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Effect of Amino Silane coupling agent on Mechanical properties of Nylon-6 / Cenosphere composites

Bhaskar I. Oza

Department of Plastic Engineering
Government Polytechnic, Ahmedabad
bhaskaroza@yahoo.com

Abstract — *Untreated and treated with Amino Silane Coupling Agent composites of Nylon 6/Cenosphere were prepared with different loading (10, 20 and 30 % w/w) of CS (5-100 µm) by co-rotating twin screw extruder. Injection molded specimens were prepared to evaluate mechanical properties of developed composites. Mechanical properties like Flexural strength, Flexural modulus, Tensile strength, Impact strength, Hardness and Abrasion resistance were evaluated for these composites.*

Addition of Cenosphere improves flexural modulus, flexural strength, and hardness and abrasion resistance besides reducing cost of the final product. Silane Coupling Agent modified CS improves impact strength, tensile strength and flexural strength compared to unmodified composites. As Cenospheres are generated from fly ash in thermal power plant, they are environment friendly, eco-friendly and help to preserve natural virgin filler. The results suggest application of Cenosphere filled Nylon-6 in light weight automotive parts instead of glass filled Nylon-6.

Keywords- *Cenosphere (CS), Nylon-6(PA6), Amino Silane Coupling Agent, Mechanical properties, Flexural strength, Flexural modulus, Tensile strength, Impact strength, Hardness and Abrasion resistance*

I. INTRODUCTION

A Cenosphere (CS) is a light weight, hard and rigid, waterproof, inert and hollow sphere which can be used as a cost effective filler to improve the properties of Nylon-6 and to produce a new class of engineering composite for automobile application. Cenospheres are 70% lighter than other mineral fillers. Cenospheres are unique free flowing powders composed of hard shelled, hollow, minute Spheres. A small proportion of the pulverized fuel ash (PFA) produced from the combustion of coal in power stations is formed as Cenospheres. Cenospheres are made up of silica, iron and alumina. Cenospheres have a size range from 1 to 500 microns with an average compressive strength of 3000+ psi. Colors range from white to dark gray. They are also referred to as microspheres, hollow spheres, hollow ceramic microspheres, micro balloons, or glass beads.

Nylon 6 – CS composite is less studied area and there is wide scope for research scholar to explore it in various automobile applications. The use of Cenosphere in the production of composite can turn the industrial waste into industrial wealth. This also solves the problem of storage of fly ash as well as brings down the product cost.

Continuous accumulation of fly ash during coal burning in power plant is one of the alarming environmental problems. The amount of continuously producing ash is much higher than its consumption and ash dump is continued to expand. But at the same time some unique properties of Cenospheres obtained from fly ash provides prospects for their use in many applications like ceramics, plastics and construction.

II. MATERIALS AND METHODOLOGY

A. Materials

Polymeric matrix material Nylon 6 (Grade: M28RC, Manufacturer: GSFC) was procured from GSFC, Vadodara. The MFI of Nylon 6 is 28gm/cc. The filler Cenosphere (Grade: CS100) was procured from Petra Buildcare Products, Bhavnagar). Particle size of Cenosphere is 5-100µm. The general properties of Cenosphere are:

- **Size range :** 5 - 500 micron
- **Wall Thickness :** 2 - 5 micron
- **Color :** White, Off-white or Grey
- **Bulk Density :** 0.3 - 0.6 g/cc
- **Melting Point:** 1250 - 1450 ^{0}C
- **Moisture Absorption :** < 2.5%
- **Coefficient of thermal conductivity :** 0.09W/mK
- **Specific Heat :** 0.28 Cal/g ^{0}C

- **Hardness :** 5 - 6 Mohr scale
- **Loss On Ignition :** 2% maximum
- **Solubility in Water :** Negligible

The chemical composition of Cenosphere is shown in table 1.

Table 1. Chemical composition of Cenosphere

Chemical Composition	**Wt.%**
SiO_2	55-61
Al_2O_3	26-30
Fe_2O_3	4-10
CaO	2-6
MgO	1-2
Na_2O, K_2O	0.45 - 0.55
CO_2 Gas	70%
N_2 Gas	30%

Silicon Coupling Agent aminoethyl amino propyl trimethoxysilane (Grade: Xiameter OFS-6020 Silane, Manufacturer: Dow Corning Corporation) was procured from Alekh Testing Centre, Vatva, Ahmedabad.

B. Silane treatment

The inorganic filler CS was surface treated with a silane coupling agent before being added to Nylon 6. The SCA was added (5% by weight of CS) in water with continuous stirring up to 30 minutes and 5% silane concentrated aqueous solution is prepared. This solution is then distributed through the Cenosphere particles and mixed so each particle was covered with coupling agent. Then it was air dried initially and after 24 hours oven dried to remove any water residue. The technique is widely used in industrial applications because large amounts of filler can be treated in short time.

C. Composite and specimen preparation

Nylon 6 – Cenosphere composites were prepared by Co-rotating Twin screw extruder (Make: SPECIFIC ENGINEERING & AUTOMATES) in processing laboratory, HLC, CIPET, Ahmedabad.. L:D ratio of screw is 40:1 and screw diameter is 21 mm. The temperature range used was 180-220 °C. As nylon 6 is hygroscopic material, it was predried at 85 °C for approximately 3 hours to remove moisture in an oven before compounding. Cenosphere was also predried at same conditions to remove moisture. This is necessary to have void free samples. First, 3 batches of untreated and then 3 batches of treated composites were prepared. Each batch was of 3 Kg size as shown in below table.

Table 2. Batch composition of PA6 and CS (untreated)

Batch (3 Kg)	**Composition**
PA6N	Nylon-6+Cenosphere 0Wt%
PA6CS10	Nylon-6+Cenosphere 10Wt%
PA6CS20	Nylon-6+Cenosphere 20Wt%
PA6CS30	Nylon-6+Cenosphere 30Wt%

Table 3. Batch composition of PA6 and CS (treated)

Batch (3 Kg)	**Composition**
PA6CST10	Nylon-6+Cenosphere 10Wt%
PA6CST20	Nylon-6+Cenosphere 20Wt%
PA6CST30	Nylon-6+Cenosphere 30Wt%

The test specimens for various tests were prepared by using Automatic Injection Molding Machine (Make: ELECTRONICA, Model: ENDURA 90) in Processing Laboratory, CIPET, Ahmedabad. Before loading the material in the hopper, the material was predried for about 3 hours at 85 °C to remove moisture which eliminates voids in the samples. The injection molding was carried out at 230 - 275 °C and different test specimens like dumbbell, bar (3 mm and 6 mm thickness) and disc were prepared.

D. Characterization techniques

Various instruments were used to evaluate the mechanical properties of Nylon 6/Cenosphere composites. Tensile strength was evaluated at laboratory conditions using Universal Testing Machine (Make: P.S.I. Sales P. Ltd)) as per ASTM D 638 method with a crosshead speed of 50 mm/min. Flexural strength and modulus were tested by AUTOGRAPH (AG-IS) according to ASTM procedure D 790. The testing speed was 2 mm/min and the span length was 50mm. Impact strength was measured by impact tester (CEAST, Resil Impactor) at ambient condition according to ASTM D 256. Specimens for impact strength were notch cut by CEAST notch cutter before testing. Rockwell Hardness values were measure by ASTM D 785 (R scale) with 1/2" ball indenter and 60 Kg load using Rockwell hardness tester (Make: P.S.I.SALES (P) LTD). These tests eres carried out at testing laboratory, CIPET, Ahmedabad. Abrasion resistance test was measured by TABER abraser 5131 according to ASTM D 4060 with 500 gm load and CS-10 abrading wheel at testing laboratory, CIPET, Bhopal.

III. RESULTS AND DISCUSSION

A. Flexural strength and Flexural modulus

Figure 1 and 2 shows the effect of Cenosphere concentration on Flexural strength and modulus of Nylon 6 for both Silicon coupling Agent (SCA) treated and untreated composite samples. Both Flexural strength and modulus increase with Cenosphere concentration as Cenosphere increases stiffness of the composites. At 10% CS loading flexural strength increases at increased rate then it increases with slow rate. SCA treated composite shows higher flexural strength than without modified composites due to increased interfacial bonding between matrix and filler.
Flexural modulus increases sharply with increase in CS content and it almost doubles at 30% loading of CS. In the case of treated CS, there is a nominal increase in flexural modulus than untreated or unmodified CS.

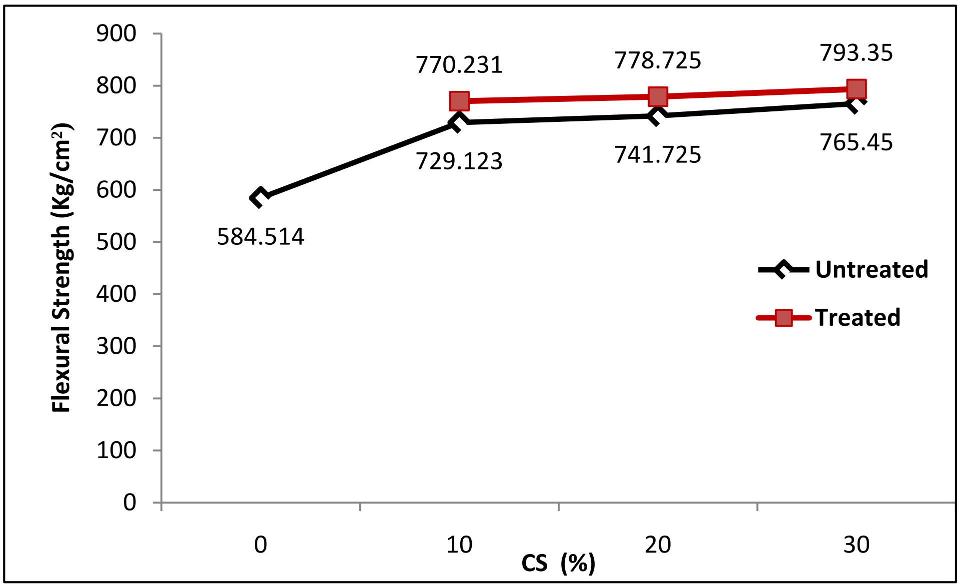


Figure 1. Effect of CS concentration on Flexural Strength of PA6/CS composites

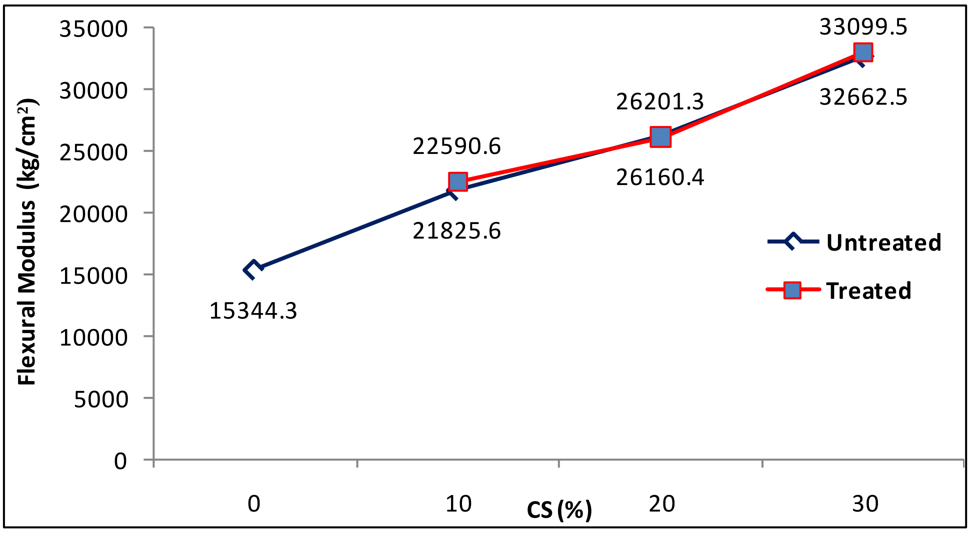


Figure 2. Effect of CS concentration on Flexural Modulus of PA6/CS composites

B. Tensile Strength

Figure 3 shows the effect of SCA treated and untreated CS on tensile strength at yield of PA6/CS composites. As CS content increases tensile strength decreases slightly, but not drastically. This reduction may be due to low interfacial bonding between filler and matrix. This is because, as CS loading increases the interfacial area also increases, worsening the interfacial bonding between filler and matrix. With treated or modified CS, tensile strength increases about 10% compare to composites of untreated CS. This is due to increased interfacial adhesion between CS and matrix Nylon 6 by treatment with aminosilane coupling agent.

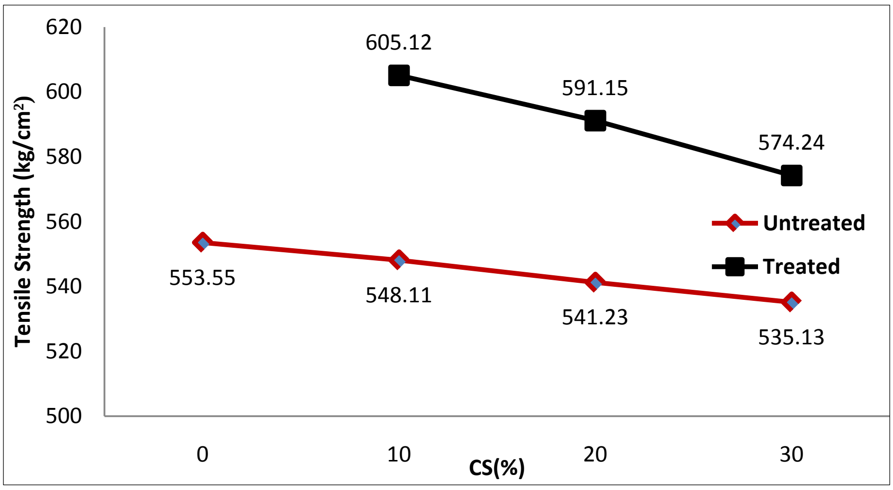


Figure 3. Effect of CS concentration on Tensile Strength of PA6/CS composites

C. Impact Strength

As shown in figure 4, Izod impact strength is reduced drastically with increase in CS content. With untreated case, it reduces up to 50% of virgin Nylon 6. CS content reduces ability of matrix material to absorb energy on sudden load. Here, reduction in impact strength is due to reduction in effective cross section of matrix and increase in stress concentration. There may be poor stress transfer between matrix PA6 and filler CS particles. Lack of interfacial adhesion between filler and matrix may also be the reason. But treatment of CS with SCA increases the impact strength about 15-20% which is still lesser than virgin Nylon 6.

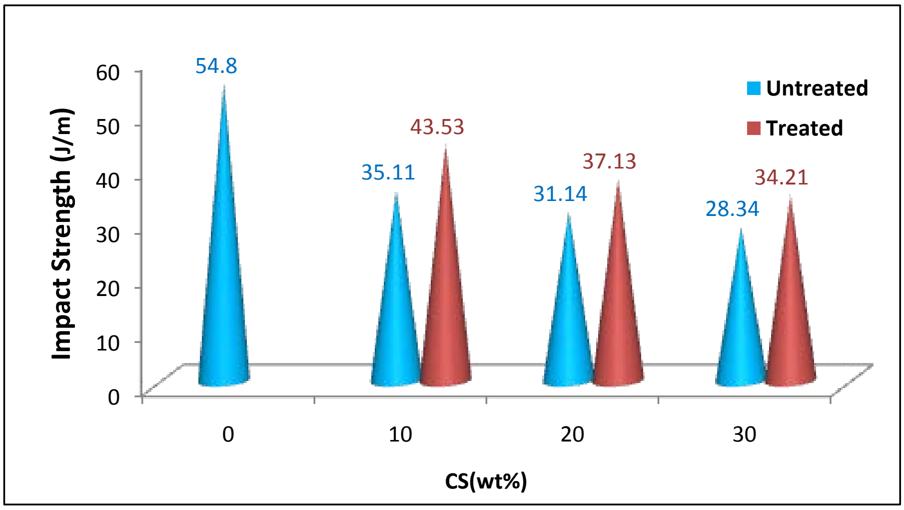
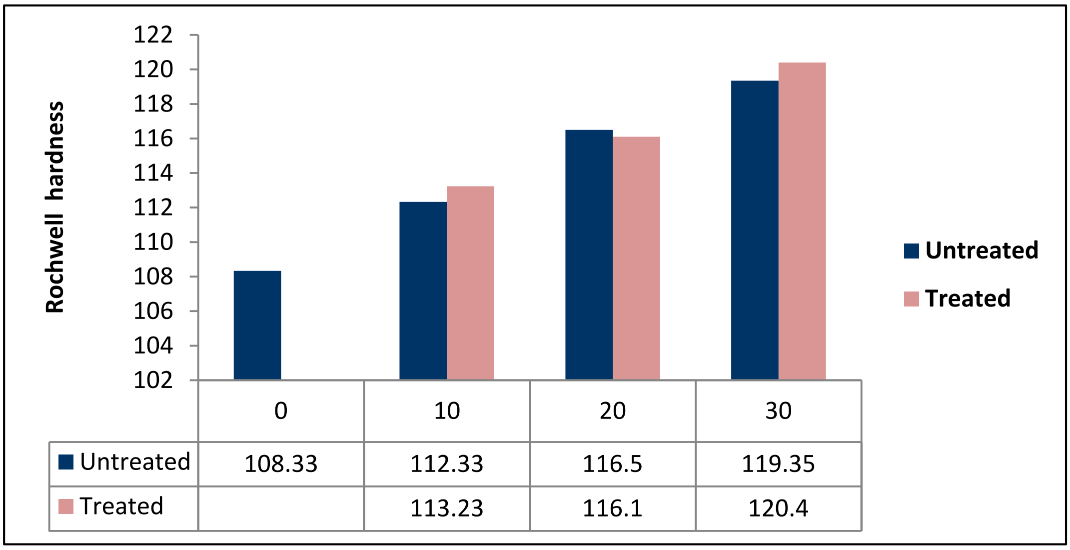


Figure 4. Effect of CS concentration on Izod Impact Strength of PA6/CS composites

D. Rockwell Hardness

Figure 5 shows that as CS concentration increases Rockwell hardness of PA6-CS composites also increases. Hardness is the measure of the modulus of elasticity. With increase in filler concentration modulus of composites also increases which results in increase in hardness value of composite. Increase in hardness is also due to the hard and rigid nature of Cenosphere particle. CS has hardness value of 5-6 Moh scale, far better among mineral fillers, which imparts this property. There is no major effect of coupling agent on the Rockwell hardness value.



	0	10	20	30
Untreated	108.33	112.33	116.5	119.35
Treated		113.23	116.1	120.4

Figure 5. Effect of CS concentration on Rockwell Hardness of PA6/CS composites

E. Abrasion Resistance

Figure 6 shows the removal of material in mg on abrasion with increase in CS concentration in PA6/CS composites. Wight loss in mg decreases with increase in CS content. This indicates abrasion resistance of PA6/CS composite increases with increase in CS content. This is due to the hard and rigid nature of Cenosphere. Hardness of Cenosphere is 5-6 Moh scale which renders composites abrasion resistant. About 20% increase in abrasion resistance is found on every 10% increase in Cenosphere content. There is no major effect of coupling agent on abrasion resistance of silane treated composites compared to untreated composites.

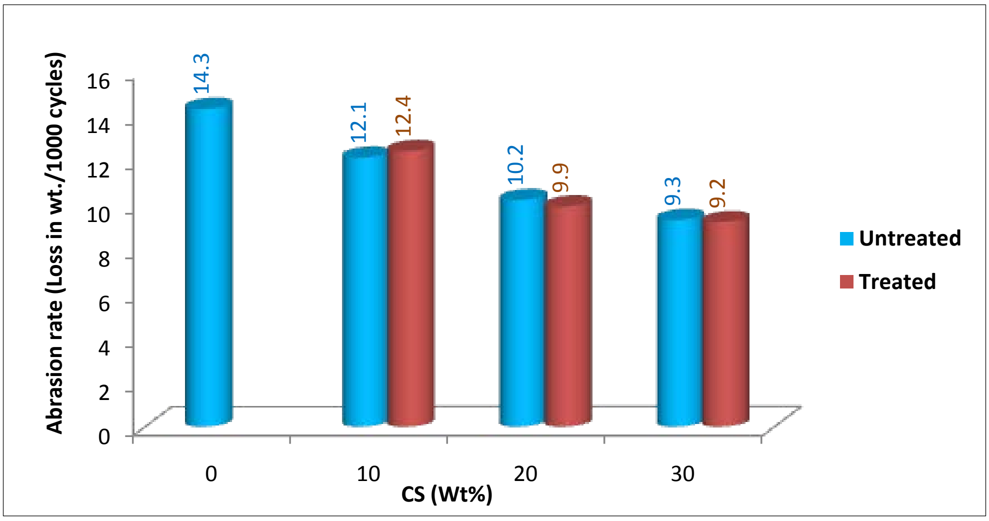


Figure 6. Effect of CS concentration on Abrasion Resistance of PA6/CS composites

IV. CONCLUSION

- With SCA treated CS, composites shows higher flexural strength than untreated CS composites due to increased interfacial adhesion between CS and matrix Nylon 6.
- Tensile strength decreases marginally with increased CS content but treated composites shows higher tensile strength than untreated composites die to increased interfacial adhesion by SCA.
- Impact strength of composites decreases drastically up to 50% of virgin Nylon 6 at 30% CS content. But SCA treated composites show increase in impact strength about 20% compared to untreated PA6/CS composites.
- Hardness and abrasion resistance of unmodified PA6/CS composites increases with CS content due to inherent hardness of Cenosphere. There is no or marginal effect of coupling agent on hardness and abrasion resistance.
- Amino Silane Coupling Agent treated CS can be used to enhance mechanical properties of PA6/CS composites.

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