

This work is mostly intended for anyone seeking to formulate purge compounds from recycled plastics, in other words the study is not for professionals. The work did not cover the effect of the formulated purge compound on the extruder over a long period of time. The purge compound produced is intended to remove residual resins like Acetal, ASA blends, Fluoropolymers, Nylon, Polybutylene terephthalate, polycarbonates, Acrylonitrile butadiene styrene, PEI, PET, polyester alloys, polyolefins, PPS, PVC and TPE, it is also effective in changing colors and can be used to remove colored resins from the extruder.

1.5 Limitations

Below are the limitations encountered while trying to produce the purge mixture:

a) Unavailability of data as to constituents of commercial purge compounds,
b) The extruder in the lab has low contaminants to test the effect of the purge,
c) Lack of standard procedure to check material removal rate by purge compound,
d) Inability to get Organoclay which was substituted by Green Clay.

2 LITERATURE REVIEW

This chapter reviews the meaning and concepts of purging as applied to plastic extrusion, also the base resin for making plastic purge compounds is reviewed so as to get a better choice of the purge compounding in Arcada's Plastic Laboratory. As the use of additives is paramount to making good purge compound for plastic purging, additives are also reviewed in addition to the possible contaminants that may be present in the extruder after use.

2.1 Purge compounds

There are many options available for cleaning extruder screws and barrels between and after productions. Usually the first step is to empty the extruder completely and using appropriate purge material to remove residual materials from the extruder. However, the extruder could be opened and the screw and barrel cleaned manually. The disadvantage of the later process is the time consumed in opening and cleaning the machine parts manually. In industrial production process, where time management is critically important in saving cost, purge compounds are preferred.

A purge compound is any type of plastic resin produced to clean or remove contaminants from plastic machineries like injection molding machines, extruder, blow molding machines, etc. Generally, purging compounds reduce the total cost of production but are especially important when changing the resin or polymer color in these machines, such that besides having fewer rejects due to defects there is reduction of waste and the machine is kept clean.[2] The processing of modern thermoplastics require the use of purge compounds for cleaning contaminants like carbon build up in screw and barrel of an extruder, die and die sets in order to improve processing efficiency, scrap rates, tool and die life, length of change over time and equipment maintenance. [6]

2.2 Types of purge compounds

There are many commercial purge compounds available in the market, it is however important to identify the appropriate purge to use for the removal of a type of resin or contaminant. The author classifies purge compounds primarily into three types, namely:

1. Mechanical purge
2. Chemical purge
3. Virgin resins or regrinds

2.2.1 Mechanical purge

Mechanical purge compounds are used for physical purging processes, they use abrasives or high viscosity plastic to mechanically scrub away contaminants from the barrels or the machines. The thermoplastic blends consist of a carrier resin and scrubbing granules that soften on the outside but remain solid on the inside so that the metal surfaces are scrubbed safely and thoroughly without wearing the machine. They are safe to use but have limitations in removal of certain contaminants like carbon build up from the extruder. More so there is limited range of application of this kind of purge compound due to temperature as the granules might melt at very high temperatures. Although mechanical purge compounds have long been used in the industry for purging purposes, the side effect of is the surface wearing of the barrel and pitting of molds caused by the abrasives and high viscosity plastics in the purge when used consistently for a long period of time. There are two categories of Mechanical purge compounds, these are

a) Mechanical nonabrasive purge and
b) Mechanical abrasive purge

Mechanical nonabrasive purge

Mechanical nonabrasive purge compounds are stiff materials, normally polyethylene (PE) based Containing cleaning and release agents. The fractional melt flow high density polyethylene (HDPE) functions well as a mechanical nonabrasive purge compound over a wide temperature range as a result of this property, Mechanical purging commonly uses a fractional melt flow HDPE. The stiff material pushes the resin being purged out in front of the HDPE. After the resin being purged is out of the extruder, the barrel temperature can be lowered and more HDPE added to remove the earlier purge material. As the screw is being pulled out of the extruder, any residue HDPE can be brushed off the screw with a brass wire brush to ensure total removal of the resin. Several commercially available materials are produced as purge compounds based on mechanical purging, the advantage is that fractional melt flow HDPE is cheap, works out well and has a wide processing temperature range.

Mechanical abrasive purge

Mechanical abrasive purge compounds are based on low viscosity abrasive mineral or glass filled material that can force resins out of an extruder while scouring and scrubbing the screw, barrel and die. Based on the abrasive additive particle size, screen pack may have to be removed to prevent them from clogging. Depending on the resin system and filler, abrasive systems can be costly, such as glass filled polycarbonate (PC), which is an excellent purging compound to clean PC or polyether amide out of an extruder. However PC is an expensive material to use for a purge resin. Cast acrylic is another material that can be used for making abrasive purge compounds, it does not completely melt in the extruder due to its high melt viscosity however it is better to remove the die prior to purging. Once cast acrylic is in the barrel, it has to be purged out or the screw removed and mechanically cleaned.

2.2.2 Chemical purge Compounds.

This type of purge uses chemical compounds to break down polymer residue in the machinery thereby reducing their molecular weight and viscosity in order to flush them out easily from the machine. The chemical purge reacts with the materials left in the barrel by plastication of the resin or causing depolymerization of the polymers into lower molecular weight components to lower the viscosity so that it can be purged out. Whereas the mechanical purges force materials out with a high viscosity and scrubbing action, chemical purges react with the residue and make it easy to remove from the barrel. The surfactant purging material uses surfactant that penetrates and loosens residue on the barrel, screw, and die, dispersing it in the melt. The surfactant is mixed with a melt flow resin that is 0.1 to 0.3 times that of the original melt to provide maximum purging effectiveness. The mechanism is using a thermally stable surfactant –type additive that attaches to any residual polymer or polymer build up to loosen it from the barrel, screw and die dispersing it into the polymer melt. The surfactant function is to bond the anionic and nonionic surface active agents to the degraded polymer or gel, softening and loosening the particles from the metal, allowing it to be carried out with the viscous polymer melt. Normally the purge is sold as an additive or concentrate to be added to existing resin or to be mixed with PE, PS, or cast acrylic as carrier.[1]

Chemical purge is a popular choice for many molders because of the high moldablity rate as scrap generated can also often be used to remold parts. It is also a faster and more efficient way to remove residue due to the chemical reaction with resin and pigments considering the small quantities of purging material required and a wide variety of purging applications. The use of commercial grade chemical purging compound is considered a sound investment that can greatly minimize a company's production cost and ensure optimal functioning of the machinery. It is however susceptible to degradation and may not be very effective in removing carbon build up in extruders. Due to the soaking time required during usage, chemical purges can take longer downtime and cause a loss in productivity due to time. Some chemical ingredients might react with the surface parts of the machinery causing staining, pitting or wearing. Some chemical purge compounds are concentrates that require accurate measurement and mixing with the carrier resin, their effectiveness is reduced if they are not blended or dispersed properly in the carrier resin.

2.2.3 Virgin resins or regrinds.

These are pure resins that are used as purge compounds. They are not commercial purge compounds but are useful in removing resins of the same family and color from the extruder. These purge compounds are however more expensive and less effective to use than the commercial purge compounds. Virgin resins or regrinds cannot clean carbon deposits or negative flow areas in the extruder, moreover wearing may occur with continuous use of these resins because large quantity of the resin may be needed to purge a little contaminant out of the barrel due to the fact that they are not specially made to remove special contaminants from the extruder and may become contaminants by itself requiring a purge compound to purge them out of the extruder. [1][2][4]

2.3 Importance of purge compounds in Plastic molding

There are many benefits for using purge compounds in plastic molding, below are some of the benefits

a. Reduced wastage of materials during production
b. Reduced downtime
c. Reduced number of rejects
d. Facilitated changeover of materials and color
e. Reduced overall cost of production
f. Highly usefull in shutdowns

2.4 Extruder contaminants and their formation

Contaminants can cause disruption and quality problems in the extrusion process. These contaminants range from solids to liquids and gases produced during the extrusion process, common contaminants deposited in the barrel in an extrusion process are:

a. Un-melted resin
b. Degraded residue
c. True gels
d. Foreign contaminants
e. Un-dispersed additives
f. Un-dispersed modifiers
g. Moisture
h. Air bubble or void

2.4.1 Un-melted resin

Un-melted resin is the common cause of contamination and quality problems during extrusion, they formed when the extruder pumps the resin faster than it can melt it, causing deposit of solid or soft resin in the barrel. Un-melted resins can be difficult to test for and resolve, it can however be identified by examination of an extruded material for presence of previously extruded material. For instance if a plastic resin has the same color as the previously extruded base resin, it is most likely that the base resin is the cause of the contamination. The quick fix for this kind of contaminant is to slow down the extruder until the problem goes away or addition of denser screen packs to increase back pressure and improve melting. the pictures below show a screw with this type of contaminant before and after purging with a purge compound.

Figure 1 Screws containing un-melted resins before and after purging with [11]

2.4.2 Degraded residue

Degraded residue results from resin that stagnates on process surfaces and degrades. The resultant materials can then release from the surface and contaminate the flow. When resins and additives degrade in the extrusion process the products form carbon and other compounds. If the resulting contaminants from the machinery involves a lot of brown and black specs which are not sourced from other contaminants, it is most likely to result from carbon deposits. This Carbon is formed when the resin is left in the machine at high processing temperatures in the presence of air over time. Metal adhesiveness increases during carbon development and progression causing deposits to form in the barrel. The carbon deposits and other contaminants formed this way can be removed using appropriate purge compounds and by mechanical cleaning. The picture below shows two screws with degraded residue.

Figure 2 screws containing degraded resin [16]

2.4.3 True gels

A true gel is defined as a very high molecular weight version of the same resin, and may be cross-linked such that its viscosity is very high and will not disperse into the melt stream. True gels usually have similar optical properties as the base resin, but clog screen packs and therefore distort extrusions.

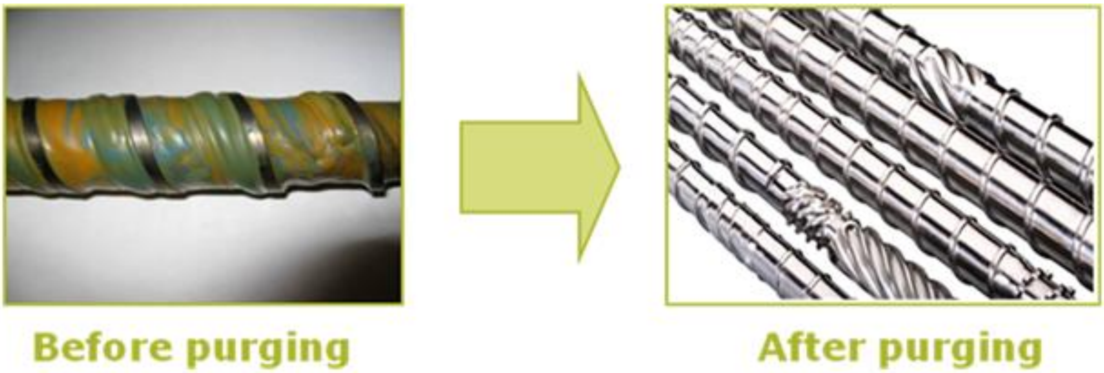

Figure 3 screws containing un-dispersed gels before and after Purging [17]

2.4.4 Foreign contaminants

Foreign contaminants are materials like wood from pallets, paper, scrap plastics, dust, transfer pipe cross contamination, pallet stuck to shoes and any other material that is not part of the material to be extruded but enter the extruder at any point during the extrusion process. These contaminants can be detected using microscopes periodically during production to examine the extruded resin.

Figure 4 Section of a screw showing contaminants [20]

2.4.5 Un-dispersed additives

Usually, additives are added to resins in powder or as master batch pellet for inclusion in the melt. The additives are melt-mixed, dispersed and distributed in the resin. When powdered additives are mixed directly with resins in an extruder, they often experience high pressure and form agglomerates which are very difficult to break down after they are formed. The result is a white or colored contaminant in the extrusion. Agglomerates that are visible to the eye are normally not supplied by the additive powder producer, but a screen mesh test of the powder can be performed to rule the additive supplier out as the possible source. One of such additive powders are Modifiers in powdered form, typically these type modifiers are added in the reactor or in an extrusion compounding step to improve the properties of the resin. However, the improper dispersing of the modifiers in the melt can cause contamination in the extruder.

Figure 5 Screw contaminated with un-dispersed additives [11]

2.4.6 Moisture

Moisture is a very common source of defects in materials after extrusion. Some resins can be processed with higher moisture as long as the extruder is vented. However, different resins react differently to the presence of moisture in the extruder. Some resins will degrade when moisture is present, resulting to a low viscosity melt while others may form bubbles and affect the molding process. [5]

2.5 Base resin for the purge compound

Although there are different base resins that could be used for making purge compounds, this thesis work focuses on High Density Polyethylene (HDPE) as polymeric material for purge compounding due to its good chemical, thermal and physical properties among others. This thermoplastic material can be used as base resin in purge compounds for removing a range of contaminants from an extruder as it readily combines with contaminants of various types and flush them out of the extruder. The choice as to the base resin for making a purge compound is highly dependent on the ability to remove a wide range of polymeric materials and other contaminants without damaging the extruder. The density of the carrier resin for the purge compounding is usually higher than that of the resin to be removed when considering mechanical mechanism for purging. More so, the carrier resin should readily combine or carry the contaminants during the purging process. Resins that do not readily combine with the contaminants or have Viscosity high enough to push the contaminants will be less effective for use as base resin for mechanical purge compounds since the mechanism for this process is based on viscosity and ability to mix.

2.5.1 HDPE a better choice for the purge mixture.

High density polyethylene has a wide range of purging applications when used as a base vresin for purge mixtures. HDPE can be used to purge Acetal, Acrylonitrile Styrene Acrylate (ASA) blends, Fluoropolymers, Nylon, Polybutylene terephthalate, polycarbonates, Acrylonitrile butadiene styrene, Polyetherimide (PEI), Polyethylene terephthalate (PET), polyester alloys, polyolefins, Polyphenylene sulfide (PPS), Polyvinyl chloride (PVC) and Thermoplastic elastomers (TPE). [1] Besides this wide range of applications HDPE also has good physical, chemical and thermal properties that are useful in in purge compounding.

2.5.2 Properties of Polyethylene (PE)

Polyethylene is a long chain polymer of repeated units of ethylene. Ethylene is a simple hydrocarbon with a pair of double-bonded carbon atoms and four hydrogen atoms, it has the formula C2H4. It has a central chain of carbon atoms each bonded to two Hydrogen atoms that may be branched or un-branched depending on the type of PE. The structural differences between PE types help in the determination of their functions. Both HDPE and LDPE are common plastics made from Petroleum. Below is a structural diagram of ethylene and polyethylene.

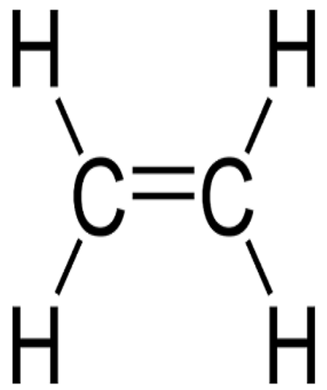

Figure 6 structure of ethylene [9]

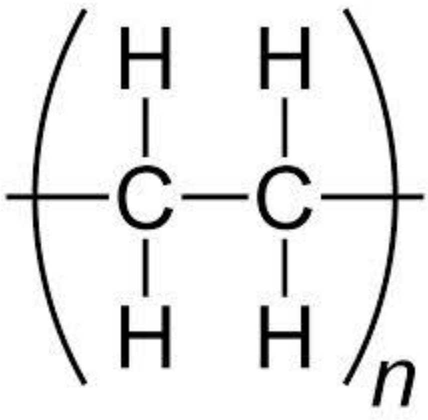

Figure 7 Structure of polyethylene [9]

HDPE is un-branched and as a result the polymer chains can be packed together much more tightly than LDPE, this makes HDPE denser than other members of the group. Though there are exceptional cases of PE formed from long chains of Ultra high molecular weight Polyethylene (UHMWPE), they have higher density than the Un-branched PE. Below is a density distribution of PE

a) Branched Low Density of 0.910 - 0.925 g/cc
b) Medium Density of 0.926 - 0.940 g/cc
c) High Density of 0.941 - 0.959 g/cc
d) Linear High Density to ultra-high density > 0.9

The branched chain of LDPE gives it a lower density than HDPE, it is also more flexible and easy to clean. The differences in structure and density make HDPE tougher, stronger, and more opaque than LDPE. HDPE also has better ability to withstand heat and resist chemicals than LDPE. [8][9] The melting temperature of LDPE is lower than HDPE, although this property would make LDPE a good material for purge mixture but due to the density and other physical properties, HDPE is preferable. Below are tables showing the different properties of PE.

Mechanical Properties of different types of PE

Table 1 Mechanical Properties of Polyethylene [9]

	Branched Low Density (LDPE)	Medium Density(MDPE)	High Density(HDPE)	Linear High Density(UHDPE)
Density(g/cm^3)	0.91- 0.925	0.926- 0.94	0.941-0.95	0.959-0.965
Crystallinity(%)	30 to 50	50 to 70	70 to 80	80 to 91
Molecular Weight(g/mol)	10K to 30K	30K to 50K	50K to 250K	250K to 1.5M
Tensile Strength, (Mpa)	4.13 – 15.86	8.27 – 20.68	21.37 – 37.92	34.47 – 41.37
Tensile Modulus, (Mpa)	172.31 – 282.69	262 – 517.10	1034.21–1089.37	1034.21–1089.37
Tensile Elongation, (%)	100 - 650	100- 965	10 - 1300	10- 1300

The table above shows that besides UHD, HDPE has better density, crystallinity, molecular weight, Tensile strength, modulus elongation, impact strength and hardness than the other members of the group.

Physical Properties of polyethylene

Table 2 Physical Properties of Polyethylene [9]

	Branched Low Density	Medium Density	High Density	Linear High Density
Optical	Transparent to opaque	Transparent to opaque	Transparent to opaque	Transparent to opaque
Tmelt (˚C)	98 – 115	122 – 124	130 – 137 C	130 –137
Tg(˚C)	-100	-100	-100	-100
H_20 Absorption	Low < 0.01	Low < 0.01	Low < 0.01	Low < 0.01
Oxidation Resistance	Low, oxides readily	Low, oxides readily	Low, oxides readily	Low, oxides readily
Alkaline Resistance	Resistant	Resistant	Resistant	Resistant
Acid Resistance	Oxidizing Acids	Oxidizing Acids	Oxidizing Acids	Oxidizing Acids

The table above shows the different physical properties for the different types of Polyethylene. Besides their melting temperature that increases from low to ultra-high density PE, the other physical properties like, optical properties, glass transition temperature, water absorption, oxidation resistance, Solvent resistance, alkaline resistance and acid resistance are constant for the different types of PE. These properties are very important as they show the author what the application temperatures and possible reactions with certain substances which are added to the purge as additives during synthesis. For instance HDPE has a melting temperature of 130˚C, and therefore cannot be used as base resin to purge plastics with melting temperatures higher than 130˚C.

Processing Properties of different types of PE

Table 3 Processing Properties of different types of Polyethylene [9]

	Branched Low Density	Medium Density	High Density	Linear High Density
Tmelt(˚C)	98 – 115	122 – 124	130 – 137	130 –137
Recommended Temp Range in Extrusion(˚C)	121C – 232C	121C – 232C	177C – 274C	177C – 274C
Molding Pressure(Mpa)	0.034– 0.100	0.034 – 0.100	0.082 – 0.100	0.082– 0.100
Mold (linear) shrinkage (in/in)	0.015 – 0.050	0.015 – 0.050	0.015 – 0.040	0.015 – 0.040

The table above shows the different processing temperatures of the various types of PE in extrusion, the processing temperature and pressure for molding fairly increases from LDPE to UHDPE.

The comparison of HDPE and other members of the group points out HDPE as a good polymeric resin in terms of physical, chemical and thermal properties which are also applicable in purge compounding for a mechanical purge.

2.5.3 Advantages and Disadvantages of PE

Advantages of Polyethylene

I. Lower cost.
II. Broad range of properties and applications.
III. Good density for a purge compound.
IV. High chemical resistance properties.
V. Low moisture absorption.
VI. High chemical resistance.

VII. Good for food packaging
VIII. Good processing temperature.

Disadvantages of Polyethylene

I. High thermal expansion.
II. Low stiffness with standard grades.
III. Low UV resistance.
IV. Low oxidation resistance
V. Not transparent like PP

2.5.4 Recycled High Density Polyethylene

The availability of HDPE is another important characteristic for choosing it as a purge resin for making the purge compound. HDPE is recyclable and also one of the most used plastic material around the world. For instance, recycled HDPE is readily available in Arcadas plastic laboratory. It is usually produced by cutting molded HDPE materials originally produced for experimental purposes into smaller granules that can be remolded and reused as purge for extruders. This recycling process makes the raw material for the purge mixture readily available.

2.6 Plastic Additives

Plastic additives are foreign substances or materials added to enhance the properties and performance of plastics for specific applications. Before the discovery of additives early plastics were usually unsatisfactory due to lack of desirable characteristics like shapes, durability and poor processing among others. Plastic additives are now essential in plastic molding even though they cost money in the short term and incorporating them into plastics can be an additional expense, however they reduce overall production cost and make products last longer. Additives help to save money and conserve raw material reserves. Processing plastics into useful and saleable articles without additives is virtually impossible. In the production of purge compounds additives are very important as they serve as the essential ingredient for the purge. As stated earlier, purge compounds com-pose of the base resin and appropriate additives for specific purposes. Below are general functions of plastic additives an why they are important in purge production.

2.6.1 Functions of plastic additives

Below are few general functions of additives in Plastic molding. Additives:

a. influence the physical properties and resistance to deterioration in plastics,
b. reduce defects and rejects,
c. adjust flexibility without deterioration of physical properties,
d. reduce cost of production,
e. modify mechanical, thermal and electrical properties,
f. lower processing temperatures and increase output and
g. modify chemical properties of the resin to get a desirable property.

2.6.2 Common additives used in plastic molding.

The formulation of a purge mixture involves the compounding of additives and the carrier resin, it is important to outline some additives used in polymer processing and their effect in the melt. Below are some common plastic additives of which some have been added to the purge mixture.

Fillers

Fillers are inorganic particles added to thermoplastic polymers to increase their Young's modulus, improve thermal stability and reduce wear under friction. Fillers however lead to a reduction in the fracture strain of polymers by embrittlement. In filled polymers many modes of deformation are distinguishable, for instance brittleness, quasibrittle fracture during neck formation, fracture during neck propagation, stable neck propagation, micro-uniform yielding and yielding in crazes. In filled polymers crazes are caused by debonding of particles and by stretching of polymers in the space between the neighboring particles. The thickness of the fibers is therefore determined by the distance between the neighboring particles. Fillers like gypsum, calcium carbonate (lime stone), kaolin (clay), Talc and Alumina trihydrate can also be added to polymer resins to enhance performance and reduce manufacturing cost. They make polyester resins more chemical and corrosion resistant, act as fire retardant, enhance shrink resistance and thermal stability as well as increase their ability to withstand adverse weather conditions. [14] The filler used in the purge compounding is the organo clay, this clay absorbs dirt in the extruder and also have good combination properties with the carrier resin as well as the contaminants. Organoclay is an organically modified phyllosilicate, usually derived from a naturally clay mineral, it consists of bentonite, composed mainly of the clay mineral montmorillonite, which is modified with quaternary amines a type of surfactant that contains Nitrogen ions. The bentonite in its neutral state is capable of absorbing up to seven times its weight in water. When treated with amines the absorption rate becomes 5 to 10 per cent of its weight in water, and 40 to 70 per cent in oil, grease, and other sparingly-soluble, hydrophobic chlorinated hydrocarbons. [26] Organoclays are produced by reacting natural clays with intercalants such as surfactants. The absorption effect of the organoclay is the main reason for using as a purge filler to absorb contaminants in the extruder.

Calcium carbonate accounts for about 65% of total filler consumption in plastics with an annual world-wide usage of about six million tones. It is also the most widely used filler in terms of number of applications. The cheapest grades are low in price and are used primarily to reduce costs. By contrast, the finest grades are an order of magnitude higher in price and are used to modify various properties, both during processing and in the final compounds. Due to the cheap cost of calcium carbonate, it is used in high quantities to reduce the cost of production. Ground calcium carbonate and precipitated calcium carbonate products serve as functional fillers in plastic and rubber applications. Calcium carbonate is also the major engineered filler used in plastics with more than half of the total mineral consumption as it is widely used in polyvinyl chloride (PVC), polyolefin, polypropylene (PP), polyethylene (PE) and unsaturated polyester resins applications. Calcium carbonate is used for its excellent optical properties, ability to improve impact strength, role as a processing aid and ability to replace expensive plastic resins. In conclusion, the predominance of calcium carbonate in filled plastics is primarily related to its widespread occurrence as white and pure mineral deposits, combined with the low cost of processing. However, in many large volume applications, it gives important functional benefits and would still be the filler of choice, even if prices were increased [14][15]. Talc is also commonly used filler in plastic molding, it is used to stiffen polypropylene in the manufacturing of automotive parts, house hold appliances and engineering plastics.

Scrubbing agent

Scrubbing agents are materials added to purge compounds to scrub contaminants off the extruder while reinforcing the plastic resin. One very important additive needed for making mechanical purge compounds is a scrubbing agent, it is needed to scrub the extruder barrel during the cleaning process. Although for this thesis work Silicon Dioxide has been chosen for this purpose but a better option would have been glass fiber due to its particulate nature. Silicon dioxide also known as Silica is an oxide of silicon that exists in nature as quartz. It has very high melting point of 1600˚C to 1725˚C, it is crystalline and hard, properties that makes it a good scrubbing agent when added to purge mixtures. There are different types of glass fibres that can be used to reinforce plastics, the most widely used is E-glass, where the silica network is modified by other oxides like calcium, aluminum and boron. S-glass, with higher aluminum content and magnesium has higher tensile strength. Glass fibres are formed by melt spinning - for E-glass, gravity fed extrusion through fine holes at a temperature typically of about 1300°C. The fibers are attenuated by being collected on a rotating drum at a speed higher than the extrusion velocity to produce a continuous-filament strand, with average diameters in the range 2 to 25 µm, according to the attenuation conditions. Short fibers are produced either by chopping continuous filaments to the required length or by melt blowing, a process in which air directed at the freshly extruded fibers extends them, and which produces short fibers of variable length and diameter. [15]. Although Glass fibers are dimensionally anisotropic, they are also structurally isotropic, apart from minor differences between the surface and the interior. They are very brittle to protect them against surface damages leading to crack initiation during production and storage, and also to improve their affinity with and adhesion to polymer matrices, by applying sizes and coupling agents. When added to PP, it improves the physical properties such as thermal property and dimensional stability while maintaining the excellent surface finishes of the product. Ground fiber is mainly used as material for improving the molding properties of different kinds of composites and their physical properties. It displays effectiveness particularly when used in Reinforced Reaction Injection Molding (R-RIM), Fiber-Reinforced Thermo-Plastic (FRTP) and also in coatings and gel coats to prevent cracks. In purge compounds it plays significant role in scoring and scrubbing residue contaminants in the extruder. In this thesis work the clay added to the carrier resin acts as a scrubbing agent.[10] [15]. The combination of polymers and fillers and other additives produce a new class of polymeric composites with desirable properties and characteristics.

Processing aid

The ease with which melt is forced through the molding machines depends on the physical and chemical properties of the plastic material in use, it is for this reason that processing aids are important in molding plastics as they improve both the physical and chemical properties of plastics. Processing aids enhance product quality and processibility by eliminating surface defects and reducing torque and power requirements as well as die plate pressure. During material processing in extruders, the process aids become liquid and form a film around colored particles so that they mix better, with other additives the polymer particles adhere better and melt more quickly thereby lowering the molding temperature and reducing heat damage to the plastic. Depending on the type of carrier resin used for forming the purge mixture, a processing aid might be essential, for instance some plastics like PVC are very difficult to process as they become viscous and sticky when they melt, processing aids like lubricants therefore help to reduce viscosity by creating a film between the polymer melt and barrel. A very good example of this processing aid is the flouropolymers. Flouropolymer based processing aids provide a range of benefits like melt fracture elimination, improved production capacities, better control of molecular orientation and final physical properties. Polypropylene does not suffer from melt fracture as much as HDPE and LDPE, however Flouropolymer based polymer processing aids can lower PP viscosity and extruder pressure and eliminate die drool. However for mechanical purge compounds such as the one produced in this work, the processing aid was not used as the processing temperature of HDPE is high enough and the density good enough to ensure intercalating of materials and components in the mix. It is however important to note that this type of processing aid might be used to improve the properties of a purge compound especially when working with big extruders and a wider range of plastic materials.

Surfactants

Surfactants are important additives in the manufacturing of purge compounds as they increase compatibility of the mixture. Surfactants are additives that consist of hydrophobic and hydrophilic groups covalently bonded together. In other words, they are raw materials that possess both water-soluble and oil-soluble characteristics. The hydrophobic 'tail' of a surfactant is a hydrocarbon chain containing 8-16 methylene groups that is either straight or branched chain. In applications the single tailed surfactants are most widely used however, a number of double tailed compounds are also in use. The hydrophilic head group of a surfactant might be a charged or uncharged polar group. The charged polar groups form ionic surfactant, like all salts which must contain a counter-ion in order to be electrically neutral. Cationic surfactants are situated around quaternary nitrogen atoms and are usually available as chloride or bromide salts. For instance Anionic surfactants likealkylsulfates and alkylbenzenesulfonates show these common structures. Non-ionic surfactants however contain uncharged polar groups, for instance oligomeric ethylene oxide chain. The number of ethylene oxide units in this type of non-ionic surfactant may vary from 2 to 100 or even more. Industrially non-ionic surfactants are made by a condensation reaction of ethylene oxide with an alcohol or alkylphenol, hence contain a statistical distribution of ethylene oxide chains. There are other forms of surfactants that use sugar residue as their hydrophilic part and are important because of their biodegradability. [15]

In most cases, solids like metal particles, mica, pigments and titanium dioxide used in a dispersed state in polymers have high energy hydrophilic surfaces which can be made more compatible with polymers by coating their surfaces with a layer of surfactant in form of dispersants. Surfactants like Sodium di-alkyl sulphosuccinates, used as wetting agents, is commonly used for this purpose. At the polymer-polymer interface, surfactants like block copolymers improve the compatibility of polymers by reducing the interfacial tension between the components of the polymers and adsorbing at interphase surrounding the domains. This is important in compounding because of the immiscible nature of most polymer blends due to the entropy of mixing high molecular weight compounds as plastics. In the form of wetting agents, surfactants are added to polymer surface to in order to produce complete spreading of water as continuous layer rather than forming contact angles that can make wrapping films unclear. For instance mono and di-glycerides are used as food grade surfactant for this purpose. As foam control agents, Surfactants are added during the foaming stage to influence the rheology and gas interface of the polymer, in order to control the stability of the thin polymer film between adjacent gas bubbles in the foam. Silicone based surfactants are commonly used for this purpose especially for polyurethane foams. As Lubricants surfactants are added to polymer resins to improve their flow characteristics during processing an important characteristic needed in purge compounds while using to remove contaminants from the extruder.

Heat Stabilizers

In general, Heat stabilizers are used to prevent thermal degradation of materials while processing. Typical additives used for this purpose are soaps which are metal salts of alkyl carbonates or metal salts of other organic acids like phenol. The commonly used cations in this case are tin, zinc, calcium, barium, and cadmium. Tin salts are especially useful in PVC. When PVC is heated C-Cl bonds disintegrates at the weakest points of the polymer chains, for instance in allylic or tertiary positions which occur at branching sites in the polymer or at sites adjacent to unsaturated terminal bonds. The Cl radical abstract hydrogen from adjacent CH groups and create another weak allylic C-Cl bond which disintegrates easily to form HCl unzipping reactions. Basic metal soaps are commonly used to prevent such degradation since they have a variety of actions like neutralizing HCl to stopm the autocatalytic chain reactions, displacing chlorine from the polymer chains and replacing it with an alkyl ester group, preventing free-radical processes such as oxidation reactions and also disruptingconjugation in the polymer chains from which HCl has been removed, thus inhibiting discoloration of the plastic. [15].

2.7 Mixing and compounding of additives with polymers

Compounding in plastic extrusion refers to the formulation of plastics by mixing or blending polymer resins with additives in a molten state using machines. This is the method that has been adopted for the formulation of the purge mixture in this thesis work. The mixing of plastic additives with polymers resins involves different processes and machines which can be broadly categorized into two types, batch mixing and continuous mixing processes.

2.7.1 Batch mixing

In batch mixing the compound is manufactured in discrete quantities, the mixers are usually two roll mills and internal mixer. The two roll mills are slow, manual and could pose health effects to the operator. These limitations limit the use of two roll mills for laboratory scale activities irrespective of their effectiveness in mixing polymers and additives. The mills are made up of a pair of contra rotating rolls that are 300mm wide and 200mm diameter for laboratory type mills. Mill mixing may be slow and labour intensive but the quality of mixing produced is high because of shearing encountered in the process, such that the compound temperature can be controlled very well. Prolonged periods of mixing are possible with little risk of degradation. Internalbatch mixers consists of a two roll mills enclosed in a chamber where the mixing is done. The disadvantage of this method is in the exact volume measurements of the materials to be mixed. With very large size of the batch, the unmixed material remains at the base of the feed throat at the end of the mixing cycle. Smaller batch makes the process slow and inefficient. The volume of a batch is about 66% of the total internal volume of the mixer.

2.7.2 Continuous mixing

In continuous mixing, the components of the mix are fed continuously into the machine. Usually continuous mixing is carried out using either single screw extruders or twin screw extruder.

Single screw extruders

The purge mixture produced in this thesis work was done using a single screw extruder, the mixing process was continuous till a desirable mixture was achieved. The single screw extruders are generally poor in terms of dispersing and distributing additives in the mix. To use the single screw extruder, the additives would have been previously compounded into the polymer to form a uniformly blended feedstock, or compounded into a small quantity of the polymer to produce a master batch or added as liquid which can be injected into the extrude part way along the barrel. Mixing processes in a single-screw extruder is laminar and the materials being mixed exist as discrete layers. The process of mixing involves a reduction in the striation thickness, this being related to an increase in interfacial area between the major and minor components of the mixture. Mixing in single screw extruders is affected by the die head pressure, as the higher the pressure the lower the flow rate and the higher the melt temperature. Another factor that could affect mixing is the screw cooling, if the entire length of the screw is cooled with cooling water, high melt pressure might develop even enough to deform the barrel of the extruder. Finally, reducing the barrel temperature increases shear on the melt.

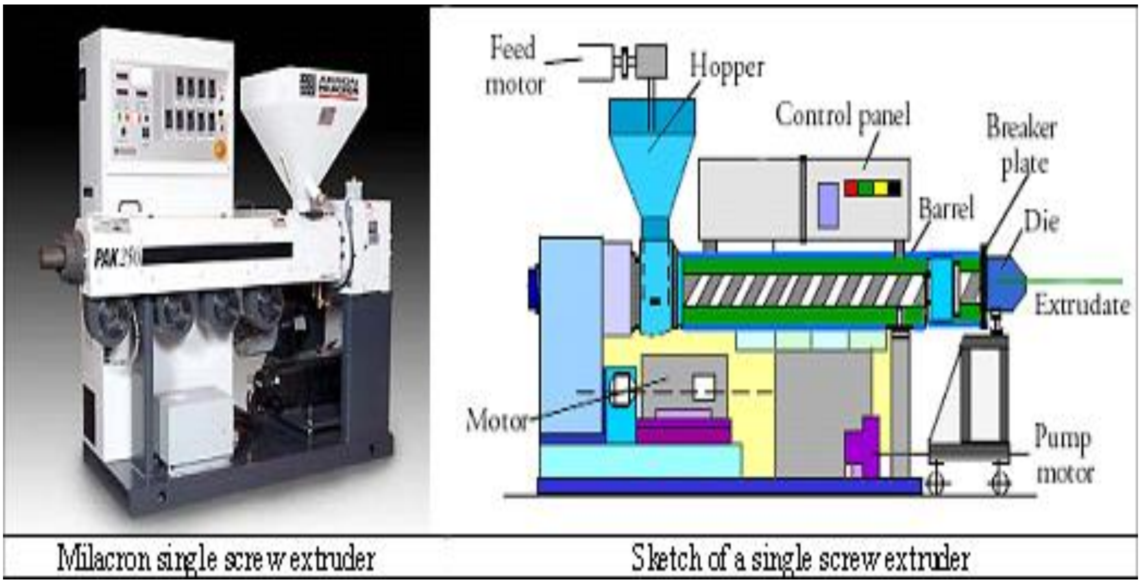

Figure 8 Cincinnati Milacron Single screw extruder PAK 250 [19]

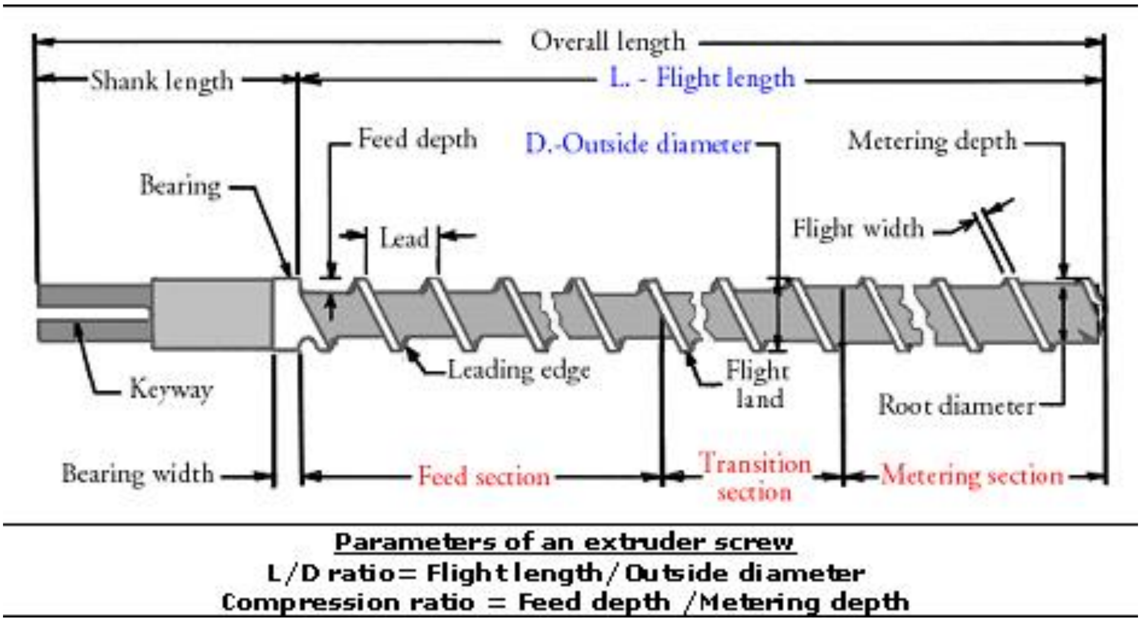

Figure 9 Parameters of an extruder screw [19]

Twin screw extruders

Besides the single screw extruder, twin screw extruders can also be used for mixing. In fact the twin screw extruder is a better machine for the purge formulation. Twin screw extruders can either be non-intermeshing or intermeshing. The twin screw extruder is said to be non- intermeshing or tangential if they contain two single-screw extruders in the same barrel. This type of extruder is not a good mixer. Intermeshing extruders however have co or counter-rotating screws, conjugated or non-conjugated screws and parallel or conical screws. As the compounding process requires continuous feeding of the additives into the extruder, this can be achieved using volumetric or gravimetric (loss-in-weight) feeder systems. The gravimetric systems are more accurate and also more expensive to use as mixer. Volumetric feeders are usually calibrated before use so that feed compositions can be maintained. Modern intermeshing machines are often based on a system of segmented screw and barrel components, this allows for development of specific screw and barrel geometries to suit a wide range of compounds. The advantages of twin-screw extruders over single-screw machines in terms of intermeshing include better mixing and pumping of the melt in addition to less sensitivity to die head pressure. [15]

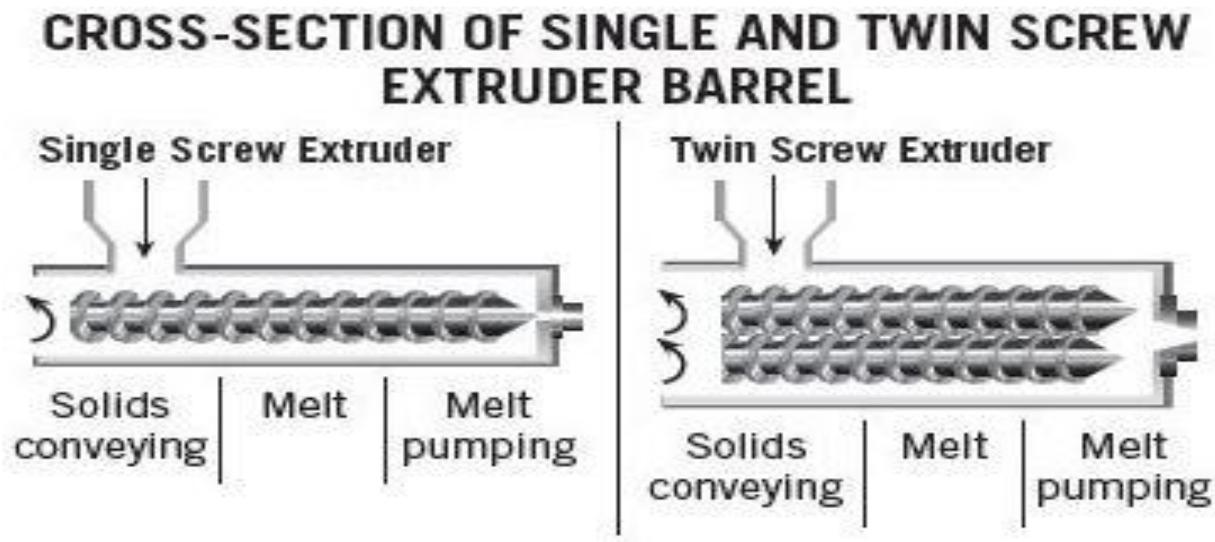

Figure 10 cross Section of single and Double screw extruder barrels [18]

Figure 11Twin screw and barrel [22]

Figure 12 Kaichi Parallel Twin Screw Extruder SHJ 75 [21]

3 METHODOLOGY

The method used for the production of the purge mixture is compounding and testing to check the effect of the compounded purge on contaminant removal. The compounding process involves mixing the additives and base resin in ratios while the testing involved running the mixture through the extruder after the extrusion of a known plastic material.

3.1 Equipments, materials, Additives and resins used

3.1.1 Equipments

Below is a table showing the equipments used for the production<

Table 4 Equipment

S/N	Equipment	Model
1	KFM single screw extruder	Eco Ex

2	Labotek flexible modular drying unit	FMD-MM-25-40-v
3	Rapid shredder	150 series

3.1.2 Materials

Table 5 materials used and details

S/N	material	grade	manufacturer	Purpose
1	CPC	U-T4307	ASACLEAN	Purging and comparison
2	Recycled HDPE	HMA/025		Base resin
3	pellets of HDPE	HMA/025		Purging and comparison
4	Silicon dioxide	53-73% purity, 0.20g/cm^3 density and 40 – 80 µm in size	KEVRA	scrubbing agent
5	Green Green clay	Containing Smectites, Illites, Chlorite, Kaolinite, Dolomite, Calcite, quartz, Feldspar. Silica (Si02) 35.79&, Titanium (Ti02) 0.62%, Aluminium (AI203) 11.64%, Iron (Fe203) 5.00%, Magnesium (Mg0) 1.98%, Calcium (Ca0) 14.90%, Sodium (Na20) 0.73%,Potassium (K20) 2.18%,Waste on fire 21.94%	ARGITAL	filler
6	Precol Blue	PE 323		Colorant

3.2 Experimental Procedure

3.2.1 Recycling HDPE

Used HDPE plastic materials (mainly dog bones produced in Arcadas Plastic laboratory and for tensile testing) were collected, washed and recycled by passing them through the shredder. After shredding, the product was dried in the plastic dryer for 2hrs to remove moisture and any intercalating material on the surface of the recycled plastic. The recycled plastic was then used for the purge mixture production.

Figure 13 Rapid Shredder 150 series [Author]

Figure 14 Recycled HDPE for the purge Mixture [Author]

3.2.2 Extruding the mixture

Five different samples of the purge mixture was prepared and labeled A,B,C,D, and E. each purge specimen contained the base resin, SiO_2 and green clay in constituent as shown below:

Table 6shows the constituents of each purge mixture

S/N	Recycled HDPE(%)	Silicon dioxide(%)	Green clay(%)
A	90.9	7.3	1.8
B	83.3	12.5	4.2
C	71.4	14.3	14.3
D	90.9	4.5	4.5
E	87.0	4.3	8.7

Each specimen was extruded after cleaning the extruder with the commercial purge compound and removing remnants of the commercial purge by purging with pellets of HDPE. The products each specimen extrusion was collected and labeled after pelletizing and drying at 80˚C for 2hrs.

Figure 15 Picture of the colorant used[Author]

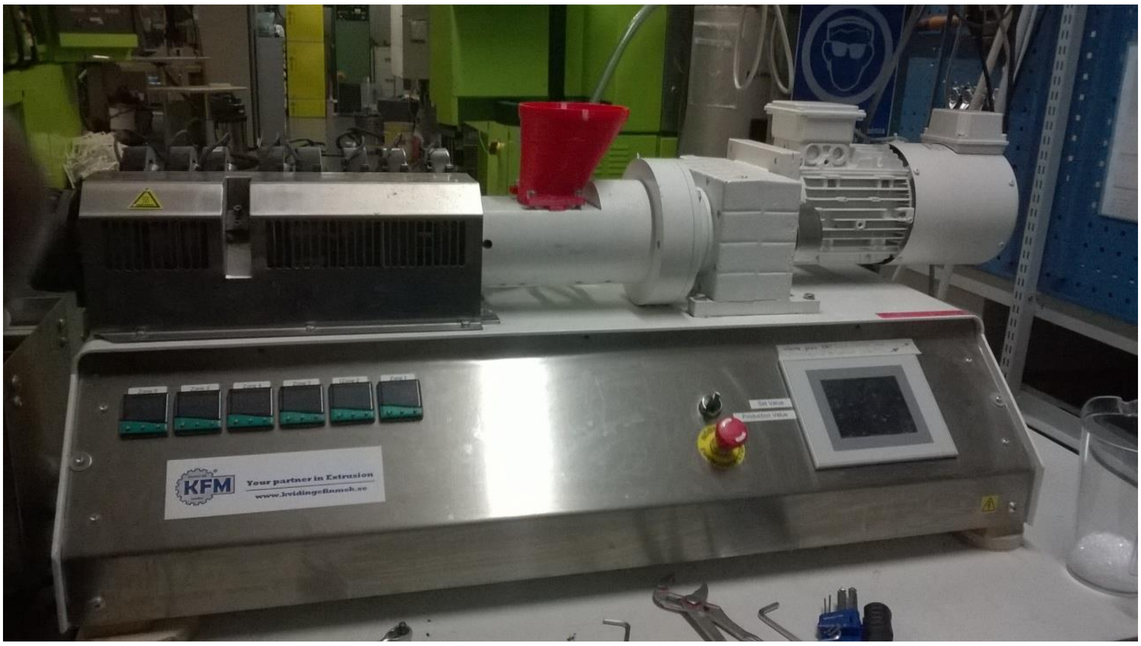

Figure 16 KFM single screw extruder Eco Ex [Author]

Figure 17 Picture showing the extrusion and pelletizing process [Author]

3.3 Safety consideration

To ensure safety and proper production of the purge, the following should be taken as precaution while carrying out the process:

a) Ensure that the clay is dry before usage,
b) Some colorants and additives might react with the mixture and produce dangerous gases to avoid this, check for reaction with clay and SiO_2 before use,
c) Ensure that the plastic resins are dry before extrusion to avoid degrading purge mixture,
d) Use breathing mask when working with SiO_2 as the particle size is easily inhalable and hazardous.
e) Mixing of purge should be done at low speeds to ensure homogeneity.

3.4 Testing the purge mixture

Testing the purge mixture required comparing the effectiveness of the mixture with that of a commercial purge compound. The commercial purge compound used in this case is a mechanical purge designed to remove residue resins from extruders as well as change colors, the weight of the purge was taken as well as the time for purging. Equal amounts of each specimen mixture were used to purge equal amount of HDPE containing 3% precol colorant from the extruder. After each purging process, the extruder was cleaned with CPC and purged with HDPE before repeating the process for another specimen.

Figure 18 Labotek flexible modular drying unit FMD-MM-25-40-v (Author)

4 RESULTS AND DISCUSSION

4.1 Result of the tests

The result of the specimens testing and commercial purge compounds are summarized in the table below.

Table 7 shows the result of the testing using specimens A,B,C,D,E and Commercial purge compound20g of HDPE with 3% precol colorant.

S/N	Quantity used(g)	Purge status	Time of purge(min)	Material & color removal rate(g/Min)
A	180	complete	21	8.6
B	100	Complete(degraded)	8	12.5
C	80	Incomplete(stopped)	-	-
D	160	complete	18	8.9
E	120	complete	11	10.9
Commercial purge compound	110	complete	10	11

From the result above, specimen C could not be extruded completely while specimen B completed in 8 minutes but was degraded, specimens A, D, E and the commercial purge compound completed successfully in 21, 18,11 and 10 minutes respectively. Considering the time of purge and quantity used the material removal rate was calculated as in table 6 above. The highest material removal rate is shown by specimen B which degraded in the process and cannot be used as purge compound. The runner-up is the commercial purge compound followed by specimen E which was chosen as the purge mixture in this case.

Figure 19 Picture of the mixture extruded through the die [Author]

Figure 20 Picture showing color change with the tested resins [Author]

4.2 Cost effectiveness

Making an own purge mixture might be challenging due to the need for reagents and equipments, it is however cheaper to make a purge mixture as the clay and Silicon dioxide which are the basic ingredients for the purge are cheap to afford and the recycled HDPE is easily reachable too. The cost of the commercial purge compound used for the testing is 166.36 EUR per 25kg [23], while the production cost of purge mixture of the same size is about 1.29EUR considering only the cost of raw materials like Green clay and silicon dioxide to be 12.71 EUR/500g [24] and 0.44 EUR/kg [25] respectively, the recycled HDPE to be 1.12 EUR/metric ton [25]. The estimated cost of the raw materials used for making the purge mixture is summarized in the table below:

Table 8shows the cost of raw materials for the purge Mixture

S/N	Price (EUR/kg)	Quantity used(kg)	Cost/quantity(EUR/kg)
Recycled HDPE	0.0011	0.2	0.00022
Silicon dioxide	0.4400	0.001	0.00044
Green Clay	25.420	0.002	0.05084
total			**0.0515**

In addition to other material and production cost, the purge mixture is cheaper than the commercial purge compound.

4.3 Description of the purge mixture

The purge mixture is a good performing compound specially produced for material and color changes for extrusion machines. It has a wide range of processing temperature that is suitable for many plastic resins removal. It is a HDPE based mechanical purge compound produce by blending HDPE and a mixture of clay and silicon dioxide. It can be used to purge Acetal, ASA blends, Fluoropolymers, Nylon, Polybutylene terephthalate, polycarbonates, Acrylonitrile butadiene styrene, PEI, PET, polyester alloys, polyolefins, PPS, PVC and TPE as well as change color of this materials.

To use the purge, weigh appropriate quantity of the purge into a container depending on the intensity of the color and material to be purged, run purge after production at temperature ranges of 180˚C to 200˚C, at a speed of 4-6rpm for best performance until contaminant is completely purged from extruder.

Figure 21 Picture of the produced purge mixture (Authur)

Figure 22 Picture of Commercial purge compound (Author)

5 CONCLUSION AND RECOMMENDATION

The growing need for purge compounds in plastic manufacturing motivated the production of simple purge mixture that can be formulated easily and still have a near effect of commercial purge compounds. The cost of commercial purge compounds is very high when compared to the cost of producing a simple purge that can do similar work.

This thesis work used HDPE as base resin in addition to silicon dioxide and clay to produce a mechanical purge compound. The method used for the production was a continuous compounding and testing until desirable compound was produced. The purge mixture was tested for effectiveness by comparing with known commercial purge compound, it was observed from the experiment that the material removal rate of the commercial purge mixture is 11g/minute while that of the produced mixture is 10.9g/minute.

Although the purge mixture is effective in changing materials like Acetal, ASA blends, Fluoropolymers, Nylon, Polybutylene terephthalate, polycarbonates, Acrylonitrile butadiene styrene, PEI, PET, polyester alloys, polyolefins, PPS, PVC and TPE as well as color changing, other additives can be added to the purge mixture to increase its effect in removing other form of contaminants in extruders and improve usage with other resins besides those mentioned above. The suitable application temperature of the purge is 180˚C to 200˚C at a speed of 4-6rpm to cover a wide range of plastic materials.

For future work it is strongly recommended that the effect of this purge compound be checked with regards to the wear it might cause to the extruder barrel.
More so more additives can be added to the mixture and tested for purging effects as this thesis work only focused on producing a purge mixture that is effective in color and material changes only. It should be understood also that some color batches and additives react with the clay and produce hazardous effect, it is however recommended to check compatibility of chemical compounds before adding it to the mixture. Finally it will also be helpful to try adding abrasives to the mixture and examine its effect in scrubbing the extruder barrel.

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