Effect Of Nano-Caco₃ Content On Flexural And Impact Properties Of PPS/GF Ternary Composites

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ABSTRACT: In this paper a way to Prepare and determination of mechanical properties of a kind of nanometer calcium carbonate(nano-CaCO₃) filled glass fibre-reinforced Polyphenylene sulfide (PPS/GF) hybrid composites were introduced. The hybrid composites of PPS with 40% GF/ nano-CaCO₃ were fabricated by micro-compounder with DSM Micro 10cc injection moulding machine. Flexural modulus, flexural strength ,flexural extension at break point, Impact strength for Un notched and notched specimens of PPS/ GF/ nano-CaCO₃ were measured at room temperature using universal testing machine & Izod impact test machine, to identify the influence of the nano-CaCO₃ content on the mechanical properties (flexural &impact) of hybrid composites. It was found that there were relatively evident reinforcing and toughening effects of the nano-CaCO₃ on the PPS/GF hybrid composites. The flexural strength, flexural modulus of these composites increased nonlinearly with an addition of the nano-CaCO3 weigh fraction ($\emptyset_f$) when $\emptyset_f$ was less than 7%, and they reached the maximum at $\emptyset_f$ = 7%, and then decreased; while the flexural extension at break point increased as $\emptyset_f$ was less than 3%, and then decreased up to 5% and then improved . Notched and un-notched impact strength of these composites increased nonlinearly with an addition of the nano-CaCO3 weight fraction($\emptyset_f$) when $\emptyset_f$ was less than 5%, and then decreases. Surface morphology of flexural facture of this hybrid composite was analyzed by using SEM (Scanning Electron Microscope). Amount of elemental weight %, atomic % and compound% were identified using EDS (energy dispersive spectrum) software.

Key words: hybrid composite, PPS,GF, Nano-CaCO₃, Flexural modulus , EDS, SEM.

Introduction

Polyphenylene sulfide (PPS) has higher strength and good dimension stability, heat proof, flame retardant and chemical resistance, as well as better processing properties. However, the application of PPS is limited to a great extent due to its poor impact toughness and expensive price[1]. To improve the impact toughness and reduce cost, blending [2–4], fibre reinforcing [5] and particle filling composite[6,7] are usually used in industry. Akhtard and White[2] studied the properties and characteristics of binary and ternary blends of poly (p-phenylene sulfide) with poly (bisphenol A) sulfone and polyetherimide. Lim et al.[3] researched the thermal property and morphological study of polyphenylene sulfide-polycarbonate blends. Choi and Lim[4] investigated the effect of an epoxy on compatibility of poly(phenylene sulfide)=polycarbonate blend. Chen and his colleague[5] studied the friction and wear mechanisms of PA66=PPS blend reinforced with carbon fiber, and noted that the abrasive wear caused by ruptured CFs (for lower CF content) and the load bearing ability of CFs (for higher CF content) are the major factors affecting the wear volume. Recently, PPS was modified by using nanometer inorganic particles, such as nano-SiOX[6], nano-apatite[7] and the effect was good. Lu et al.[6] measured impact strength and crystallization property of nano-SiOX=poly (phenylene sulfide) (PPS) composites with heat-treated PPS. Yang and his colleague[7] studied the properties of a biocomposite of nano apatite=poly (1,4-phenylene-sulfide)-poly (2,4-phenylene sulfide acid). The results showed that the NAP=PPSPPSA biocomposite not only had good homogeneity but also had high NAP content of 60%, which was a potential bioactive material to be used as load-bearing implants or fixation in bone repair. PPS is a generally known to be insoluble in any solvent below 200°c. it has a high degree of crystallinity and good retention of physical

properties at elevated temperatures, so it is widely used for applications including mechanical, electrical and electronics. PPS has a glass transition temperature of 80-90^0 c and its melting point ranges between 285-295^{0}c.There are several methods to overcome the marginal properties of PPS including improving impact strength and high heat distortion temperature.

EXPERIMENTAL
Raw Materials

Polyphenylene sulfide with 40% GF was supplied by RK polymers ,mumbai,India. The melting temperature and density of this material are 285°C and 1600kg/m^3 The nanometer calcium carbonate was produced by Anyuan Technical Chemical Industry Co. Ltd, Jiangxi, China. The trade mark of the nano-$CaCO_3$ was CC-A, and the mean diameter and density of the particles were 80nm and 2500 kg/m3, respectively.

Fabrication of Composites and Specimens

Polypheylene sulfide with 40% GF were pre-dried for 3hours at 110-120^{0}C and nano-$CaCO_3$ pre-dried for 2 hours at 80^{0}c in vacuum oven. The nano-$CaCO_3$ particles were mixed with the PPS resin and the glass fiber according to designed blending ratios . In this work, the weight fraction of the glass fiber was fixed as 40%, and the weight fractions of the nano-$CaCO_3$ were 3, 5 and 7%. And then the PPS/GF/ nano-$CaCO_3$ blends were extruded in micro-compuder (15 ml DSM Xplore) .The extruder barrel temperature range was 290-295^{0}C. After extrusion process then go for molding by mini-injection jet machine to form the test specimens for impact and flexural tests. The specimens for impact and flexural tests were fabricated according to ASTM D256 standard and ASTM D790 respectively. In this work, the V-notched and un-notched specimens were prepared for impact test.

Instrument and Methodology

The impact properties of the PPS/GF/ nano-$CaCO_3$ ternary composites were measured at room temperature by means of a Izod impact machine (Tinius olsen, germaney) .Tests were conducted according to ASTM D256 standard. The flexural properties of the PPS/GF/ nano-$CaCO_3$ ternary composites were measured at room temperature by means of a universal materials testing machine (INSTRON,3382). Tests were Conducted according to ASTM D790M standard with cross-head descending speed of 2 mm/min.

In these tests, Each group specimens contained 5pieces, and the average values of the mechanical properties were used from the measured data.
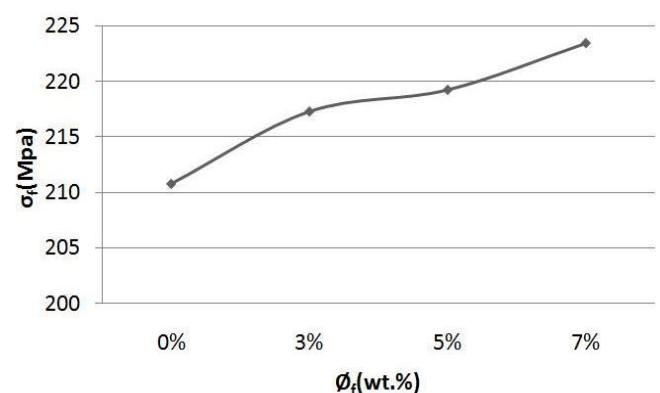
RESULTS AND DISCUSSION

Flexural Strength:

fig (a) shows the dependence of the flexural strength of the PPS/ GF/ nano-$CaCO_3$ composites on the weight fraction of nano-$CaCO_3$ particles.

When $\emptyset_f$ is smaller than 7% flexural strength increased non linearly .

When compared to the weight fraction ($\emptyset_f$) of 0% nano-$CaCO_3$, The Maximum. increase of the flexural strength at $\emptyset_f$ = 7% was 6.03% .
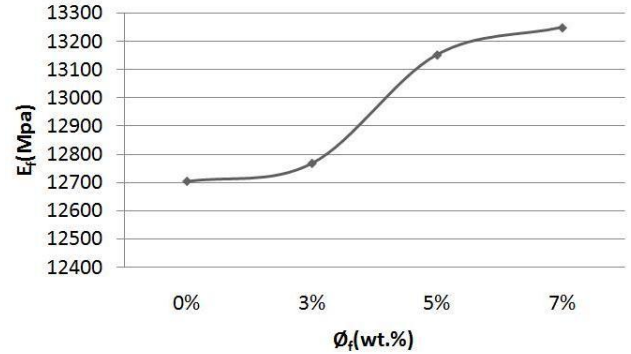
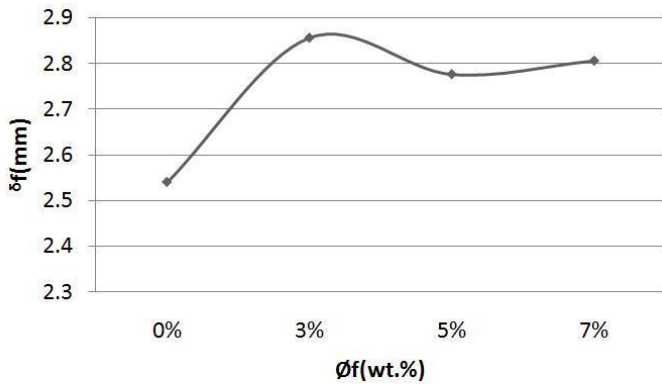Fig(a): Effect of weight fraction of NaNo CaCo3 on flexural strength

Flexural Modulus

Flexural modulus is an important parameter for characterization of the flexural stiffness of material. Fig(b) shows shows the dependence of the flexural strength of the PPS/GF/ nano-CaCO$_3$ composites on the weight fraction of nano-CaCO$_3$ particles. It can be seen that flexural modulus increased non linearly with the addition of weight fraction of nano-CaCO$_3$ ($\emptyset_f$). It means that the flexural stiffness of the PPS/GF binary composite materials filled with the increasing nano-CaCO$_3$ concentration will be enhanced effectively.

The Max increase of the flexural modulus (E_f) at $\emptyset_f$ =7% was 4.2% compared to the weight fraction of nano-CaCO$_3$ at 0%.

Fig(b): Effect of weight fraction of NaNo CaCo3 on flexural modulus

Fig(c): Effect of weigth fraction of NaNo CaCo3 on flexural extension at break point

Flexural extension at break point:

Flexural extension at break is one major parameter for characterizing the flexural fracture toughness of materials.

Fig(c) Shows the effect of the weight fraction of the distribution of the particles in the matrix and the interfacial morphology between them are improved better. Consequently, the flexural fracture toughness of the PPS/GF/nano-CaCO$_3$ hybrid composite was enhanced correspondingly.

The Max. increase of the flexural extention at break point (δf) at $\emptyset_f$ =3% was 12% compared to the weight fraction of nano-CaCO$_3$ at 0%.
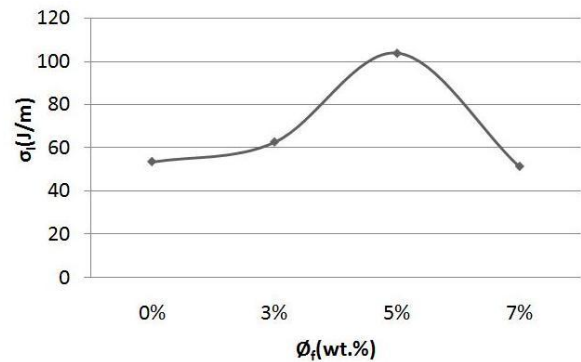
Here the flexural extension at break point (δf) increased non linearly up to $\emptyset_f$ =3% , with the addition of wt. fraction of nano-CaCO$_3$ then decreases up to $\emptyset_f$ =5% then slightly increases up to $\emptyset_f$ =7%.

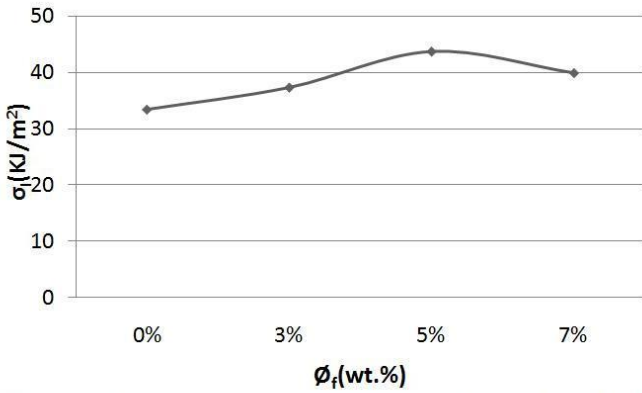## V-Notched Impact Strength and Un-notched impact strength
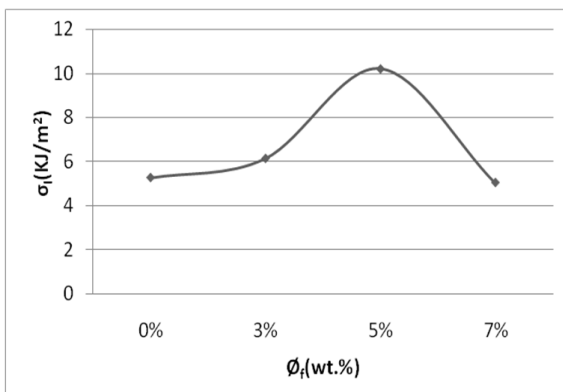
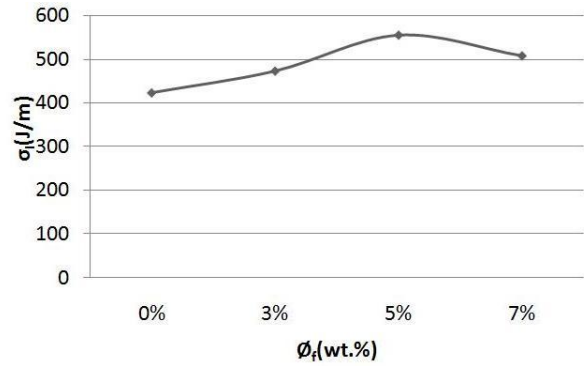Fig(d): relation between notched impact strength(σ_I) and weight fraction of Nano CaCO3($\emptyset_f$)

Fig(e): relation between notched impact strength(σ_I) and weight fraction of Nano CaCO3($\emptyset_f$)

Fig (d) and fig (e) shows the relationship between V-notched impact strength (σ_I) VS weight fraction of nano-CaCO$_3$ ($\emptyset_f$) in units KJ/m^2 and J/m.

From fig (d) and fig(e) When $\emptyset_f$ is smaller than 5% impact strength increased non linearly . maximum notched impact strength was observed at 5%. The Max. increase of notched impact strength was observed at wt. fraction of 5% nano-CaCO$_3$ was 96% compared to wt. fraction of 0% nano-CaCO$_3$ then decreased non linearly .
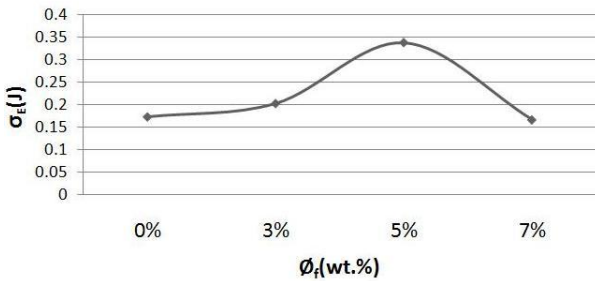
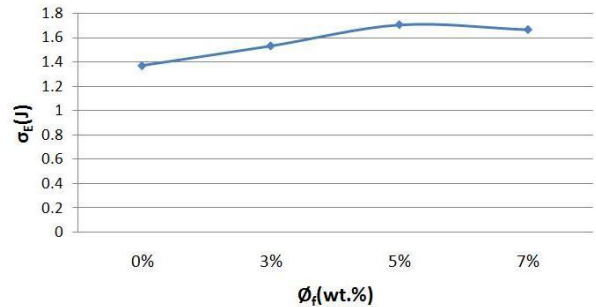Fig(f): relation between un-notched impact strength(σ_I) and weight fraction of Nano CaCO3($\emptyset_f$)

Fig(g): relation between un-notched impact strength(σ_I) and weight fraction of Nano CaCO3($\emptyset_f$)

Fig (f) and fig (g) shows the relationship between un-notched impact strength (σ_I) VS weight fraction of nano-CaCO$_3$ ($\emptyset_f$) in units KJ/m^2 and J/m.

From fig.(f) and fig.(g) When $\emptyset_f$ is smaller than 5%   impact strength was increased non linearly maximum un-notched impact strength was observed at 5%. then decreased . when compared to wt. fraction of 0% nano-CaCO$_3$ the Max. increase of un- notched impact strength was observed at 5% wt.fraction of nano-CaCO$_3$ was 33 %.

Fig(h): relation between notched breaking energy(σ_E) and weight fraction of Nano CaCO3($\emptyset_f$)

Fig(I): relation between un-notched breaking energy(σ_E) and weight fraction of Nano CaCO3($\emptyset_f$)

Fig(h) shows the relationship between notched impact breaking energy(σ_E) VS weight fraction of nano-CaCO$_3$ ($\emptyset_f$)

Breaking energy increased non linearly with increase of addition of wt.fraction of nano-CaCO$_3$ upto $\emptyset_f$ =5% then decreased. when compared to wt. fraction of 0% nano-CaCO$_3$ the Max. increases of breaking energy at weight fraction of 5% nano-CaCO$_3$ was ~75% .

Fig(I) shows the relationship between un- notched impact breaking energy(σ_E) VS weight fraction of nano-CaCO$_3$ ($\emptyset_f$).

Breaking energy increased non linearly with increase of addition of wt.fraction of nano-CaCO$_3$ upto 5% then decreased . when compared to wt. fraction of 0% nano-CaCO$_3$ The Max. increase of breaking energy at weight fraction of 5%nano-CaCO$_3$ was 21.5% .

The preceding results indicate that it is beneficial to improve the impact toughness of the PPS/GF composite materials as inclusion of the nano-CaCO$_3$ particles. When the Nano composite sytems are subjected to Impact load, the matrix resin will produce deformation and the interface between the filler and matrix will generate a debonding phenomenon to form voids to absorb impact deformation energy owing to the nano-CaCO$_3$ being rigid inorganic particle and also might absorb impact deformation energy ,improving the impact facture toughness of these composite systems. In addition , the nano-CaCO$_3$ particles may block the further expanding of the micro cracks , resulting from the enhanced impact toughness of the composite system(8-10).
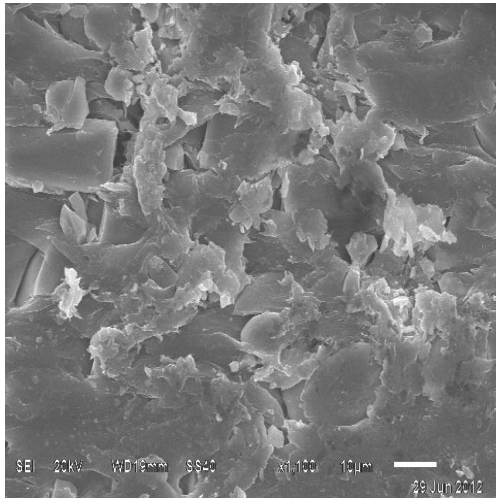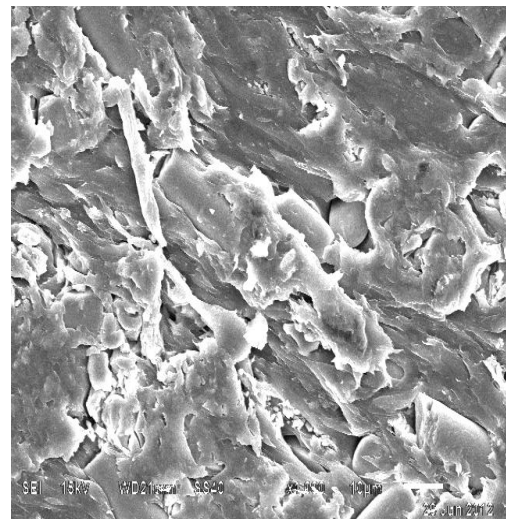

SEM : PPS/40%GF/0% nano-CaCO$_3$
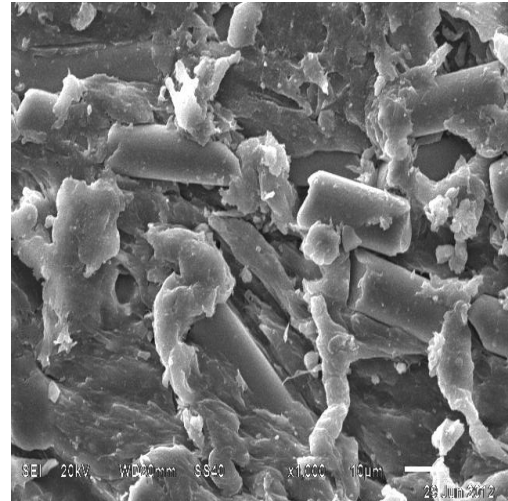
SEM : PPS/40%GF/3% nano-CaCO$_3$
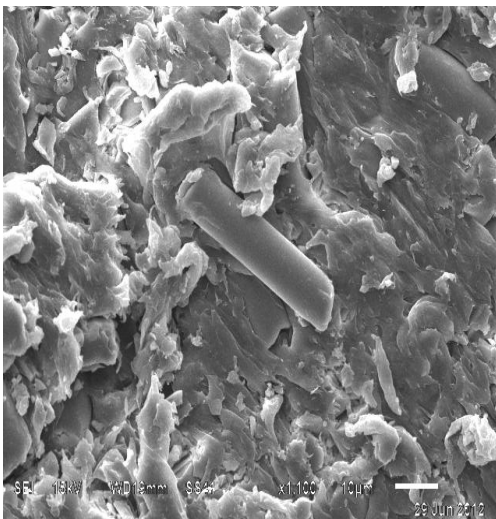
SEM : PPS/40%GF/5% nano-CaCO$_3$

SEM : PPS/40%GF/7% nano-CaCO$_3$

SEM: flexural facture of the polymer nano composites of different %weight fraction of nano-CaCO$_3$.

SEM(scanning electron microscope) photo graphs shows facture due to flexural load .These are shows surface morphology of the of polymer nano composites. i.e. how the nano particles distributed with increase of %wt. blending with PPS with 40%GF. and also seen , With an increasing loading of nano-CaCO$_3$, the nano particles could not be evenly distributed and form agglomerates. The incompact particle agglomerate would lead to a decrease in flexural.
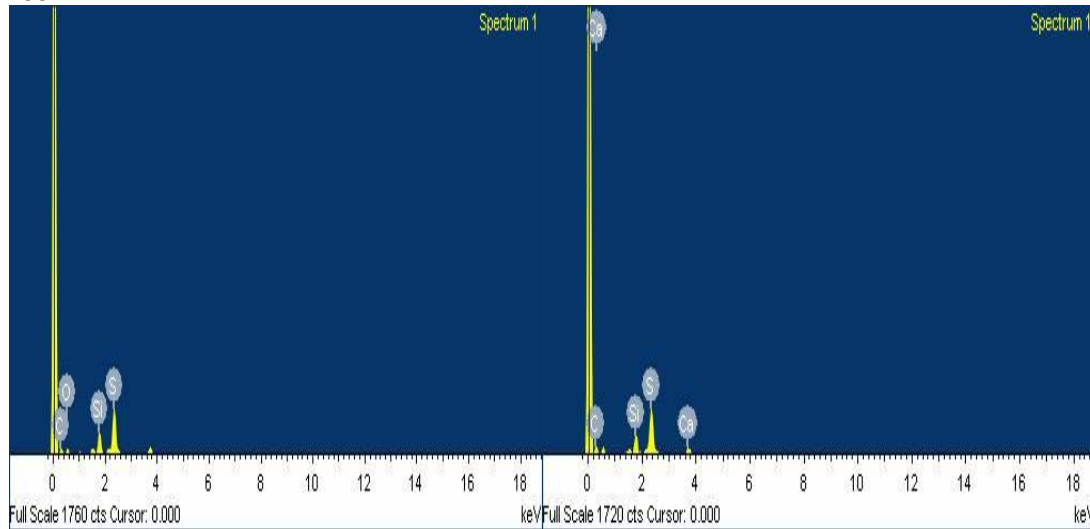

FIG1
EDS : PPS/40%GF/0% nano-CaCO$_3$

FIG2
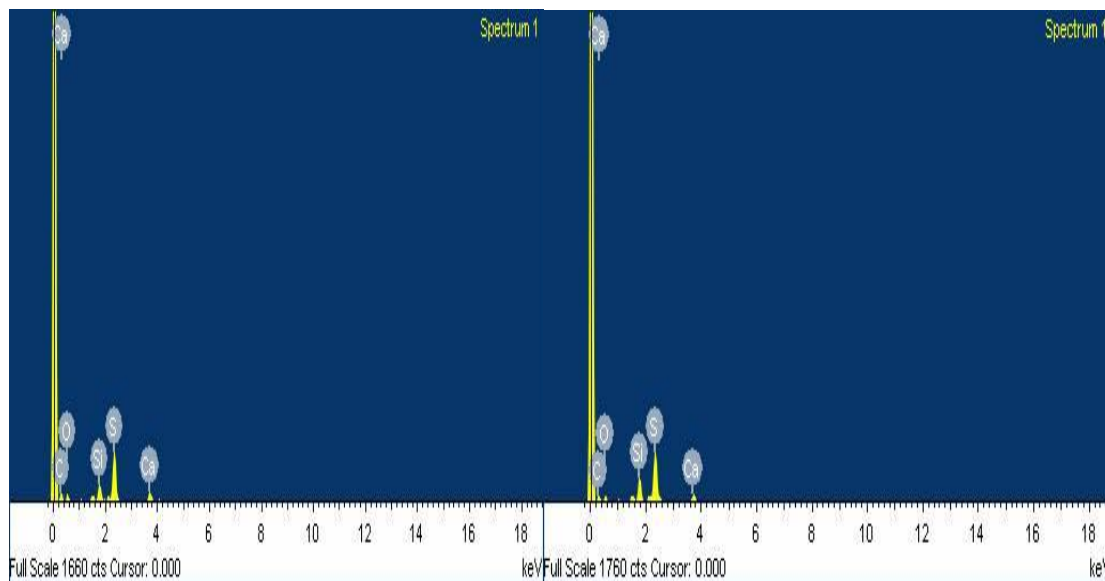EDS: PPS/40%GF/3% nano-CaCO$_3$

FIG3
EDS : PPS/40%GF/5% nano-CaCO$_3$

FIG4
EDS : PPS/40%GF/7% nano-CaCO$_3$

EDS: FIG1-4 Shows EDS RESULTS FOR DIFFERENT % WEIGHT FRACTIONS OF Nano- CaCO$_3$

DISCUSSION: As stated above, the mechanical strength of composite materials depends, in a great extent, upon the interfacial adhesion between the filler and the matrix. For rigid sphere particle-filled

polymer composites, Nicolais and Narkis[11] proposed a following equation on the basis of poor interfacial adhesion:

$$\sigma_c = \sigma_m (1-1.21 \, \emptyset_f^{2/3}) \qquad\qquad (1)$$

where σ_m is the tensile strength of the matrix resin. In fact, there should be a certain adhesion strength in the interface between the inclusions and the matrix. On the basis of this, Liang and Li[12,13] introduced a concept of interfacial adhesion angle (h), and derived a modified tensile strength equation:

$$\sigma_c = \sigma_m (1-1.21 \sin^2\theta \, \emptyset_f^{2/3}) \qquad\qquad (2)$$

It is known by comparing both equations that Eq. (2) is Eq. (1) when θ is equal to 90. In other words, the smaller the θ , the better the interfacial adhesion state.

It is a good interfacial adhesion when θ is equal to zero. In this case, $\sigma_c = \sigma_m$. It can be seen that in Fig.(a) that σ_c is greater than σ_m. This indicates that there is a good interfacial adhesion between the nano-CaCO3 particles and the PPS/GF matrix in the conditions.

CONCLUSIONS

There are a certain reinforcing and toughening effects of the nano-CaCO$_3$ on the PPS/GF composite materials. It was found that when the weight fraction of the nano meter particles was less than 6%, the flexural strength and flexural modulus at break increased non linearly with an increase of $\emptyset_f$. In addition The Maximum increase of the flexural strength was 6.03% and Maximum increase of the flexural modulus was 4.2%. the flexural extension at break point ($^\delta$f) increases up to $\emptyset_f$=3% , with the addition of wt. fraction of nano-CaCO$_3$ ($\emptyset_f$) then decreases up to $\emptyset_f$ =5% then slightly increases . The Max. increase of the flexural extention at break point ($^\delta$f) at $\emptyset_f$=3% was 12%.

Maximum notched impact strength was observed at 5%.The Max. increase of notched impact strength was 96% compared to wt. fraction of 0% nano-CaCO$_3$ then decreases .

Maximum un- notched impact strength was observed at 5%. then decreases .The Max. increase of un- notched impact strength was 33% . Breaking energy increased non linearly with increase of addition of wt.fraction of nano-CaCO$_3$ up to $\emptyset_f$ =5% then decreased. The Max. increases of breaking energy was ~75% . Breaking energy increased non linearly with increase of addition of wt.fraction of nano-CaCO$_3$ up to 5% then decreased . The Max increase of breaking energy was 21.5% .The mechanical property measurement results showed that there was a good interfacial adhesion between the nano-CaCO3 and the PPS matrix..

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