Melt Flow Rate Study of Recycled High Density Polyethylene

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Melt Flow Rate Study of Recycled High Density Polyethylene

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Abstract-- **This research work focused on melt flow rate investigation of recycled high density polyethylene (HDPE) and virgin high density polyethylene. Melt flow rate of the developed material were tested to determine the quality of the plastics. From the experimental test results, some of the samples (scrapconcentration up to 60 wt. % and virgin concentration up to 40 wt. % by mass) are found to meet the standard requirements.**

Keywords: **HDPE scraps, MFR, MFI, Polymer, Virgin HDPE.**

I. INTRODUCTION

Materials are probably more deep seated in our culture than most of us realize. Transportation, housing, clothing, communication, recreation, and food production virtually every segment of our everyday lives is influenced to one degree or another by materials. As materials are so important in the development of human civilization; they are classified in groups by different criteria, like crystal structure, properties, or use. Based on this the main classes of present engineering materials are metals, ceramics, polymers, composites, semiconductors and biomaterials [1].

In plastic manufacturing; industries produce reasonable amount of waste during production, scrap plastics are generated from the manufacturing of plastic products, packaging and from manufactured materials. Plastic manufactures are responsible for the generation of these scraps and wastes; accordingly the amount of waste generated by plastic industrial plants becomes an increasingly costly problem for manufacturers.

Around 4% of world oil and gas production, a non-renewable resource is used as feedstock for plastics and further 3 to 4% is expended to provide energy for their manufactures. A major portion of plastic produced each year is used to make disposable items of packaging or other short lived products that are discarded within a year of manufacture [2].

The major problem faced by the polymerisation industry is that the resin characteristics that define polymer quality, such as melt flow index (MFI) and density cannot be measured on-line. Properties, such as MFI, are difficult to measure and usually unavailable in real time since it requires close human intervention [3].

Melt Flow Index

Melt flow index (MFI) is basically defined as the weight of the polymer (g) extruded in 10 min througha capillary of specific diameter and length by pressureapplied through dead weight under prescribed temperatureconditions.

Melt flow index (MFI) is a measure of the ease of flow of the melt of a thermoplastic polymer. It is defined as the mass of polymer, in grams, flowing in ten minutes through a capillary of a specific diameter and length by a pressure applied via prescribed alternative gravimetric weights for alternative prescribed temperatures.

Polymer processors usually correlate the value of MFI with the polymer grade that they have to choose for different processes, and most often this value is not accompanied by the units, because it is taken for granted to be g/10min.

Melt flow rate is an indirect measure of molecular weight, with high melt flow rate corresponding to low molecular weight. At the same time, melt flow rate is a measure of the ability of the material's meltto flow under pressure. Melt flow rate is inversely proportional to viscosity of the melt at the conditions of the test; through it should be borne in mind that the viscosity for any such material depends on the applied force.

Melt flow rate is very commonly used for polyolefin, polyethylene being measured at 190^{o} C and polypropylene at 230^{o}C. The plastics engineer should choose a material with a melt index high enough that the molten polymer can be easily formed into the article intended, but low enough that the mechanical strength of the final article will be sufficient for its use.

Mathematical mass flow rate can be calculated by the following formula as per ISO 1133.

$$MFR(\Theta, m_{nom}) = t_{ref} * m / t \quad .....(1)$$

Ɵ is the test temperature, in degrees Celsius, mnomis the nominal load in kgm is the average mass in grams of the cut offs, tref is the reference time (10min), in second (600s), t is the cut-off time interval in seconds.

[7] Have suggested a method of determining the MWD h m MFI using the values of MFI evaluated under not less than two different load conditions.

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A detailed study of a number of low-density polyethylenes in order to develop relationships between MFI, density, and molecular parameters was presented [8].There is a close relationship between solution viscosity and MFI, as has been pointed out by a number of workers [9-11]. Earlier it was shown there is an inverse relationship between MFI and zero shear viscosity [12]

II. Literature Review

Ethiopia plastic Industry was established in 1960 by a group of five entrepreneurs [13].In Ethiopia; the 1st plastic pipe industry is Ethiopia Plastic Industry PLC (EPI). It was established by five Italian private entrepreneurs in 1960 E.C. In 1973, the military government in power; at that time took ownership of 55% of the company, leaving the remaining 45% to the original owners. Consequently, in 1978 the same government nationalized the company [14], and now the company was incorporated into the Metal and Engineering Corporation (METEC) in2011.Normally, the test conditions for measurement of melt flow rate are specified in the material standard with reference to this part of ISO 1133. To conduct the tests "Plastics-Determination of the Melt Flow Rate of Thermoplastics," was followed [15].

It can be seen that seemingly small temperature gains are quite significant in terms of achieving higher polymer MFI [6]. Researcher who was first to develop and simulate a comprehensive process model of loop reactors for propylene polymerization [4]. [5] Studied the molecular weight distribution and the weight average molecular weight of polypropylene produced in a single industrial loop reactor.

III. Materials And Methodology

3.1. Materials

In this research work virgin PE100raw materials and its pipe scraps are used. PE100 indicates the designation of polyethylene materials as shown in Table 1 based on the long term strength of the respective materials, known as the minimum required strength (MRS) in accordance with ISO 12162.

Table 1:
MRS classification of materials in pipe form [16]

Material Designation	Minimum Required Strength (MRS) MPa
PE 100	10.0

3.2. Methodology

MFR test of HDPE was conducted usingISO1133 [15] granuling sample of 3 to 6 gm was taken during conducting of this test.Melt Flow Rates of Thermoplastics was conducted using ExtrusionPlastometer. The basic principle employed in the melt flow index test by any of the standards is that of determining the rate of flow of molten polymer through a closely defined extrusion plastometer shown in Figure 1.

This test covers the determination of the rate of extrusion of molten thermoplastic resins using an extrusion plastometer. After a specified preheating time, resin is extruded through a die with a specified length and orifice diameter under prescribed conditions of temperature, load, and piston position in the barrel. In which, the units of measure are grams of material/10 minutes. It is based on the measurement of the mass of material that extrudes from the die over a given period of time.

A plastometer is a tool used to determine the flow properties of plastic materials. Alternatively, a head-measuring device developed by German eugenics Robert Burger Villingen and used by the Nazis to determine alleged racial characteristics.Other Equipmentsuch asWeight balance, table vice, open mold cavity, manual furnace, lath machine, sand paper, file, micrometer, thermocouple, clamping vice and hand hack saw. All photograh were taken at BIWT during MFR testing. Extrusion plastometer equipment is shown in Figure 1 given below. During plastometer preparation Woven and plastomter is shown in Figure 2 given below.

Figure 1. Extrusion Plastometer

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Prepared both scrap and virgin HDPE materials and weighed the sample on digital weight balance according to the designed proportion i.e. 5 gram from virgin for 100% virgin MFR test, 5 gram from scrape for 100% scrap MFR test, 4 gram from scrap and 1 gram from virgin for 80% HDPE scraps with 20% virgin HDPE MFR test, 3.5 gram from scrap and 1.5 gram from virgin for 70% HDPE scraps with 30% virgin MFR test, 3 gram from scrap and 2 gram from virgin for 60% HDPE scraps with 40% virgin HDPE MFR test, 2.5 gram from scrap and 2.5 gram from virgin for 50% HDPE scraps with 50% virgin HDPE test. Proportionate sample weighting was done by using digital weighing balance as shown in Figure 3 given below. Clean the barrel of MFI apparatus by white soft woven.

Figure 2. Woven and Plastometer

Figure 3. Digital weight balance

After weighed the scrap and virgin sample were mixed and the polymer sample (5 kg) was inserted in to MFR apparatus, Photograph taken at BIWT Plc. during discharging of mixed HDPE materials as shown in Figure 4.Photograph taken at BIWT plc. During material packing in the barrel shown in Figure 5, the material was packed properly inside the barrel to avoid formation of air pockets. The sample was preheated for a specified amount of time (5 min at 190^{o}C for polyethylene). After preheating a specified weight (2.16 kg) was introduced into the piston, Photograph taken at BIWT plc. during dead weight insertion as shown in Figure 6. The weight exerts a force on the molten state polymer and it has been started to flow through a die. A sample of the melt after extruded cutoff photograph taken at BIWT plc. as shown in Figure 7 was taken after the desired period of time. The required data was recorded (mass) and MFR from digital Extrusion plastometer as Photograph taken at BIWT plc. during extruded cutoff mass reading shown in Figure 8 given below. MFR is expressed in grams of polymer per 10 minutes of duration of the test.

Figure 4. Discharge of mixed HDPE material in plastometer

Figure5. Packing of discharged material in the barrel

Figure 6. Position of dead weight in plastometer

Figure 7. Extruded cut off sample

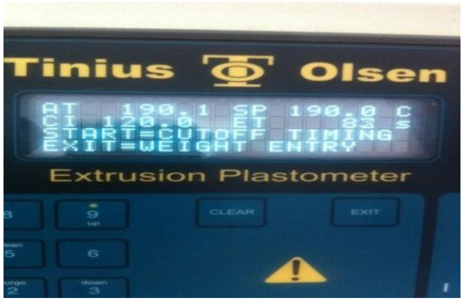

Figure 8. Extruded cutoff mass reading

3.3. Experimental Data and Analysis

3.3.1. Melt Flow Index of 100 wt. % Virgin HDPE

From 100 wt. % virgin HDPE materials plastometer extrusion test [15] we have got the following result as shown in Table 2. When it compare with other sample data, smaller amount of extruded cut off mass were recorded; because, virgin HDPE have better bond strength throughout its entire molecular structure than HDPE scraps. As it known for smaller melt flow rate; there is large molecular weight and strong bond strength

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Table 2:
Melt flow index test result for 100 wt.% virgin HDPE

No. of Test	Mass (gram)	MFR (g/10 min)	Av. MFR (g/10 min)	Overall Av. (g/10 min)
Round one				0.0599
1	0.0113	0.057	0.0568	
2	0.0119	0.060		
3	0.0110	0.055		
4	0.0115	0.058		
5	0.0114	0.054		
Round two				
1	0.0120	0.060	0.059	
2	0.0119	0.060		
3	0.0116	0.058		
4	0.0119	0.060		
5	0.0114	0.057		
Round three				
1	0.0129	0.065	0.064	
2	0.0130	0.065		
3	0.0131	0.066		
4	0.0127	0.064		
5	0.0120	0.060		

Graphically the average value of each round test and overall average value of the entire process are represented as in Figure 9 given below.

In which; as operation time is long, the average value of the test result is increased from left to right. As a result, when operation time is longer a material tends to melt more. That's why mass flow rate is increased from left to right.

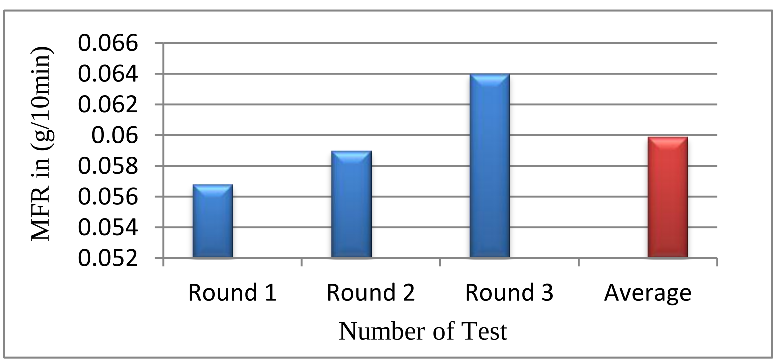

Figure 9.MFR-Number of Test for 100 wt.% virgin HDPE

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From the above graph it can be understood that mass flow rate of 100 wt.% virgin materials are fulfill the standard value as stated in appendix[17]. In which, when it camper with other test sample it mass flow rate is very small; because, virgin materials have strong bond strength than scrap materials.

3.3.2.Melt Flow Index of 50 wt.% HDPE Scraps with 50 wt.% virgin HDPE

From 50 wt.% scraps with 50 wt.% virgin PE100 materials plastometer extrusion test, fifteen cut off mass has been taken as the rest of the experiment that were conducted before.

In this experimentation, amount of cut-off mass are much smaller than 100 wt.% scraps extrusion test result. This is due to the present of much amount of virgin material in the sample. As it has been seen before, when amount of virgin materials increases bond strength also increases; as a result extruded cut off mass were decreased. Melt flow index test result for 50 wt.% HDPE scrap with 50 wt.% virgin HDPE is shown in Table 3 given below.

Table 3:
Melt flow index test result for 50 wt.% HDPE scrap with 50 wt.% virgin HDPE

No. of test	Mass (gram)	MFR (g/10 min)	Av MFR (g/10 min)	Overall Av (g/10 min)
Round one				
1	0.0118	0.059		
2	0.0114	0.057		
3	0.0116	0.058	0.0578	
4	0.0114	0.057		
5	0.0115	0.058		
Round two				
1	0.0121	0.061		0.060
2	0.0119	0.060		
3	0.0118	0.059	0.0594	
4	0.0117	0.059		
5	0.0116	0.058		
Round three				
1	0.0122	0.061		
2	0.0126	0.063		
3	0.0128	0.064	0.0628	
4	0.0125	0.063		
5	0.0126	0.063		

Graphically, the average value of mass flow rate for each rounds and overall average of the entire process are represented as follows.

Here below the graph in Figure 10 shows mass flow rate increases from left to right as span of operation time is long.

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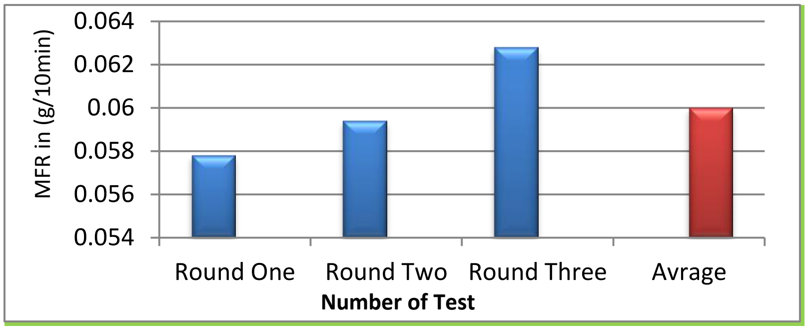

Figure 10. MFR-Number of Test for 50 wt.% HDPE scraps with 50 wt.% virgin HDPE

From Fig. 10, it can be understood that MFR are increased from left to right trough out the entire process. But, when compare it with other experimentation that are conducted with smaller virgin material concentration than this, mass flow rates are decreased. This indicates that when virgin material concentration increases in scrap its melt index becomes decreased; this due to the presence of virgin material in scraps that it increases its bond strength.

3.3.3. Melt Flow Index of 60 wt.% HDPE Scraps with 40 wt.% Virgin HDPE

Within this proportion fifteen sample of extruded cut-off mass were also taken as shown in Table 4 below. In which, as amount of virgin materials are increased cut-off mass are decreased due to extra bonding strength between carbon and hydrogen molecules. This is true for comparing it with smaller virgin material concentration experimental data. But when we see the entire process the amount of cut-off mass are increased from beginning to end.

Table 4:
Melt flow index test result for 60 wt.% HDPE scrap with 40 wt.% virgin HDPE

No. of test	Mass (gram)	MFR (g/10 min)	Av. MFR (g/10 min)	Overall Av. (g/10 min)
Round one				0.0611
1	0.0117	0.059	0.0588	
2	0.0116	0.058		
3	0.0119	0.060		
4	0.0116	0.058		
5	0.0117	0.059		
Round two				
1	0.0122	0.061	0.0608	
2	0.0121	0.061		
3	0.0118	0.059		
4	0.0123	0.062		
5	0.0122	0.061		
Round three				
1	0.0137	0.069	0.0638	
2	0.0130	0.065		
3	0.0120	0.060		
4	0.0124	0.062		
5	0.0126	0.063		

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Each round of average test result can be represented graphically as follow. From this value it can understood that the test result is within standard value.

Hence, the increment of cut-off mass in the entire process is due to the effect of heat energy during span of operation time.

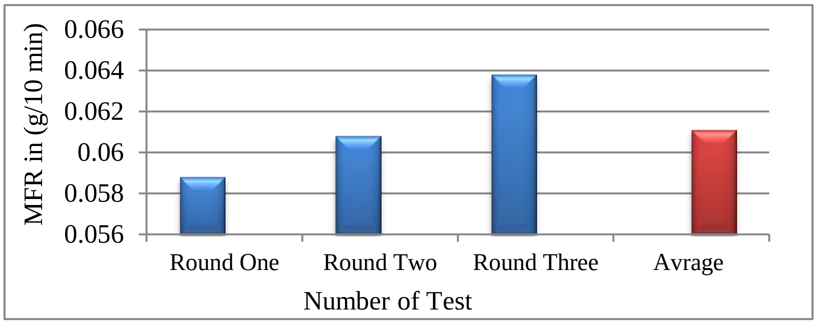

Figure 11. MFR-Number of Test for 60 wt.% HDPE scraps with 40 wt.% virgin HDPE

From the above graph Figure 11, it can be understood that, when virgin HDPE concentrations are increased, its mass flow rate decreased. This shows that as virgin material concentration increases in scraps, bond strength also increases. Consequently, as bond strength increase, much heat energy is required to break the bond. That's why, at the same operation and duration different data are recorded as scrap concentration decrease and virgin concentration increases.

3.3.4..Melt Flow Index of 70 wt.% HDPE Scraps with 30 wt.% virgin HDPE

By this proportion fifteen cut-off mass with three rounds test were taken as shown in Table 5 below. From this experimental data it can be understood that as amount of scraps increases mass flow rate decreases; or as amount of virgin material increase, bond strength of material also increase; as a result melt flow rate decrease.

Table 5:
Melt flow index test result for 70 wt.% HDPE scrap with 30 wt.% virgin HDPE

No. of test	Mass (gram)	MFR (g/10 min)	Av. MFR (g/10 min)	Overall Av. (g/10 min)
Round one				0.0615
1	0.0120	0.060	0.0604	
2	0.0120	0.060		
3	0.0123	0.062		
4	0.0121	0.061		
5	0.0117	0.059		
Round two				
1	0.0125	0.063	0.0612	
2	0.0121	0.061		
3	0.0122	0.061		
4	0.0118	0.059		
5	0.0123	0.062		
Round three				
1	0.0127	0.064	0.063	
2	0.0125	0.063		
3	0.0127	0.064		
4	0.0124	0.062		
5	0.0124	0.062		

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Hence, amount of mass is decrease with relative to large amount of virgin materials. This is due to the need to much heat energy to break bond strength between molecules.

Amount of MFR (g/10 min) in entire process beginning to end is increasing. Graphically the average value of each round and overall average value are represented as follows in Figure 12 given below.

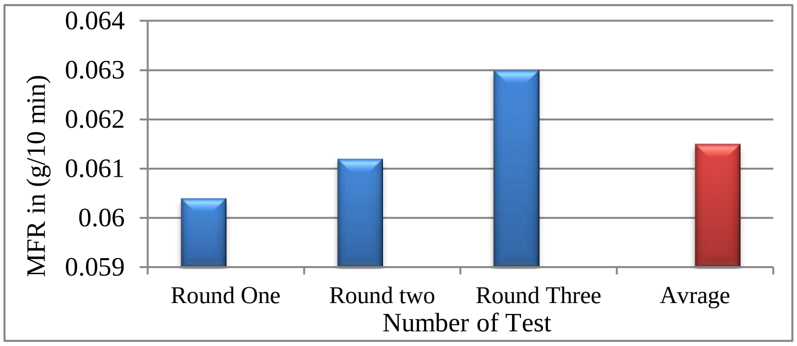

Figure 12. MFR-Number of Test for 70 wt.% HDPE scraps with 30 wt.% virgin HDPE

From the above graph it can be understood that mass flow rate increases throughout the entire process but it decreases as compared to large amount scrap concentration sample. This is due to the absence of strong bond between molecules.

3. 3.5.Melt Flow Index of 80 wt.% HDPE scraps with 20 wt.% virgin HDPE

In this experimentation fifteen number of cut-off mass and mass flow rate were taken with three divisions as shown in Table 6. From this experimental data it can be understand that mass flow rate is increased; as span of time increased. But it decreased when it compare with 100 wt.% scraps test data. This is due to the present of virgin material in scraps i.e. the present of virgin material in scraps; increase bond strength between hydrogen and carbon molecule.

Table 6:
Melt flow index test result for 80 wt.% HDPE scraps with 20 wt.% Virgin HDPE

No. of test	Mass (gram)	MFR (g/10 min)	Av. MFR (g/10 min)	Overall Av. (g/10 min)
Round one				0.0628
1	0.0120	0.060	0.0588	
2	0.0119	0.060		
3	0.0118	0.059		
4	0.0116	0.058		
5	0.0114	0.057		
Round two				
1	0.0127	0.064	0.0624	
2	0.0129	0.065		
3	0.0123	0.062		
4	0.0120	0.060		
5	0.0122	0.061		
Round three				
1	0.0135	0.068	0.0672	
2	0.0133	0.067		
3	0.0137	0.069		
4	0.0132	0.066		
5	0.0131	0.066		

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As shown in this data table mass flow rates are increased from first round to last. These indicate that materials are more melted as operation time is length wise.

These incremental values are represented as follows in Figure13 given below. In this graph, the overall average value also has been shown.

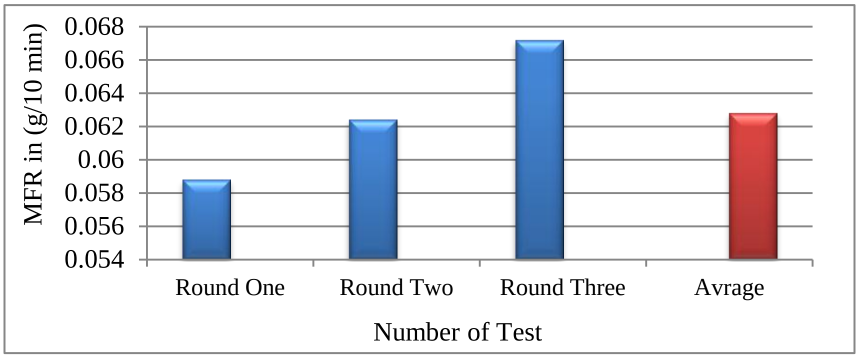

Figure 13. MFR-Number of Test for 80 wt.% HDPE scraps with 20 wt.% virgin HDPE

From this graph it can be understood that amount of mass flow rate is lower than 100% scraps. This indicates that when virgin material is available in scraps it increases its bond strength. That's why lower mass flow rate is gained from 80 wt.% scraps with 20 wt.% virgin material concentration.

3.3.6. Melt Flow Index of 100% HDPE Scraps

In this experiment three round of experimentation were conducted based on ISO 1133. In each experiment mass of extruded cut off and mass flow rate in gram per 10 minutes are recorded as shown in Table 7.

From this experimental result it can be understood that; as span of operation time is increased mass flow rate also increased to some extent and all of the test results meets standard value.

Table 7:
Melt flow index test result for 100 wt.% HDPE scraps

No. of test	Mass (gram)	MFR (g/10 min)	Av. MFR (g/10 min)	Overall Av. (g/10 min)
Round one				
1	0.0119	0.060		
2	0.0117	0.059		
3	0.0122	0.061	0.0602	
4	0.0119	0.060		
5	0.0122	0.061		
Round two				
1	0.0122	0.061		
2	0.0130	0.065		0.06293
3	0.0125	0.063	0.0628	
4	0.0125	0.063		
5	0.0123	0.062		
Round three				
1	0.0128	0.064		
2	0.0135	0.068		
3	0.0133	0.067	0.0658	
4	0.0128	0.064		
5	0.0131	0.066		

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From above data table we can correlate the average value of each round test and overall average value of the experimentation on the following graph.

The graphical representation of MFR for each round test results of 100 wt.% scraps is shown in Figure 14.

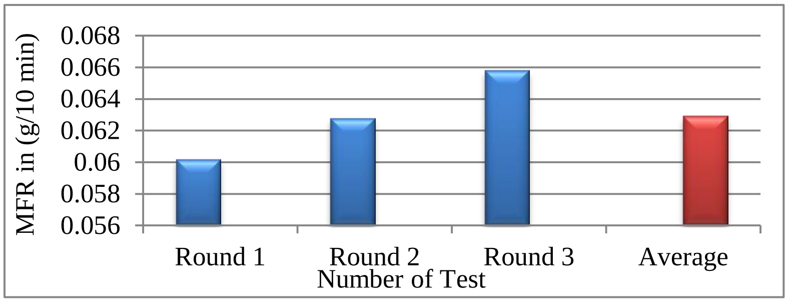

Figure 14. MFR-Number of Test for 100 wt.% HDPE scraps

From the above graph it can be understood that all round of tests are fulfilled the required standard value as shown in appendix A. In addition to this, mass flow rate trend increases from first round test up to the end. This indicates that when operation time increases in melting process, heat energy distribution through the entire process became equilibrium and melt flow rate also be increased.

IV. Results And Discussion

Melt flow index test experimentations were conducted in three rounds for each selected sample with 5 cut off extruded mass for each round. Generally, 15 cut of extruded mass were taken for one selected sample and MFR are calculated according to recorded result.In this experiment all selected sample fulfill recommended standard test value. Generally, overall average of test results are summarize in the following Table 8.

Table 8:
Overall average value of melt flow index for each sample

S/N	Selected/designed sample	MFR in (g/10 min)	Recommended standard value
1	100% Virgin HDPE	0.0599	MFR < 0.15 g/10 min
2	50% HDPE Scraps with 50% Virgin HDPE	0.06	
3	60% HDPE Scraps with 40% Virgin HDPE	0.0611	
4	70% HDPE Scrapswith30% Virgin HDPE	0.0615	
5	80% HDPE Scraps with 20% Virgin HDPE	0.0628	
6	100% HDPE Scraps	0.06293	

In melt flow index test, all selected sample fulfills minimum standard value. But the values are varying as proportion varies. This indicates that as scraps concentration increases mass flow rate also increases. As it is observed from Table 8, 100 wt.%virgin HDPE has lowest MFR (0.0599 g/10 min) in comparison to 100 wt.% HDPE scraps (0.06293g/10 min). MFR-Number of Test for 100 wt.% HDPE scraps and 100 % virgin with average of round one, two and three there is a small high in scraps in comparison to virgin.

It also shows that increasing in scraps percentage there is an increase in MFR. It indicates that the increasing percentage of the scraps melt flow rate is also increases. This can be understood that melt fluidity is higher in 100 wt. % scraps HDPE and reason behind this is due to ease bond break energy over them.

From the result it can be understood that upto 60 % HDPE scraps can be used for applications. Scrap concentration up to 60 wt. % and virgin concentration up to 40 wt. % by mass are found to meet the standard requirements.

V. Conclusion And Recommendation

Experimental characterization studies melt flow index were conducted and finding results were discussed. On the basis of finding the following conclusion can be extracted as:

In melt flow rate, all results fulfill the minimum standard requirement followed by ISO 1133 test method. Melt fluidity is slight higher in 100 wt. % scraps HDPE. On the basis of the result it can be understood that upto 60 % HDPE scraps can be used for applications. In general, from the experimental finding; it can be concluded that as scraps concentration increases MFI are slightly increased.

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APPENDIX

A. Mechanical properties of compressive molded HDPE [17]

Property		Value	Unit	Test Method	Test Specimen
Density at 23ºC		0.958	g/cm^3	ISO 1183	10mm x 10mm x 4mm
Viscosity Number		380	ml/g	ISO 1628-3	0.1% solution of granules in decahydronaphthalene
Melt Flow Rate	MFR 190/5	0.23	g/10min	ISO 1133	granules sample weight 3g to 6g
	MFR 190/2.16	<0.15	g/10min		
Tensile Properties	Yield Stress	23	N/mm^2	ISO 527, Test Rate 5 mm/min	ISO 3167, 4mm thick (test specimen no. 3, 4mm thick according to DIN 53 455
	Enlonggation at Yield Stress	10	%	ISO 527, Test Rate 5 mm/min	
	Tensile modulus of Elasticity (secant between 0.05 & 0.25% strain)	900	N/mm^2	ISO 527	
	Tensile Creep Modulus (1 hour value)	650	N/mm^2	ISO 899, Test Load 2M/mm^2	
	Tensile Creep Modulus (1000 hour value)	350	N/mm^2		
Flexural Properties	Flexural Creep Modulus (1 min value)	1100	N/mm^2	DIN 54852-Z4 σb=2N/mm^2	110mm x 10mm x 4mm loaded flat
	Flexural Stress (3.5%deflection)	20	N/mm^2	ISO 178, Test Rate 2mm/min	80mm x 10mm x 4mm
Stiffness in Torsion		180	N/mm^2	DIN 53447	60mm x 6.35mm x 3mm
Hardness	Ball Indentation Hardness	41	N/mm^2	ISO 2039 part 1 Test Load 132N	4mm sheet
	Shore Hardness D (3 sec value)	61	~	ISO 868	6mm sheet
	Shore Hardness D (15 sec value)	59	~		
Nothed Impact Strength acN (test specimen from compression moulded sheet)	at 23ºC	20	kJ/m^2	ISO 179/1eA	80mm x 10mm x 4mm
	at -30ºC	10	kJ/m^2		