IMPACT ANALYSIS ON COMPOSITE OF PET

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ABSTRACT

Analysis has been conducted on thermoplastic Polyester Polyethylene Teraphthalate (PET) with present of two types of fillers and Thermoplastic Elastomer (TPE). PET has shown unique and various characteristics when added with two types of additives or fillers which are Calcium Carbonate (CaCO₂) and Wollastonite (CaSiO₂) with or without existence of 10% weight of TPE. These fillers were added to PET at 5%, 10%, 15%, 20%, 25% and 30% with respect to weight of PET. PET has the low impact strength among the other polymers. Therefore this research has been carried out to PET with adding some filler to increase the impact strength. In order to produce the specimens, the fillers need to be sieved with size 45µm using sieve shaker machine. The specimens prepared according to 5%, 10%, 15%, 20%, 25%, and 30% weight of fillers using injection molding machine. Charpy impact testing has been carried out to analysis the energy absorbed by the specimen before fractured. Based on the results, the percentage of additives that can increased the impact strength are 90% PET + 10% CaCO₃, 95%PET + 5% CaSiO₃, 60% PET + 30% CaCO₃ + 10% TPE, and 60% PET + 30% CaSiO, + 10% TPE. However, highest impact strength is given by 70% PET + 20% CaCO₃ + 10% TPE and 65% PET + 25% CaCO₃ + 10% TPE with value 0.407 J/mm2. In wrapping up, impact strength given by CaSiO, is higher than CaCO, without the existence of 10% TPE. 10% TPE is playing an importance role in increasing the impact strength of PET with CaCO₃. Naturally PET is non toxic, so it can be use as food wrapper, and bottles. Besides that, PET+20% CaCO₃+10%TPE, PET+25%CaCO₃+10%TPE, PET+20% CaSiO₃, and PET+30% CaSiO₃+10%TPE also increased the impact strength of PET, so it can be use to produce telephone casing, mouse, key board replace with ABS if it would reduce the manufacturing cost.

KEYWORDS: Polyester Polyethylene Teraphthalate (PET), Thermoplastic Elastomer (TPE), Calcium Carbonate (CaCO₃) and Wollastonite (CaSiO₃)

1.0 INTRODUCTION

PET is a thermoplastic polymer resin of the Polyester family and is used in synthetic fibers, beverage, food and other liquid containers; thermoforming applications; and engineering resins often in combination with glass fiber. It is one of the most important raw materials used in man-made fibers. Depending on its processing and thermal history, it may exist both as an amorphous (transparent) and as a semi-crystalline material. Its monomer can be synthesized by the esterification reaction between terephthalic acid and ethylene glycol with water as a by-product, or the transesterification reaction between ethylene glycol and dimethyl terephthalate with methanol as a by-product. Polymerization is through a

polycondensation reaction of the with ethylene glycol. The chain structure of PET monomer is presented in Figure 1.

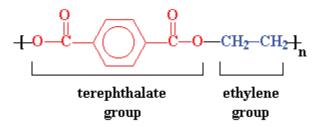


FIGURE 1
PET repeating unit (www.eng.buffalo.edu)

Organic or inert nucleating agents are added to allow PET crystallization to occur during the extrusion process. A target crystallinity of approximately 30% is desired for semi-crystalline CPET dual oven trays (www.eng.buffalo.edu).

Invention of PET in industry has successfully replaced metal, thermoset, and other thermoplastic. Among the advantages of PET are such as good dimensional stability, less moisture absorption, good surface finishing, colour and easy manufacturing. PET also has been used as fiber synthetic, photography, video tape, oven container, computer, magnetic tape and in various types of bottles. The disadvantage of PET is not applicable for higher impact strength. The characteristics of pure PET and the impact strength of PET with addition of fillers are highlighted in Table 1 and Table 2 respectively. The addition of fillers with polymer increased the impact strength.

TABLE 1 Characteristics of PET (Richardson, 1997)

Characteristics of LET (Kichardson, 1997)				
CHARACTERISTICS	PET			
Molding quality	Good			
Relative density	1.34 -1.39			
Tensile strength (MPa)	59 – 72			
Impact strength (ft Ib/in) or (J/mm)	0.01 - 0.04 or $0.2 - 0.8$			
Hardness	Rockwell M94-M101			
Thermal expansion, 10-4/°C	15.2 – 24			
Heat resistance, °C	80 - 120			
Dielectric strength, V/mm	13780 – 15750			
Water absorption (24 h), %	0.02			

TABLE 2 Impact strength of PET with addition of fillers (Krosschwitz, 1990)

Thermoplastic	Izod, J/m		
	22°C -44°C		
PET	-	-	
30% of glass filler PET	101	96	
Reinforcement of 30% glass PET	139-235	123-160	

1.1 FILLERS / ADDITIVES

a. CALCIUM CARBONATES (CaCO₃)

 $CaCO_3$ is used as filler in thermoplastic and thermoset as well. It's widely used in rubber and plastic application because can reduce the cost of raw material. $CaCO_3$ also can be found in lime stone which is from natural minerals. Among the carbonates, $CaCO_3$ which can get in calcite form is one of the importance fillers. Calcite is a soft mineral. Utilization of $CaCO_3$ as fillers able to produced cable, floor, lather made, pipe, profile, film. On the other hand, $CaCO_3$ widely used in rubber manufacturing for extruded and moulded white articles.

TABLE 3
Physical Characteristic of CaCO₃ (www.calcium/calciumcarbonateanddolomitefillerapplications.htm)

Fillers	Density	Mohr	Modules	Melting	Degradation
	(g cm ⁻³⁾	Hardness	(GPa)	Temperature,	temperature
				Tm (°C)	
CaCO3	2.65	2.5-3	179	1340	800

b. WOLLASTONITE (CaSiO₃)

Wollastonite, or $CaSiO_3$, is soft and has crystalline structure where it is in circular shape. $CaSiO_3$ is used as effective filler in melamine, polyester, epoxy, vinyl, polyethylene and nylon composite. Beside that $CaSiO_3$ is important in ceramic and paint manufacturing where is act as filler. $CaSiO_3$ was formed from limestone which contents calcite, $CaCO_3$, and silica (SiO_2) in magma as in equation below (Richardson, 1997):-

$$CaCO_3 + SiO_2 \rightarrow CaSiO_3 + CO_2$$

TABLE 4
Physical characteristic of CaSiO₃ (Richardson, 1997)

Fillers	Density (g	Mohr	Melting	Degradation
	cm ⁻³⁾	Hardness	Temperature,	Temperature
			Tm (°C)	_
CaSiO ₃	2.8-2.9	5-5.5	1390-1410	750

CaCO₃ and CaSiO₃ have ability to improve the material characteristics such as heat resistance, hardness, and process of manufacturing highlighted (Table 5).

TABLE 5
Mechanical characteristic of CaCO₂ and CaSiO₃ (Richardson, 1997)

Fillers	Effect			3	3 .	•	
	Procession	Heat resistance	Stiffness	Hardness	Humidity resistance	Impact strength	Dimensional stability
CaSiO ₃	X	X	X	X	X	X	X
CaCO ₃	X	X	X	X	-	-	-

Mechanical characteristic of polyamide-6, with and without fillers are highlighted (Table 6). The filler added according to weight of polymer. This table proved that CaCO₃ and CaSiO₃ can be used as fillers.

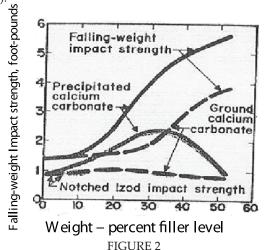
TABLE 6 Mechanical strength of Polyamide 6,6 with and without addition of fillers (Anthony et.al., 1994)

Characteristics	Without filler	CaCO ₃	Wollsatonite
		(40% of weigth)	(CaSiO ₃)
			(40% of weigth)
Density	1.14	1.48	1.51
(g cm-3)			
	81.4	72.2	72.2
Tensile Strength			
(MPa)			
Impact strength Izod	2.67	26.7	32
Notch (J m)			

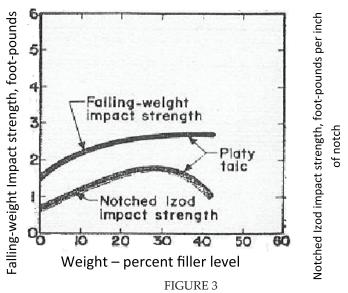
Even though, these fillers are having ability to improve the impact strength of PET, however the strength of PET depends on the structure bonding with fillers. Therefore, the analysis of impact strength has been carried out to ensure PET composite is suitable for application which is involving the impact strength.

c. THERMOPLASTIC ELASTOMERS (TPE)

TPEs are a family of polymers that can be repeatedly stretched without permanently deforming the shape of the part. Unlike rubber-like elastomers, they do not require curing or vulcanization, as they are true thermoplastics. Thermoplastic elastomers (TPEs) may be processed by conventional thermoplastic techniques such as injection molding, extrusion and blow molding. Thermoplastic elastomers have replaced rubber in many applications, most importantly the automotive industry (www.ides.com/generics/TPE.htm).



Falling weight impact strength (black) and notched Izod impact (green) of blends of PS with calcium carbonate and 10% TPE (Joseph, 1977)



Falling weight impact strength (black) and notched Izod impact strength (green0 of blends of crystal PS and platy talc, with 10% TPE (Joseph, 1977)

According Figure 2 and 3 shows relation between three types of fillers with 10% of TPE added to polystyrene. Based on Figure 2, when TPE added with CaCO₃ it will increase the impact strength of polystyrene. In other word, TPE acts as a surface treatment for CaCO₃ (Joseph, 1977).

2.0 PROBLEM STATEMENT

Some of the impact strength of polymers was highlighted. Polyethylene Terephthalate (PET) showed lowest impact strength among the other polymers. Therefore a research has been carried out to PET with adding some filler to increase the impact strength.

TABLE 7 Impact strength of polymers (Bucknall, 1999)

	Impact Strength (ft Ib/in)			
Polymers	Min	Max		
ABS	1.4	1.6		
Acetyl	1.1	2.3		
Epoxy	0.3	0.4		
Phenolic	0.24	0.4		
Polyamide	1.6	2.0		
Polycarbonate	1.4	2.0		
Polypropylene	0.4	1.4		
Polyethylene Terephthalate (PET)	0.01	0.04		

3.0 RESEARCH PURPOSE

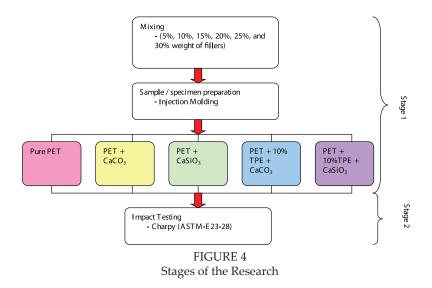
The main purpose of this researches to measures the impact strength for PET with fillers, which is $CaCO_3$, $CaSiO_3$, with and without existence of 10% of TPE. The results that obtained from this research will be compared with pure PET. The fillers will be added with PET according to difference percentages of 5%, 10%, 15%, 20%, 25% and 30% respect to weight of PET.

Therefore to achieve the research purpose, two objectives has been carried such as:-

- i. analyze the impact strength of PET with addition of fillers.
- ii. Obtain a suitable ratio of PET and additives.

4.0 MATERIALS AND METHODS

In order to achieve this research objective, the following steps (Figure 4) has been carried out.



Stage 1: Raw Material and Specimen Preparation

In order to produce the specimens, the fillers need to be sieved with size $45\mu m$ using sieve shaker machine. Both fillers were sieved in same size so that, can make comparison. The specimens prepared according to 5%, 10%, 15%, 20%, 25%, and 30% weight of fillers. PET and these fillers need to ensure wheatear both were mixed homogenously before carry out specimens' preparation.

Injection molding is a one of the manufacturing technique for making parts from thermoplastic material in production. Injection molding was chosen for specimen preparation because of the quality better than other process. Hence it is easy to control parameter such as pressure and temperature. Before specimen preparation, mold should be cleaned using toluene or benzene. Then, raw material (PET and fillers) added to injection unit. Heating coils are encircling the injection so that it could melt the polymers (PET and TPE). Volume of the raw materials should not more than

capacity of the injection unit. Otherwise, deformity will occur during the specimen preparation. Injection molding operation including mold is fixed and pressed in the injection molding machine using mechanical pressing power. Charpy Impact Mold ISO 179 has been selected for specimen preparation to perform Charpy test. Besides that, the injection process is operating with pressure existence. The maximum pressure was set to 8 bars for specimen preparation. The heating temperature is adjustable according to type of polymers that has been used. The tool temperature and polymer melting temperature for PET is 95°C and 275°C respectively.

Basically, the temperature and pressure is depends on the size of mold. Spur of mold should be placed exactly on nozzle of injection molding before inject carried out. The pressure remains constant during the injection process. While the injection unit reach the melting temperature, the melting flow of PET and fillers are good and easily flow and fill up the mold completely. This is a feeding process where it feed the melt polymer in a mold. Holding time is a time count during injection process to obtain a complete specimen in the mold. It's important to prevent short shot and sink marks on specimens during samples preparations.

Stage 2: Impact testing

Impact test are designed to measure the resistance to failure of a material to a suddenly applied force. The test measures the impact energy, or the energy absorbed prior to fracture. Beside that the impact energy is a measure of the work done to fracture a test specimen. When the sticker impacts the specimen, the specimen will absorb energy until it yields. At this point, the specimen will begin to undergo plastic deformation at the notch. The test specimen continues to absorb energy and work hardens at the plastic zone at the notch. However, fracture occurs when the specimen unable absorb excees energy.

Specimen for Charpy test is fixed as horizontal in place at both ends and the striker where the notch directed behind the striker. Size of the specimen for Charpy test is 10mm of width, 4mm of height and 79mm of length as well. Besides that, there were five specimens needed for each ratio of samples. Furthermore, the Charpy test was carried out with the pendulum which has standard weight lifted up at maximum height. Then the striker let free oscillate. As a result the striker will strike the notched specimen which was fixed. However the striker stopped by brake. Next the reading has been taken as energy absorbed using drag indicator. The same following methods used for other specimens.

5.0 RESULTS

5.1 Stage 1: Specimen Preparation Using Injection Molding

The prepared specimens Figure 5, 6, 7 and 8 with the existence of fillers were used for the impact testing purpose. Based on the figure, it's clearly shown that, pure PET looks brighter before added with fillers. The colour changed to yellowish (dimmer) when filler added to pure PET. The process of specimen preparation using injection molding become more difficult and consumed more time when the percentage of fillers increased. However, the process of specimen preparation becomes easier after added 10% of TPE. TPE successfully decreased the melting temperature and enhanced the melting flow rate (Table 8, 9, 10 and 11). Therefore, the specimen preparation became smooth.

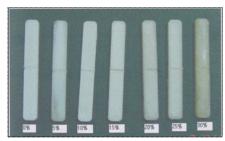
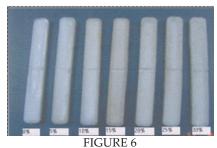


FIGURE 5
Sample specimen of PET with existence of % CaCO₃



Sample specimen of PET with existence of % CaCO₃ and 10% of TPE

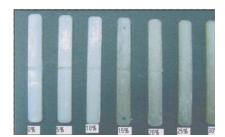


FIGURE 7
Sample specimen of PET existence of % CaSiO₃

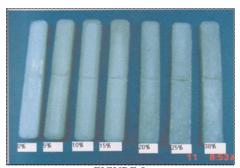


FIGURE 8
Sample specimen of PET with existence of % CaSiO₃ and 10% of TPE

TABLE 8
Parameter Injection for PET + CaCO₃

Specimen	Temperature (⁰ C)	Pressure (Bar)	Injection time(s)
PET	275	8	6
PET + 5%CaCO ₃	275	8	6
PET + 10%CaCO ₃	275	8	9
PET + 15%CaCO ₃	280	8	10
PET + 20%CaCO ₃	283	8	12
PET + 25%CaCO ₃	285	8	13
PET + 30%CaCO ₃	285	8	13

TABLE 9
Parameter Injection for PET + CaCO₃+10%TPE

ratameter injection for FET + CaCO ₃ +10/61FE						
Specimen	Temperature(⁰ C)	Pressure(Bar)	Injection time(s)			
PET	275	8	6			
PET + 5%CaCO₃ +10%TPE	240	8	8			
PET + 10%CaCO ₃ +10%TPE	240	8	9			
PET + 15%CaCO ₃ +10%TPE	240	8	9			
$PET + 20\%CaCO_3 + 10\%TPE$	240	8	10			
PET + 25%CaCO ₃ +10%TPE	240	8	10			
PET + 30%CaCO ₃ +10%TPE	240	8	11			

TABLE 10
Parameter Injection for PET + CaSiO₃

			3
Specimen	Temperature (⁰ C)	Pressure (Bar)	Injection time(s)
PET	275	8	6
PET + 5%CaSiO3	280	8	8
PET + 10%CaSiO3	280	8	9
PET + 15%CaSiO3	285	8	9
PET + 20%CaSiO3	285	8	10
PET + 25%CaSiO3	290	8	11
PET + 30%CaSiO3	290	8	13

TABLE 11
Parameter Injection for PET + CaCO.+10%TPE

Specimen	Temperature	Pressure	Injection
	(⁰ C)	(Bar)	time(s)
PET	275	8	6
PET + 5%CaSiO ₃ +10%TPE	240	8	8
PET + 10%CaSiO ₃ +10%TPE	240	8	9
PET + 15%CaSiO ₃ +10%TPE	240	8	9
PET + 20%CaSiO ₃ +10%TPE	240	8	10
PET + 25%CaSiO ₃ +10%TPE	240	8	10
PET + 30%CaSiO ₃ +10%TPE	240	8	11

5.2 Stage 2: Impact Testing

In order to obtain the impact strength, absorbed energy by fractured specimen has to be divided with the area of the notch. So that, the area of the notch has been calculated as shown in Figure 9.

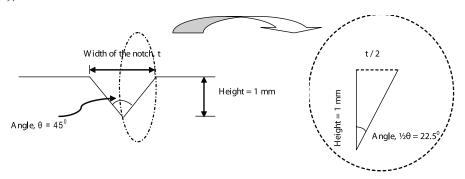


FIGURE 9 The Diagram of Notch

Assume that the area of notch for all specimens is same. The calculation for half of the notch as below:-

$$\tan 22.5^{\circ} = \frac{t/2mm}{1mm}$$

The width of the half of the notch = 0.383 mm

Therefore the area of half of the notch, A = $\frac{1}{2}$ x width x height = $\frac{1}{2}$ x 1 x 0.383 = 0.1915 mm2

So, the area of the notch can be calculated as $= 2 \times 0.1915 \text{ mm}.$ = 0.3830 mm.

Impact Strength =
$$\frac{Energy \quad Absorbed}{Area \quad of \quad Notch}$$
Impact Strength for pure PET
$$= \frac{0.102}{0.3830}$$

$$= 0.266 \text{ J/mm}^2$$

The impact test was carried for the pure PET and the mixture of PET with fillers. The Table 12, 13, 14 and 15 show the results that obtained for the impact test, average value as well. It need to be highlighted that the specimens are free from defects such as incomplete specimen, porosity, flash, and blister to obtain an accurate result.

TABLE 12
Impact strength of PET with addition of CaCO₃

		The va	lue of	Charpy	Testin	g, (J)	Impact strength
Sample	1	2	3	4	5	Average	,
						value	(J/mm^2)
PET	0.12	0.12	0.09	0.08	0.10	0.102	0.266
PET + 5%CaCO ₃	0.10	0.10	0.10	0.09	0.09	0.096	0.250
PET + 10%CaCO ₃	0.11	0.05	0.08	0.09	0.10	0.086	0.225
PET + 15%CaCO ₃	0.09	0.06	0.08	0.08	0.06	0.074	0.193
PET + 20%CaCO ₃	0.10	0.11	0.09	0.06	0.08	0.088	0.230
PET + 25%CaCO ₃	0.11	0.11	0.11	0.10	0.09	0.104	0.272
PET + 30%CaCO ₃	0.10	0.10	0.12	0.11	0.10	0.106	0.277

TABLE 13
Impact strength of PET with addition of CaSiO₃

	The value of Charpy Testing, (J)						Impact strength
Samp	1	2	3	4	5	Average	,
le						value	(J/mm ²)
PET	0.12	0.12	0.09	0.08	0.10	0.102	0.266
PET + 5%CaSiO ₃	0.11	0.10	0.13	0.10	0.12	0.112	0.292
PET + 10%CaSiO ₃	0.12	0.12	0.11	0.10	0.12	0.114	0.298
PET + 15%CaSiO ₃	0.13	0.12	0.11	0.12	0.15	0.126	0.329
PET + 20%CaSiO ₃	0.13	0.15	0.14	0.13	0.15	0.140	0.366
PET + 25%CaSiO ₃	0.10	0.11	0.10	0.12	0.10	0.106	0.277
PET + 30%CaSiO ₃	0.10	0.15	0.12	0.15	0.12	0.128	0.334

TABLE 14
Impact strength of PET and 10% TPE with addition of CaCO₃

	The value of Charpy Testing, (J)					Impact	
Sample	1	2	3	4	5	Average	strength,
						value	(J/mm^2)
PET	0.12	0.12	0.09	0.08	0.10	0.102	0.266
$PET + 5\%CaCO_3 + TPE$	0.12	0.11	0.10	0.10	0.11	0.108	0.282
PET + 10%CaCO ₃ + TPE	0.12	0.12	0.10	0.12	0.13	0.118	0.309
PET + 15%CaCO ₃ + TPE	0.12	0.12	0.11	0.10	0.11	0.112	0.292
$PET + 20\%CaCO_3 + TPE$	0.18	0.15	0.15	0.15	0.15	0.156	0.407
$PET + 25\%CaCO_3 + TPE$	0.15	0.16	0.16	0.16	0.15	0.156	0.407
PET + 30%CaCO ₃ + TPE	0.14	0.15	0.12	0.11	0.10	0.124	0.324

TABLE 15
Impact strength of PET and 10% TPE with addition of CaSiO₂

	The value of Charpy Testing, (J)					Impact strength	
Sample	1	2	3	4	5	Average	, _
						value	(J/mm ²)
PET	0.12	0.12	0.09	0.08	0.10	0.102	0.266
PET + 5%CaSiO ₃ + TPE	0.13	0.12	0.11	0.10	0.11	0.114	0.298
PET + 10%CaSiO ₃ + TPE	0.12	0.15	0.15	0.14	0.13	0.138	0.360
PET + 15%CaSiO ₃ + TPE	0.13	0.12	0.15	0.13	0.15	0.136	0.355
PET + 20%CaSiO ₃ + TPE	0.14	0.12	0.13	0.11	0.10	0.120	0.313
PET + 25%CaSiO ₃ + TPE	0.12	0.12	0.12	0.13	0.13	0.124	0.324
PET + 30%CaSiO ₃ + TPE	0.15	0.15	0.13	0.15	0.15	0.146	0.381

Finally, the overall results from table 12, 13, 14 and 15 are simplified in Fig.10. The results shows, that the impact strength of PET decreased when CaCO₂ added from 5% to 15% without 10% TPE. For example, pure PET gives 0.266 J/mm2 but adding 5% CaCO₃ causes the impact strength of PET reduced to 0.25. However, impact strength of PET had improved when the percentage of CaCO₂ increased from 25% to 30% without 10% TPE. Existence of 10% TPE together with CaCO₃ increased the impact strength of PET for all percentage for CaCO₂. Based on descriptions above, small percentage of CaCO₃ from 5% to 15% does not improve the impact strength of PET but adding 10% TPE increased the impact strength of PET. Therefore, TPE acts as surface treatment for CaCO₂. Contrastively CaSiO3 gives better toughness results compared to CaCO₂ even without 10% TPE. The highest impact strength obtained when 30% of CaSiO₂ added according to weight of PET. Generally, adding 10% TPE together with CaSiO₂ gives much better results where the impact strength improved further compared previous results without TPE except for 20% CaSiO₃. For example, adding 10 % TPE with 30 % CaSiO₂ has improved the impact strength from 0.334 to 0.381. As a conclusion, CaSiO₂ has shown better impact strength compared to CaCO₃. However, existence of 10% TPE more effective to CaCO₃ compared to CaSiO₃ seems CaSiO₃ itself gives better results. But the impact strength increased slightly when 10% TPE added to CaSiO₂.

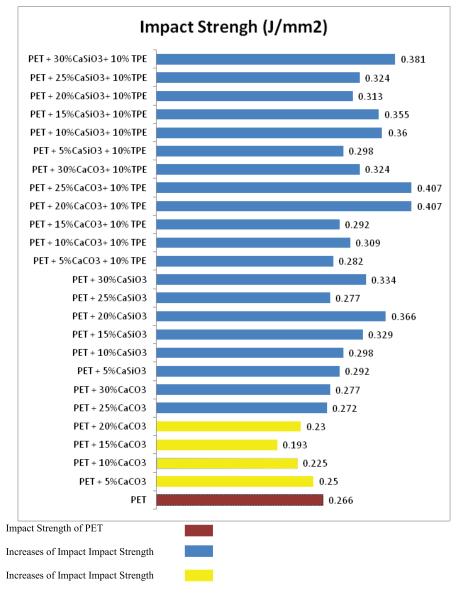


FIGURE 10 Impact Strength

6.0 CONCLUSION

In an engineering process, composition and the materials characteristic is very important for designing. This research results show that there were different in impact strength after fillers (CaCO $_3$ or CaSiO $_3$) and 10% TPE was added to PET. 10% TPE play an importance role in increasing impact strength of PET with CaCO $_3$. Adding CaCO $_3$ more than 25% of weight PET slightly increased the impact strength of PET however, CaSiO $_3$ able to increased the impact strength even without existence of TPE. Based on the finding, it can be concluded that the suitable ratio between PET and CaSiO $_3$ is 4:1, ratio between PET, CaCO $_3$, and 10%TPE is 7:2:1 and 6 ½:2½:1 as well. The suitable ratio of PET, CaSiO $_3$, and 10%TPE is 6:3:1. The highest impact strength for PET was given by 20% of CaCO $_3$, and 25% CaSiO $_3$ with 10% TPE. Therefore, this research had achieved the objectives.

Besides that, this research supported the research of (Joseph A.R, 1977) in Plastic Engineering Magazine who stated addition of TPE in a small amount successfully increased the impact strength of Polystyrene with $CaCO_3$ as filler. Richardson in his book "Industrial Plastics and Application" stated that $CaSiO_3$ also can be used as a filler to improve the impact strength. Therefore, this research also in line with theory that stated by Richardson where the experiment show that $CaSiO_3$ has higher impact strength.

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