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The effect of $CaCO_3$ filler component on mechanical properties of polypropylene

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Abstract. The presence of filler can reduce the production cost and in certain condition it can also improve the mechanical properties. CaCO3 is one type of inorganic filler that widely used in polypropylene materials, since it can cover those two requirements above. This work aimed to study the influence of CaCO3 filler in tensile properties of polypropylene. CaCO3 was sieved through a screen to obtain uniform particles size and then filled into polypropylene with filler percentage of 5%, 15%, and 25% of weight, respectively. The mixture was shaped into dumbell specimens by using Injection Moulding Process and a universal testing machine was used for tensile testing according to ISO 527. The results showed that the mixture of 5% (w/w) CaCO3 achieved highest tensile strain and elastic modulus but had the lowest tensile strength, and that of 15% (w/w) CaCO3 attained the highest values of strength. The highest tensile properties are 24.9 MPa for tensile strength, 3.96% for tensile strain and 963 MPa for elastic modulus. Interestingly, increasing the filler content to 25% of CaCO3 caused the reduction in strength, strain and elastic modulus of the composite. Variant with 5% of filler content has the highest value of impact strength, around 168 J/cm2, whereas the lowest value of impact strength can be found in the variant with 25% of CaCO3, the hardness value decreased on the 25% CaCO3. This situation is related to the filler distribution and dispersion within the specimen due to mixing effect in injection moulding process. Scanning Electron Microscope (SEM) was used to explain this condition by observing the fractured area of specimens.

1. Introduction

Nowadays, polypropylene (PP) is well known as one of the most common polymeric materials that are used in many application sectors such as household, construction and automotive [1]. In many cases, force and load are often subjected on those products, so the products should have a good mechanical properties to carry the subjected load. To improve mechanical properties and reduce production costs, PP is often reinforced by adding fillers into the matrix. From plastic industries point of view, talc and CaCO3 are attractive filler for thermoplastic especially polypropylene (PP) because they can fulfill those two requirements, but this study was only focus on $CaCO_3$ only. $CaCO_3$ is considered as a common inorganic filler that is widely used incorporated with polypropylene materials [2]. It was observed that the tensile strengths decreased while the modulus increased with the filler content for both talc and boron nitride. The strength could decrease due to the low interfacial bond between the components [3]. This type of filler has a positive effect on the nucleation effect of PP; fine-grade filler enhances strength and stiffness [4]. Moreover, the presence of filler reduces the percentage of resin usage, so that the production cost become lower. Despite the benefit, high filler content may affect the processability,

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ductility, and strength of composites [5]. It necessary to know the acceptable content of filler in the polymer matrix to reach their optimum performance. When using talc as filler, 30 wt% talc-filled PP was proven to have ultimate strength in many previous studies [6].

In the polymer matrix interface, dispersion, delamination, and distribution of talc have an important effect on the properties of PP/talc composites. Talc without special treatment tends to agglomerate when it is mixed with PP, resulting in bad dispersion. In addition, low compatibility between talc surface and polypropylene matrix leads to bad interfacial adhesion and causes poor mechanical properties of the composite.

In this study, the effect of filler percentage on the tensile properties of PP were investigated. There were three variation of filler percentage added into PP resin (5%, 15% and 25%) in the form of injection-molded specimens. Then, the tensile strength, strain and elastic modulus were observed and compared.

2. Experimental method

A commercial homopolymer polypropylene PP HI10HO from Chandra Asri Petrochemical company was used as the polymer matrix. The polymer material was supplied in granulated form. Some of physical properties value of resin material is written in the Table 1; they may helpful for comparison with the properties of investigated composites.

Table 1. Properties of PP HI10HO (source: http://www.chandra-asri.com).

Properties	Typical Value	Unit
Density	0.9	g/m^3
Melt flow Index (230°C / 2.16 kg)	10	g/10 min
Tensile stress at yield (50 mm/min)	35	MPa
Tensile strain at yield (50 mm/min)	13	%
Flexural modulus (1.3 mm/min)	1500	MPa
Notched impact strength, IZOD (+23°)	30	J/m^2
Hardness Rockwell	90	R-scale
Deflection Temperature (@ 0.455 MPa)	104	°C
Vicat Softening Temperature	152	°C

Considering material availability, a local grinded $CaCO_3$ was used as the filler material. It is normally a hygroscopic material and need to be dried at 30 °C – 55 °C in order to minimize the moisture content. After drying, the calcium carbonate was sieved in a such of manner to split the agglomeration and to get smaller particle size. The filler were mixed together with PP using three variation:

- 1st variation, 5% filler content: 50 g of $CaCO_3$ added into 950 g of PP
- 2nd variation, 15% filler content: 150 g of $CaCO_3$ added into 850 g of PP
- 3rd variation, 25% filler content: 250 g of $CaCO_3$ added into 750 g of PP.

Table 2. Optimum setting paramater of injection molding process.

Parameter	Typical Value	Unit
Injection Pressure	105	Bar
Injection Time	6.5	Second
Holding pressure	90	Bar
Holding time	4	Second
Melting temperature	230	°C
Cooling temperature	35	°C

The dumbell specimens were fabricated by the 70-ton MEIKI Injection Molding machine. The standard of processing procedures were based on ISO 294, whereas the shape of the dumbell specimen followed ISO 527 type 2. The high quality of injection molded specimens are indicated by several points: ideal and uniformly shaped as per drawing, minimum sinkmark and warpage, flatness etc. Those points can be achieved by optimum parameters that obtained from trials. Table 2 shows the optimum setting parameter of injection molding process.

To fulfill the testing standard, at minimum 5 specimens were prepared for each filler variations as can be seen on Figure 1.

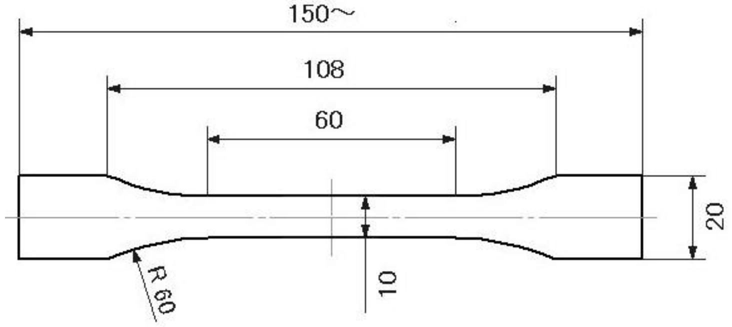

Figure 1. Dumbell specimen according to ISO 527-2 IB as result of injection molding process.

The specimens were tested by using a Zwick Roell Z020 Universal Testing Machine according to ISO 527 to get tensile properties data. Tensile testing was made at speed of 50 mm/min. Some other specimen were cut into standard dimension of test specimen (80 x 10 x 4 mm), as showed on Figure 2, and tested by Charpy Impact testing according to ISO 179 to get impact properties data.

Figure 2. Impact test specimen.

The Charpy impact test with three-points support were performed on notched specimens to evaluate the toughness behaviour of specimen. According to the standard, the support span is 62 mm. The pendulum hammer has nominal impact energy of 4 J and an impact velocity of 2.9 m/s. The sample were notched by angular single lip milling cutter, with the angle of 60° and base radius of 0.25 mm. The Shore D hardness testing according to ASTM D2240 - 15 was also perfomed on the specimen. There were three samples for each variations and for each of them, the indentation was applied. The hardness value of materials was determined by the change of depth of conical indentor when penetrating the surface of the samples. The results were also compared with the hardness of unfilled PP.

Material characterization was visually done by using Scanning Electrone Microscope (SEM); it was used to provide information in microstructures related to mechanical properties. The type of SEM is Hitachi TM3030 plus. Additionally, Energy Dispersive X-Ray Spectroscopy (EDS) was used to get semi-quantitative elemental results on the fractured area. Those two devices were provided by PT. Fajar Mas Murni.

3. Results and discussion

3.1. Tensile test

The tensile test result of PP/$CaCO_3$ with filler content variation of 5%, 15% and 25% gives three mechanical properties such as tensile strength, tensile strain and elastic modulus.

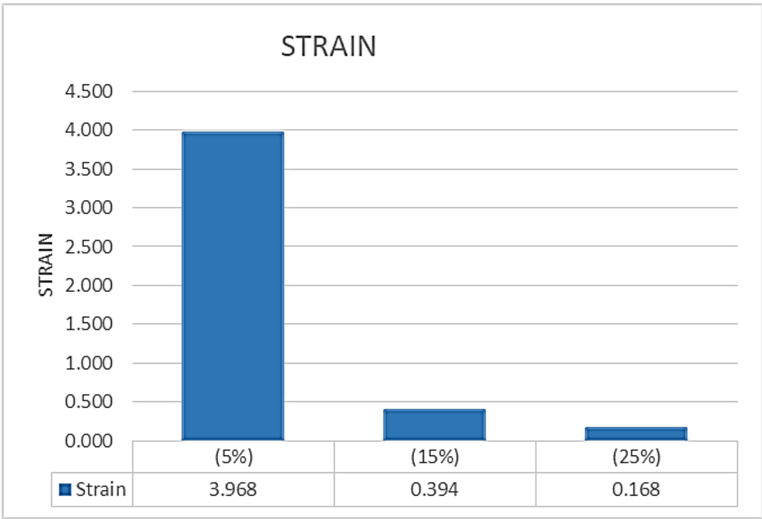

Figure 3. Strain result of three filler content variation.

According to material data, the strain of unfilled PP is 13%, Figure 3 shows that addition of filler reduces the strain of material significantly. Filler content contributes the brittleness of PP/CaCO3 composite material.

Figure 4 shows that the average value of elastic modulus of polypropylene material with filler content of $CaCO_3$ 5% is 963,2 MPa, then with 15% CaCO3 is 388,9 MPa and the lowest elastic modulus is given by 25% $CaCO_3$ which has the value at 333 MPa. It can be said that the addition of $CaCO_3$ to the polypropylene matrix reduce the elastic modulus. The first variant with 5% filler has the largest surface contact area which makes its elastic modulus higher, as described in some previous research that elastic modulus of polymer depending on the filler surface contact area [7].

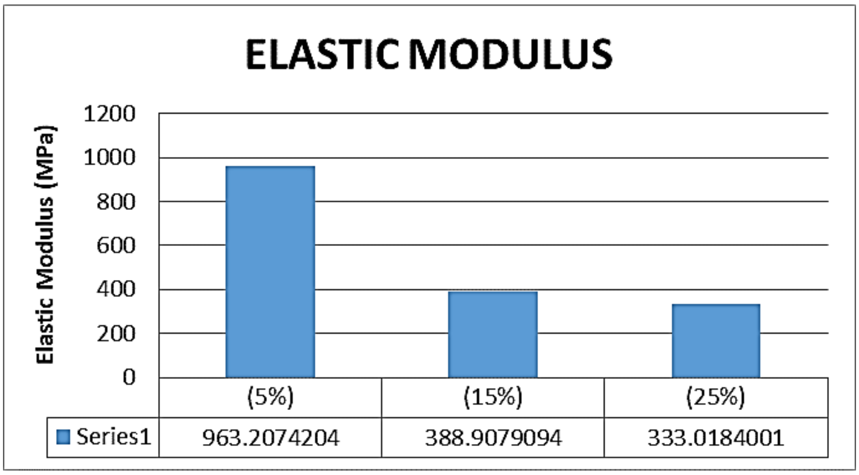

Figure 4. Elastic Modulus of three filler content variation.

Figure 5 explains that the polypropylene material with filler content of $CaCO_3$ 5%, 15% and 25% attained the tensile strength of 21.19 MPa, 24,9 MPa, and 22,13 MPa, respectively. The highest value of yield tensile strength is in $CaCO_3$ 15% filler. Interestingly, the larger contents of $CaCO_3$ (i.e. 25%)

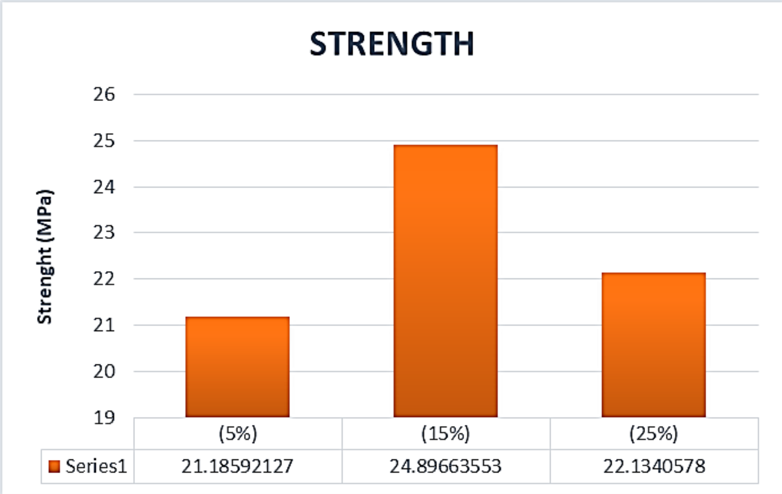

Figure 5. Yield tensile strength results.

did not bring to successive increase in the tensile strength. In contrast to the elastic modulus, yield strength depends on both surface contact area and interfacial strength [8]. Those factors were strongly related to the distribution and dispersion of filler particles contained inside matrix. Distribution is the way the particles fill the matrix, whereas dispersion is the way these particles are agglomerated or not. In order to get clear explanation of this issue, structure investigation was done on the cross section area of fractured specimen for both second and third variant by using Scanning Electron Microscope (SEM). Figure 6 shows that the filler in the second and the third variant are not fully distributed. Moreover, the dispersion on the 2nd variant is better than the 3rd variant. In general, the size of agglomerated filler (white spots) of third variant are bigger than the 2nd variant, so that the the ability to withstand pulling force is reduced, then the yield strength become lower. By increasing the magnification into 100x, as seen in Figure 7, it is clearly seen that the filler powder itself cannot blend properly. Inhomogeneous filler dispersion can lead to a considerable reduction in the mechanical strength of the composites.

Even at a smaller particle size the agglomeration was still occurred (Figure 8), indicating that the sieving process of filler was not optimal and the mixing effect in the barrel as well.

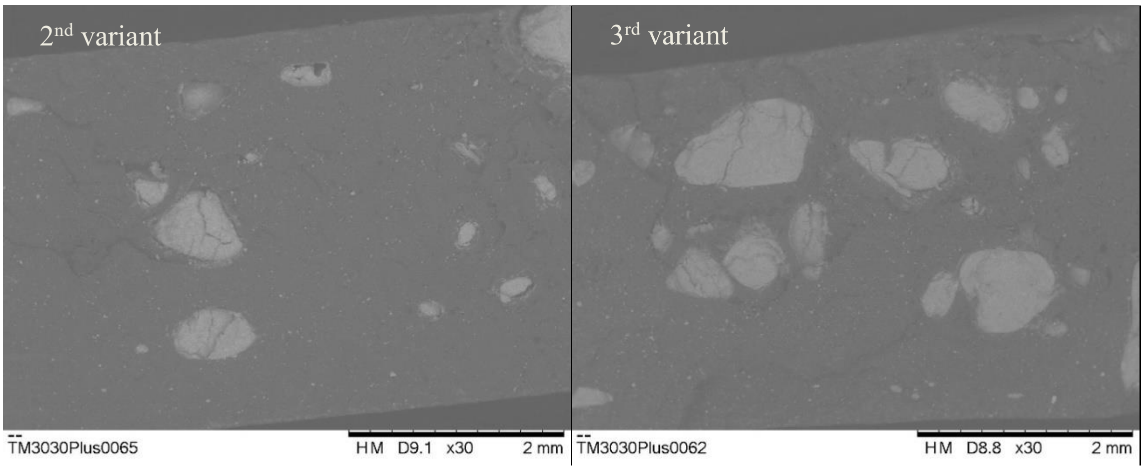

Figure 6. Filler distribution on fractured surface of 2nd variant (15% filler) and 3rd variant (25% filler).

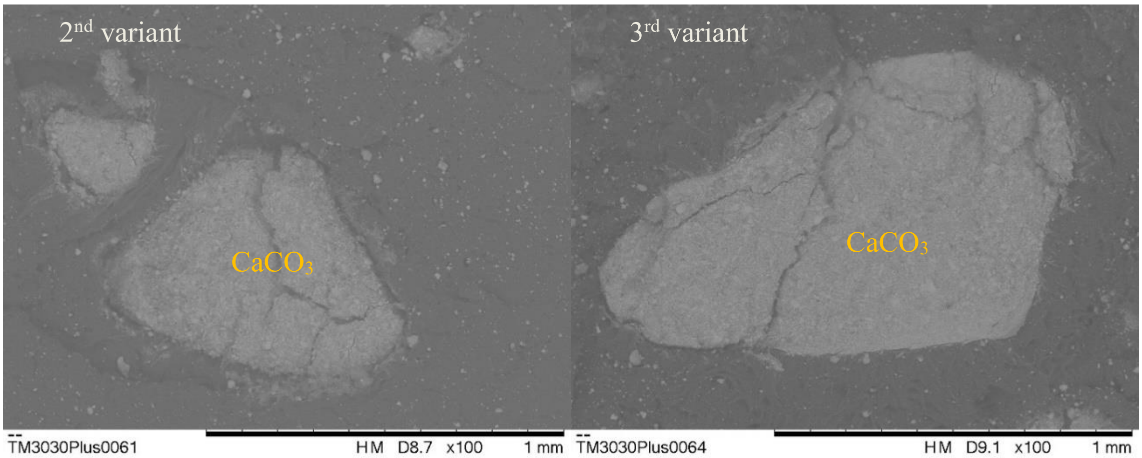

Figure 7. Filler agglomeration on fractured surface of 2nd variant (with 15% filler) and 3rd variant (with 25% filler).

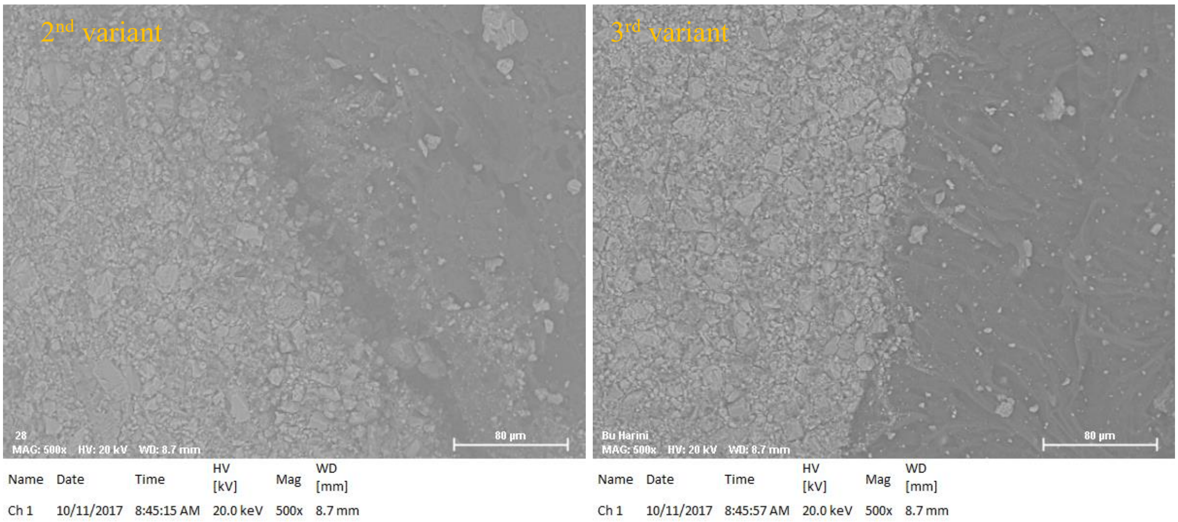

Figure 8. SEM images using 500x magnification.

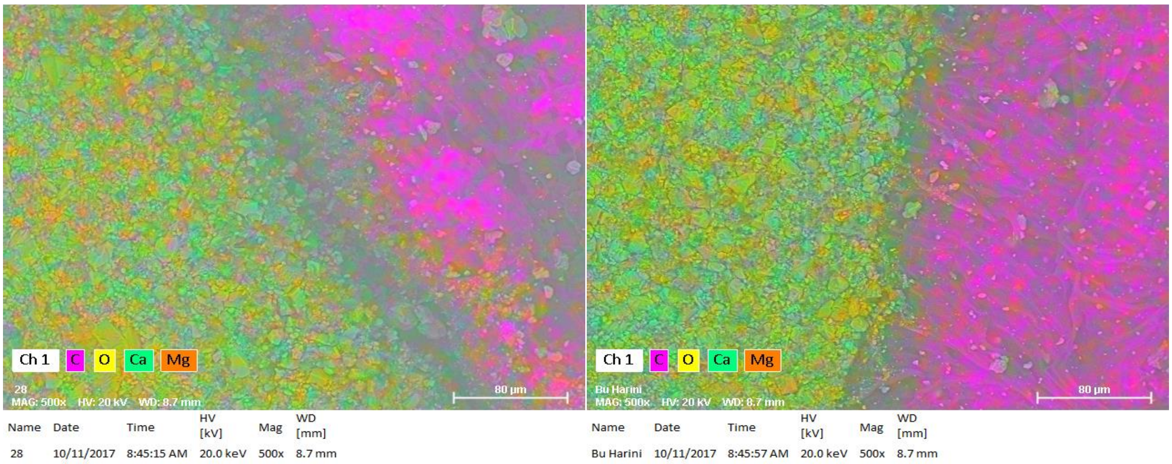

Figure 9. EDS images confirming the elemen of $CaCO_3$.

As the SEM show compositional contrast that results from the atomic distribution and different atomic number of elements, Energy Dispersive Spectroscopy (EDS) was used to identify the certain elements in the fractured area and their relative proportions. As shown in Figure 9, it is certain that the elements present on the surface of the fractured specimen consist of: C, Ca and O, which are the constituents of $CaCO_3$.

It is clear the dispersion and distribution of filler is strongly affected by two factors: first, powder preparation (sieving and drying) and second mixing effect on the injection molding process. To achieve better filler dispersion and distribution, it is necessary to have higher back pressure and faster screw rotation to get better mixing effect, unfortunately there is no screw rotation adjustment on the MEIKI 70-ton Injection Molding Machine.

3.2. *Impact test*

Figure 11 shows the relationship between filler content and the value of impact strength. The variant with 5% of filler content has the highest value of impact strength, around 168 J/cm2, whereas the lowest value of impact strength can be found in the variant with 25% of $CaCO_3$. It is understandable that the addition of $CaCO_3$ reduces the impact strength of PP, it become more brittle as shown in Figure 10. This confirmed the results of research conducted by J. Schoene *et. al.* [9].

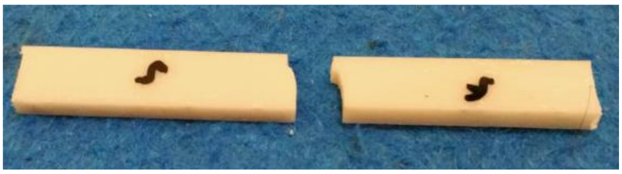

Figure 10. Brittle Fracture of PP/$CaCO_3$.

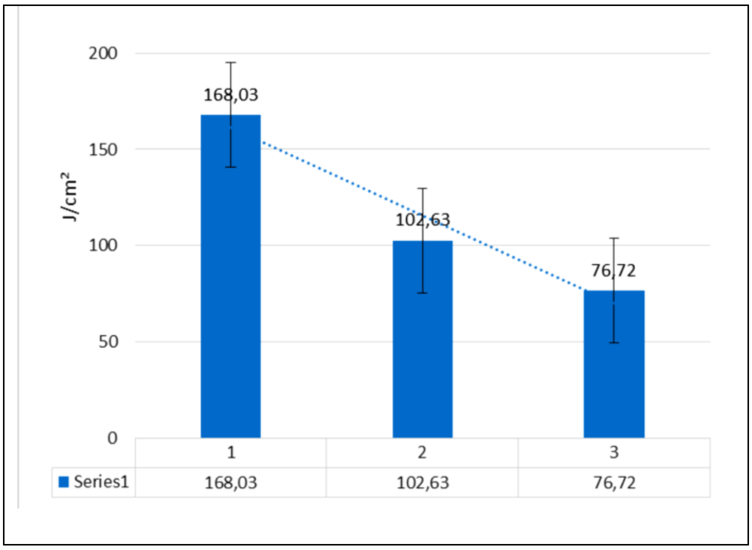

Figure 11. Impact test results.

3.3. *Hardness*

The hardness values (Shore D) of the filled PP composites with different percentage of $CaCO_3$ are shown in Figure 12. Normally, CaCO3 will reduce the molecular chain activity of the PP matrix, resulting in increasing its stiffness and and hardness correspondingly [10]. Here the situation is similar to the result of yield strength where the hardness value decreases for the 25% $CaCO_3$ content.

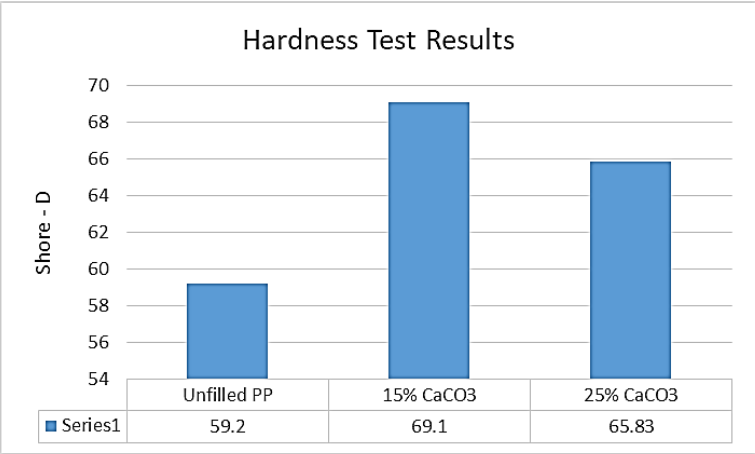

Figure 12. Shore D hardness comparison.

4. Conclusion

An investigation was carried out to study the effect of filler content on the elastic modulus, tensile strength, strain, hardness and impact strength of $CaCO_3$ filled PP. The results can be concluded as follows;

1) Elastic modulus and yield strain were decreased with the increasing of filler content
2) Tensile yield strength seems to be affected by the dispersion of the filler. This situation is related to the mixing effect in injection molding process.
3) Addition of $CaCO_3$ reduces the impact strength of PP.
4) The value of hardness increase with addition of filler but uneven distribution and dispersion impaired this properties.

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