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Study the Rheological Behaviour of PP- Runner Waste Blends towards using for Medical Syringes Production

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ABSTRACT

This research deals with study of Rheological behavior of polypropylene (PP) blended with different percentages of waste pre-injection (runner) to achieve the same characteristics required for the production of medical syringes (cylinder part). Injection machine used to produce this part operates at melt flow rate (MFR) between (22-28) g/10 min in syringes factory of Babylon city. PP provided from(HYSONG)Korean company with grad resin J801. X.R.D test is used to measure the level of crystalline and contamination in PP.10 %,20%,30%,40% and 50% of the runner mixed with PP to measure the (MFR), swelling ratio(diameter of extrudate/diameter of die), and density of each blend at three different temperatures 233 °C,230°Cand 227 °C, three different loads 2.16kg,3.80kg and 1.965 kg. and two size of runner pellet (large and small). The Instrument (ISO 1133:2005) is used to measure MFR, liquid density mode of GP-120S device used to measure the density. The XRD test shows that the blends and runner have higher contaminations and lower crystallization level than that in virgin PP. The results of first size showed that the MFR values increase and density decreases with the runner percentage, temperature and load increasing. The swelling ratio increases with the runner percentage and load increasing while decrease with the temperature increasing .For the second size the MFR , density, and swelling ratio behave in the same manner of the first size ,but with lower quantities due to the reduction in pellet size. This increases the flexibility to adding more runner to the blend and keeping the MFR within the acceptable range.

Keywords: PP melt, MFR, rheological behavior, wastes and recycling, medical syringe, injection molding

I. INTRODUCTION

The polymer products have a wide range of applications ,various polymer processing are used to produce these products in different industry fields ,such as extrusion ,injection and blowing. Thermoplastics are usually processed in the molten state. Molten polymers have very high viscosity values and present shear thinning behavior, as the rate of shearing increases, the viscosity decreases, due to alignments and disentanglements of the long molecular chains. The viscosity also decreases with increasing temperature. In addition to the viscous behavior, molten polymers exhibit elasticity. These include stress relaxation, normal stress differences and swelling. Molten polymer wastes at the injection machines enhance an economic loss and environmental problems. Different types of wastes associated with the process during the production These wastes occur at the pre- injection (Runner), during the injection (Flushing) and post-injection (disposal).and reuse it produces an economic benefit and minimize harm to the environment. The polymer melts exposure to different mechanical stresses due to the shear and extensional flow during process .The deformation occurs in the waste depended on the type and stage of the polymer process .The degradation and contamination of the wastes are the most important factors which are controlling on the recycling process. Rheological and physical properties are used as indication ,whether the recycling process in the right direction. the ease of melt flow depends upon the mobility of PP molecular chains and forces of entanglements holding the molecules together. As the free volume increases with temperature, PP molecules occupy more space due to an increase in the distance between them making it easier to slide among each other and together with the introduction of high shear rate will eventually reduce the PP matrix viscosity significantly[1].

Injection molding is a manufacturing process that solid thermoplastic material is heated and mixed in a barrel until it reaches a state of fluidity, then forced under pressure into a closed hollow space, and then cooled and hardened in the cavity until it reaches to the configuration of the cavity[2,3,4].The mould typically consists of two half. Usually one mould half contains the cavity and forms the outer shape of the part. This part of the mould is called the cavity side(fixed part). The fixed side means that the parts are fixed to the nozzle side of the molding machine. The other mould half contains a protruding shape and forms the inner shape of the part, this mould part is called the core(movable part)[5,6]. The flow of polymers is a complex process, as the viscosity of plastics, apart from temperature and pressure, depends on the molecular mass, molecular mass distribution and on the deformation rate. These make plastic processing rather difficult. During the flow in nozzle, runner system and in thin walled cavities of injection moulds, both shear and elongation deformation processes are developed, and the deformation rates are generally high [7]. Slow stress relaxation is responsible for frozen in stresses in injection molded and extruded products[8]. The best way to limit the plastic waste and to prevent rubbish from going to landfill is to reduce, reuse and recycle these materials [9]. Recycling of waste plastics should be carried in such a manner to minimize the pollution during the process and to enhance the efficiency of the process and conserve the energy[10]. In 2003, J. Aurrekoetxeaa ,et.al, reported that the effect of injection molding induced morphology on the fracture behavior of virgin and recycled polypropylene (PP). the micro hardness and the degree of crystalline are studied, virgin PP has shown higher micro hardness values and bigger plastically deformed zone at the crack tip than recycled one. These two differences are due to the higher crystalline of the recycled PP[11]. So, in 2011, S. FERRÁNDIZ, et.al, shows the study of rheological behavior of a material that recycled materials have been incorporated. The transformation process to be applied is the injection molding. The mix of materials used was of the same nature and have similar viscosities. This mixture provides virgin polypropylene and copolymer polypropylene waste materials. Then there has been a characterization of the mechanical and rheological characterization of the mixture. The characterization of the recycled polypropylene used in the blends shows us that no thermal degradation occurs during processing and whose behavior is very similar to that of virgin material used in the same application. The material could be considered like good quality [12].also 2005, N. Rust stated a degradation study of isotactic virgin and recycled polypropylene used in lead acid battery casings. A common technique used for the analysis of PP, MFI was shown to be an effective technique to maintain a good quality control system .The MFI and rheological studies of recycled polypropylene that showed a significant improvement in terms of maintaining long term polymer stability[13]. But in 2013, Ferenc Ronkay, analyzing the polycarbonate scrap arising during production and its possible secondary utilization. The analysis of morphological, rheological and thermal data revealed significant differences between the original pellets and the reground material obtained from injection molded parts. Test specimens were injection molded from various mixtures of the virgin and the reground material, and their mechanical and physical properties were analyzed. the average molecular mass of polycarbonate decreases by about 8% during the first injection molding and the subsequent grinding. Based on our test results, this 8% decrease in the average molecular mass causes about a 25% increase in the melt flow rate. In the optical studies, where the absorbance of the recycled material is higher than that of the virgin material. Based on these results, it can be concluded that the admixture of more than 20% reground recycled may deteriorate the mechanical and optical properties of the product significantly [14]. at 2005, M.M.K. Khan, et.al, shows the process of recycling mixtures of ABS and PC, that blends of virgin ABS and virgin PC at five different compositions, namely, 15%, 30%, 50%, 70% and 85% by weight of ABS

were prepared and characterized by rheological and mechanical measurements. Rheological properties of these blends in steady, oscillatory and mechanical properties, namely, tensile strength, elongation-at-break and Izod impact strength are reported. The results of this study show that the processability of PC can be improved by the addition of ABS. The shear thinning effect of ABS rich blends provides a significant power saving in the processing of these blends. Furthermore, except for the 15% blend, the viscosities of other blends fall into a narrow band which offers a wide range of operation window and room to mask possible variations in properties of recycled polymers. The results of the tensile and impact tests also indicate an improvement in the processability of PC by adding ABS. The tensile yield strength results for the blends follow the'rule of mixtures' showing a decreasing value with the increase of ABS content[15].

This work study the rheological behavior of virgin PP blend with different ratio of runner waste produced in injection machine during manufacturing the cylinder part of medical syringe .The suitable MFR setting in injection machine is between 22-28 g/10 min .MFR ,density and swelling ratio of the blends are tested under different loads and temperatures and runner pellet size to check which blend produces MFR within the range of MFR in the injection machine . Melt index type(ISO 1133:2005) is used to measure MFR. XRD test is used to check the contaminations and crystallization levels.

II. MATERIALS AND METHOD

Medical Syringes: The medical syringe consists of four parts (piston ,cylinder, Allowacher and protective) as illustrated in Fig.1 .Each part manufactured using different injection machine with different boundary conditions. This work concentrated on the cylinder part which produce in injection machine type Jung Won JMI/SPI-150. The parameter and characteristic of the cylinder part injection machine are indicated in Table(1)



Fig.(1): Medical syringe,(a) assembly of syringe, (b) Cylinder (barrel).

Name of mold	10cc- Barrel	Mold size	450*500*436
Number of cavity	1*24	Gate type	SIDE
Used Resin	Homo polymer propylene PP	Construction of mold	3 plate
Mold clamping force(bar)	140	Injection time1 (sec)	2.4
Injection pressure 1(kg/mm ²)	84	Injection time1 (sec)	0.2
Injection pressure2(bar)	83	Cycle Time (sec)	20
Cooling time (sec)	10	Cooling water temperature (°C)	22
Nozzle temperature(°C)	255	MFR g/10 min	22-28

Table (1): Characteristic and Parameter of Cylinder **Part Injection Machine**

Runner Waste and Injection Machine

The runner wastes produce from the machine during injection process, runner waste is the pre-injection waste and is higher quantity than other waste with a certain degradation depending on the flow rate, temperature and geometry. Fig. (2 a, b, and c) shows the injection machine used to produce the cylinder part, injection mold and the runner waste.



Fig.(2): (a) injection machine JMI/SPI-150 used to produce cylinder part (b) mold of cylinder part (c) Runner waste

Polypropylene (PP): The PP used in the manufacturing of cylinder part is provide from HYSONG Korean company .The physical and mechanical properties of PP are illustrated in Table(2)

Table (2): '	The physical	and	mechanical	properties of
	J	801	PP	

	Mechanical	Metric	English	Comment
				s
	Hardness,	100	100	ASTM
	Rockwell R			D785
	Tensile	40.2	5830 psi	ASTM
	Strength, Yield	MPa	_	D638
Properties	Elongation at	200%	200%	ASTM
	Break			D638
	Flexural	1.81 GPa	263 ksi	ASTM
	Modulus			D790
	Izod	0.343	0.643 ft-	ASTM
	Impact,Unnotc	J/cm	Ib/in	D256
	hed			
	Thermal			
	Deflection	125°C	257° F	ASTM
	Temperature at			D648
	0.46 MPa (66			
	psi)			
	Softening	150°C	302° F	ASTM
	Point			D1525
	Melting point	160-	266-	
		176°C	340 °F	
	Physical			
	Melt Flow	25.0 g/10	25.0	ASTM
		min	g/10min	D1238
	Density	902-906	Pellet	4 mm
		kg/m3	size PP	

Runner Pellet Preparation: The runner waste cutting to a small pellets or grain in order to mix with the virgin PP grain .cutting machine is used for that purpose ,sieve type (5 mm,4,75 ,2.36mm) is used to characterize the pellet size two size of pellet are distinguishes the first size contains all (2.36 mm, 4.75 mm and 5 mm)and the second sizes contains (2.36 mm and 4.75 mm.

XRD Test: XRD instrument type is used to measure the level of crystalline and contamination of each blends and runner waste and to check the PP type.

MFR Test: Melt indexer type International Standard (ISO 1133:2005) is used to measure the melt flow rate(MFR)of PP and PP blends. MFR of PP blends through capillary die (Dc) is 2.095mm, the ratio of the die length (L) to capillary die (Dc)is (8/2.095). Fig.(3), indicates the schematic of melt indexer.

The procedure of the test:(1)- blend the virgin PP pellet with the (10%,20%,30%,40% and 50%) of first size of the runner waste. The MFR at load 2.16 kg and three temperatures (227 °C ,230°C and ,233° C) and at temperature 230°C and three loads (1.965 kg, 2.16 kg and 3.80 kg) are measured based on the forming formula:-MFR=t ref *w/t

t ref=10 min /600s





(2) - Blend the PP pellets with the 10 %,20%30%40% and 50% of second size of runner waste and measure the MFR ,under the same boundary conditions mentioned in (1).

Calculation of shear rate: The shear rate at the wall of capillary die of melt indexer device is obtained depending on the MFR results using the following formula:

 $v \cdot w = (1840/\rho) * MFR$ (2)

 $\mathbf{y} \cdot \mathbf{w} =$ shear rate at the wall (s⁻¹)

Density Kg /m³ ρ =

Swelling ratio:- Swelling ratio or extrudate die indicated the polymer elastic behavior and directly related to MFR, shear rate and normal stress. The swelling ratio of each blend obtained using the flowing equation :-

Swelling ratio= De/ Dc

De= diameter of extrudate melt (mm)

Dc= diameter of capillary (mm)

Density test: liquid density mode of GP-120S device used to measure the density of extradate polymer for each blend according to following formula : -

density = weight of the polymer in air – weight of the polymer in water

III. RESULTS AND DISCUSSION

XRD results show crystalline and contamination of PP and PP blends. Melt indexer results show the effect of pellets size, temperatures and loads on the MFR and shear rate at the wall, which is inversely changes with the molecular weight, viscosity, and density. In the present work density test used to verify the MFR test.Swelling ratio is used to check the elastic behavior of polypropylene blends at two different pellet size (first and second size).

(A): First size of runner pellets

XRD Results: Fig.4 shows that the crystalline ratio decrease in descending manner from (a) pp to (e) 30 % runner due to the molecular weight reduction. The chain of molten polymer is broken due to the shear and extensional flow. Therefore, length of the chain and molecular weight are decreasing after each melting polymer flowing process. PP and runner are exposure to one and two melting process respectively, which is explain the difference in the test between them. The 10% ,20%, and 30% runner indicate lower 2 Θ than pp and pure runner due to the difference in molecular weight and chain length between pp and runner. Also a small quantity of contamination are observed in this figure. The contamination are increased from (a) to (d) due to the number of melting process.



Fig.(4): XRD Test Give an Indication About Crystalline and Contamination Level for (a) PP, (b) Runner, (c) 10% Runner, (d) 20% Runner, and (e) 30 % Runner Melt flow rate effect: Polypropylene is classified as viscoelastic material and exhibits a non Newtonian flow behavior during injection and extrusion processes .The rheological behavior of PP melt depended on the microstructure, physical chemical properties ,flow behavior ,deformation and rate ,boundary condition and die or mold geometry . Melt flow index test of polymer used to check and connect between shear rat ,shear stress ,pressure ,viscosity and die geometry . Fig.(5) shows that the melt flow rate (MFR) increases with the runner percentage and temperature increasing at load 2.16 Kg .The MFR increases due to the runner percentage increasing is more than that for temperature increasing .The runner percentage is already subjected to a certain degradation due to the mechanical load associated with shear flow of PP melt .This degradation directly reduce the molecular weight and chain length .The viscosity of polymer melt strongly related to the chain length . Mixing of runner PP with short chain length to the virgin PP of higher chain length produce less average molecular weight and reducing viscosity so, the MFR and shear rate increase with viscosity decreasing. Viscosity of PP melt decrease with temperature increasing due to the melt volume increase, large volume provide more space in PP melt and easy chains movement .The MFR exhibits approximately stable at high runner percentage after 40% runner.

Fig .(6) shows that the MFR increase with the runner percentage and the load increasing at 230 °C .Also the increases in MFR due to the load increases is more than that for runner percentage increasing .The load directly related to MFR, shear rate, pressure, shear stress and viscosity. The MFR and shear rate inversely change with the viscosity and that may be expressed why the load is more effects than that runner percentage .Also the effect of load change from 2.16 to 3.80 kg is larger than that from 1.965 to 2.16 due to high shear rate producing. The MFR of 100% runner in Figs. 1 and 2 at different temperatures and loads is approximately close to pp or 10% runner, this is due to the nature of chains length behavior .For each pp and 100% runner the chains length is convergent which my be describe as narrow molecular weight distribution NMWD, while the pp blends exhibits the broad molecular weight distribution.







Fig. (6) : MFR behavior with Runner percentage increasing for different load of the first size at 230

Table (3): Wall Shear Rate at different Temperatures and at load 2.16 kg

Dunnan	Shear well($a = 1$) at 2.16 leg			
Runner	Shear Wall(s 1) at 2.16 kg			
%	and three different temperatures for			
	first size			
	227 °C 230 °C 233 °C			
0	39.25	48.52	50.88	
10	49.46	50.86	50.99	
20	51.94	53.74	54.48	
30	53.53	56.45	57.19	
40	57.78	60.58	60.90	
50	58.77	60.80	61.16	
100	49.66	52.16	59.22	

Table (4): wall shear rate at different loads and at temperature 230 C

Runner	Shear wall (s ⁻¹) at 230°c			
%	and three different loads for first size			
	1.965kg	2.16kg	3.80kg	
0	42.54	48.52	95.53	
10	42.69	50.86	104.81	
20	46.68	53.74	107.78	
30	47.61	56.45	110.08	
40	49.84	60.58	110.47	
50	55.46	60.80	112.38	
100	48.29	52.16	98.23	

Shear Rate Effect: The shear rate at the wall of the capillary proportional to the MFR values. The shear flow through the capillary die is study and fully developed. The maximum velocity and shear rate occur at the center of the die and die wall respectively. The pressure gradient across the capillary die is affected by shear flow rate and geometry of the die. Also the

critical shear stress at the wall depends on the pressure gradient and die geometry. The final polymer product quality and surface defects related to the critical shear stress. **Table (3 and 4)**, indicate the capillary wall shear rate at different temperatures and loads according to equation 2. The wall shear rate increases with the temperature and load increasing.

Swelling effect :Fig. (7) shows that the swelling ratio increase with the runner percentage increases and temperature decreasing at load 2.16 Kg. The swelling ratio increases due to the runner percentage increasing is more than that for temperature decreasing .The high MFR and shear rate produce high normal stress and swelling .Viscosity of PP melt decreases with temperature increasing due to the melt volume increase, large volume provide more space in PP melt and easy chains movement. This reduces the viscosity and swelling. The swelling ratio increases with the load increasing ,it is directly affected by elastic flow and relaxation time .Normal stress and swelling ratio are important elastic phenomena .The load increasing produces shear rate and shear stress increasing . The swelling ratio increases with the shear rate, and normal stress increases. Fig.(8) shows that the swelling ratio increase with the load and runner percentage increasing. The shear rate increases with MFR increasing and the normal stress increases with the shear rate increase. The swelling ratio directly increases with normal stress increase. The MFR increases with the runner percentage and load increasing .The swelling ratio exhibits linear change up to the 30 % runner percentage and then increases with nonlinear manner due to the reduction in viscosity and normal stress increases.

At 100% runner under different temperatures and loads the swelling ratio is lower than that for 50% runner blend due to the lower MFR values.







Fig. (8) : swelling ratio behavior with runner percentage increasing for different loads of the first size at 230 °C

Density effect: Fig. (9) : illustrates that the density decreases with the runner percentage and temperatures increasing up to the 50% runner then increasing to a small value at 100% runner . The density is proportion to the molecular weight ,viscosity and chain length ,which are reduced with the MFR and shear rate increasing .The density reduction at temperature 233 °C and 230 °C is more than that at 227 °C due to the high MFR at high temperatures .The density enhances rapid drop with the runner percentage increasing up to 30 % and keep stable after 40% due to the stable MFR under different temperatures at Fig.(5) . Fig. (10) shows the decreasing of density with runner percentage and load increasing. The change in density drops rapidly at load 3.80 Kg and 2.16 Kg for runner percentage up to 30%, then enhance more stable for 2.16 Kg and keeping dropping for 3.80 Kg due to the high MFR and shear rate .At load 1.965 Kg the density decreasing gradually due to the small change in MFR in Fig. (6).



Fig. (9) :density behavior with runner percentage increasing for different temperatures of the first size at load 2.16 kg



Fig. (10): Density behavior with runner percentage increasing for different loads of the first size at 230 $^\circ C$

(B) Second Size of Runner Pellets

Melt flow rate effect: Fig. (11) shows that the MFR increase with runner percentage and temperature increasing up to 50% runner and then keep constant at 233 C, while decreases to the value corresponding to 30% runner blend at 230 C and 227 C, due to the shear flow stresses and thermal degradation which leads to molecular weight reduction .The effect of temperature on MFR is bigger than that of runner percentage. Number of reextrusion increases MFR and decreases the average molecular weight (Mw), this mechanism produces shorter chain length with easy movement and low resistance to flow, which means low viscosity and high both shear rate and MFR. The temperature effect on MFR is bigger than that the runner percentage compared with the Fig. (5), may be due to the small size runner waste blend with virgin PP .The convergent in size between the small size runner waste and the PP virgin grain may be produced higher viscosity and lower MFR .The results of MFR produced with the second size (small size) is lower than that with the first size (large size). The MFR keeps lower and approximately more stable in the second size with the runner percentage increasing Fig. (12) shows that the MFR increases with load and runner percentage increasing up to 50% runner and then keep constant at 3.8 Kg, while decreases to the value corresponding to 30% runner blend at 1.965 Kg and 2.16 .Shear thinning flow produced with the MFR Kg increasing .Viscosity reduced and the chain exhibit more alignment. The molecular reduction and chains broken in the runner waste decrease the viscosity of the blend .The MFR values are lower with load increasing and higher with runner percentage increasing compared with the Fig. (6) because of the pellet volume reduction.



Figure (11): MFR behavior with the runner percentage increasing for different temperatures of second size at load 2.16 kg



Fig. (12) : MFR behavior with the runner percentage increasing for different loads of second size at 230 °C

Table (5): Wall Shear Rate at different Temperaturesand at Load 2.16 kg

Runner %	Shear wall(s ⁻ 1) at 2.16 kg and three different temperatures for second size			
	227 °C	230 °C	233 °C	
0	39.25	48.76	50.88	
10	46.55	50.28	55.14	
20	50.45	51.95	58.33	
30	52.87	57.42	59.50	
40	55.70	59.89	59.89	
50	58.22	62.67	62.67	
100	50.98	59.62	59.62	

Shear Rat Effect: Tables (5 and 6), indicate the capillary wall shear rate at different temperatures and loads according to equation 2. The wall shear rate increases with the temperature

and load increasing. The shear rate increases due to the loads increasing is higher than that due to the temperatures increasing.

Table (6): Wall Shear Rate at Different Loads and at Temperature 230 C

Runner %	Shear wall (s ⁻ 1) at 230°c and three different loads for second size			
	1.965kg 2.16kg 3.80kg			
0	42.64	48.76	96.41	
10	44.19	50.28	97.83	
20	47.24	51.95	99.34	
30	49.81	57.42	107.18	
40	53.61	59.89	109.47	
50	57.34	62.67	110.60	
100	49.16	59.62	106.72	

Swelling effect: Polymer is a viscoelastic materials, normal stress and swelling ratio are an important elastic flow phenomena. The extrudate swell increasing with normal stress increasing. Normal stress is strongly proportional to the shear flow and MFR and temperature decreasing .Fig. (13) shows that the swelling ratio increasing with the runner percentage increasing up to 50% runner and then reduced to a value close to pp swelling due to the MFR and shear rate increasing and decreasing respectively .Also the swelling increasing with the temperature decreasing due to the viscosity increasing .Fig.(14) indicates that the swelling ratio increasing with the load and runner percentage increasing up to 50% runner and then reduced to a value close to pp swelling due to the MFR and shear rate increasing .Fig.(14) indicates that the swelling ratio increasing with the load and runner percentage increasing up to 50% runner and then reduced to a value close to pp swelling due to the MFR and shear rate increasing and decreasing respectively.







Fig. (14) : swelling ratio behavior with the runner percentage for different loads of second size at 230 °C

Density Effect: Fig.(15) shows the density decreases with the runner percentage and temperature increasing up to 50% runner, due to the MFR increasing. Polymer chain broken and molecular weight reduction associated with the shear flow of the melt through the capillary die .The density is drop rapidly up to 50% runner and then increases to the value corresponding to 10% runner. **Fig.(16)**, illustrates the density decreasing with the runner percentage up to the 50% runner and load increasing due to MFR increasing .The density decreasing rapidly at load 2.16 kg and 3.80 kg and exhibit more stable at load 1.965 kg. At 100% runner the density increases to the level close to that at 10% runner.



Fig. (15): density behavior with the runner percentage for different temperatures of second size at load 2.16 kg



Figure (16): density behavior with the runner percentage for different loads of second size at 230 °C

CONCLUSIONS

- 1. The MFR of the blends increases and density decreases with runner percentage, temperature and load increasing
- 2. The swelling ratio increases with MFR and load increase and temperature decreases
- 3. The pellets volume reduction of runner waste adding to the virgin PP may be reduces the MFR and the adding runner percentage can be increased
- 4. MFR and shear rate increase with the density, molecular weight and viscosity decreasing ,therefore ,the MFR gives a good indication about the runner waste, structure ,rheological ,thermal and mechanical properties of polymer melt.
- 5. Runner alone can be used to obtain MFR value close to that for pp.
- 6. pp and pure runner describing as NMWD due to convergent of chains length, while the pp blends behaves as BMWD due to the different in the chains length.

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