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Influence of boric anhydride reinforcement on mechanical properties and abrasive wear of nylon 6

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Abstract

To investigate the influence of boric anhydride (B2O3) on mechanical properties of nylon 6, tensile, hardness and impact test were carried out. Average tensile strength along with percentage elongation and modulus of elasticity has been calculated and plotted in this paper. Rockwell hardness and Izod impact tests were carried out to identify the hardness and toughness of materials. Boric anhydride used in this research was limited to less than 10 wt% of nylon 6 matrix material and was 2 wt%, 4 wt% and 8 wt%. Abrasion resistance of pure nylon 6 and composites was measured as weight loss due to abrasion. For the characterization of nylon 6 and its composites, Thermo-gravimetric analysis (TGA) and x-ray diffraction (XRD) were carried out. In results, it was found that the boric anhydride reinforcement increased the tensile strength and abrasion resistance when used up to 2 wt% in the nylon 6 matrix. Hardness found to be continually increased as the boric anhydride reinforcement percentage increased. The crystallinity of nylon 6 was little affected due to fillers and was found minimum for the 4 wt% boric anhydride reinforced nylon 6 composites. It was also found that the elastic modulus, Rockwell hardness and tensile strength, abrasion resistance show good correlations which are discussed in the discussion section.

1. Introduction

Polymers are having vast applications from polythene carry bags, bottles, pipes, toothbrushes, car bodies, intake manifolds, laptop and desktop computer parts, mobile phone parts, electrical and electronic instruments cabinets, medical implant materials and many more. Polymer products have become an essential part of our day to day life. Thermoplastic polymers can be reused by melting again which makes it more cost-effective material. Nylon 6 is one of the subtypes of nylon thermoplastic polymer family and have pleasant mechanical properties. Many fibers and particles were used to further improve the mechanical properties of polymers. For example, 6% aluminium oxide (Al2O3) micro-particles improved the mechanical properties of nylon 66 and 25% of iron micro-particles improved the mechanical and electrical properties of polystyrene [1, 2]. Silicon carbide micro-particles improved the hardness of epoxy thermoset material while Silica nanoparticles improved the compressive modulus of nylon 11 [3, 4]. Calcium oxide (CaO) nanoparticles in 0.5 wt% improved the tensile strength of nylon 6 while Silicon oxide (SiO2) nanoparticles of 2 wt% improved tensile strength, hardness and thermal stability of nylon 6 [5, 6]. Aluminium oxide nanoparticles of 3 wt% improved tensile strength and glass transition temperature while 5% nano clay particles improved tensile and flexural strength of nylon 6 [7, 8]. One research indicates that the 1 wt% carbon nanotubes improved tensile strength, tensile modulus, and hardness of nylon 6 thermoplastic [9]. Pristine α-zirconium phosphate nanoplatelets in 5 wt% increased tensile modulus and 1 wt% carbon nanotubes increased the tensile strength of nylon 6 [10, 11]. One research article indicates that the 30 vol% woven carbon fibers improved the bending strength of epoxy and according to other research article 30 wt% carbon fibers increased the tensile strength and reduced the coefficient of friction of polyether ether ketone [12, 13]. The hardness values of Styrene Butadiene Rubber and High-Impact Polystyrene (HIPS) blends
increased with an increase in the ratio of HIPS while abrasive wear behaviour varied at different blends percentage [14].

Fibers and particle reinforcements are generally advantageous in reducing friction coefficient and wear rate in dry conditions rubbing with the smooth surface while in the case of abrasive wear, these reinforcements generally increase it. One research stated that the carbon, graphite, molybdenum disulfide (MoS$_2$), polytetrafluoroethylene (PTFE) and short glass fibers increased the abrasive wear of polymer [15]. One study revealed that the 10 wt% of h-BN resulted in minimum specific wear rate of polyether ketone [16] while 3 wt% of neodymium oxide addition enhanced the micro hardness by 17% and resulted in lower abrasion [17].

2D materials generally have higher elastic properties when used in small amount in comparison to the corresponding bulk quantity. And because of that, the mechanical properties of 2D materials have been found decreasing with increasing content of it [18]. Boric anhydride is one of the oxides of boron and is 2D material similar to graphene. It is found in the amorphous form and has white and glassy solid appearance. It can be crystallized by the annealing process. The crystalline form of boric anhydride composed of BO$_3$ triangles. Boric anhydride is a lamellar solid lubricant like graphite and hexagonal boron nitride (h-BN) 2D materials. Boric anhydride is used by many researchers to improve the mechanical properties of materials. For example, 5 wt% of boric anhydride improved micro-hardness and strength of hydroxyapatite (Ca$_{10}$(PO$_4$)$_6$(OH)$_$_2$) which is used in human hard tissue implants [19]. In one research, 10 mol% of boric anhydride improved bending strength and Rockwell hardness of diamond composite [20]. While the mechanical properties of the phosphate-based glass fibers continuously increased with increasing boric anhydride content [21].

In this paper, the effect of boric anhydride on mechanical properties, abrasion resistance and crystallinity of nylon 6 is discussed. Fabrication of nylon 6 composites with morphology of boric anhydride by scanning electron microscopy (SEM) is also discussed in the experimental methods section. In the discussion section, results of the experiments are discussed with the comparative studies of different test results.

### 2. Experimental methods

#### 2.1. Material selection

Nylon 6 was selected as matrix material with 99% purity, 3–8 mm granule size, molecular weight 10,000 g mol$^{-1}$ and density 1.13–1.15 g cm$^{-3}$. Boric anhydride micro-particles with the purity of 99.9%, average particle size (APS) <40 $\mu$m, molecular weight 69.62 g mol$^{-1}$ and density 2.46 g cm$^{-3}$ were selected as reinforcement. High purity research-grade nylon 6 and boric anhydride were imported from nano research elements, USA. Boric anhydride micro-particles were ball milled and average particle size found after milling was <40 $\mu$m. Below table 1 represents the particle size distribution according to ASTM. 99.6% boric anhydride particles passed through 325 mesh while 88.2% particles passed through 400 mesh.

| Mesh no, ASTM | Mass retained (gram) | Cumulative % retained | Cumulative % passed | Particle size ($\mu$m) |
|---------------|----------------------|-----------------------|---------------------|----------------------|
| 270           | 0                    | 0%                    | 100%                | 55                   |
| 325           | 4                    | 0.4%                  | 99.6%               | 44                   |
| 400           | 118                  | 11.8%                 | 88.2%               | 37                   |

Figure 1 represents the morphology and particle size distribution of boric anhydride observed by SEM. Boric anhydride particles were found near to the round shape as shown in figure 1(b).

Figure 1(a) represents SEM observation of the particles at x170 magnification in which shapes of the particles were not identified. In figure 1(b), shapes of boric anhydride particles were identified at x550 magnification and of nearly to round appearance. Also, average particle size can be identified in figures 1(b) and (c). In figure 1(c), only major diameter particles were measured to identify the upper limit of particles and as shown they were averagely 40 $\mu$m in size and rest were small particles. Powder SEM characterization was carried out at PD Patel Institute of Applied Sciences, CHARUSAT, Changa, Gujarat, India.

Fourier-transform infrared spectroscopy (FTIR) confirms the composition of boric anhydride and is shown in figure 2. FTIR spectrum reveals the composition of any material which may be in the form of either solid, liquid or gas. FTIR spectra pics observed at 3217, 2261, 1454, 1194, 804 and 547 cm$^{-1}$ due to B-O bonding attribution to boric anhydride (B$_2$O$_3$). FTIR analysis was carried out at Parul University, Vadodara, Gujarat, India.

#### 2.2. Sample preparation

Twin-screw extruder with wide kneading block, high speed torque ZV 20 model, M/S specific engineering & automats was selected for the compounding of nylon 6–boric anhydride composite granules. Twin-screw is...
having the advantage of proper compounding of materials compared to single-screw extruder. Extrusion process at a low speed of 264 rpm was carried out for proper compounding of materials. Generally, wide kneading block and low rpm gives enough residence time and good dispersion quality [22]. Before compounding nylon 6 was dried in an oven at 70 °C for 6 h to remove moisture from the granules. After drying, nylon 6 and boric

Figure 1. SEM observation of boric anhydride micro-particles (a) magnification x170 (b) magnification x550 (c) particle size distribution.

Figure 2. FTIR spectrum of boric anhydride.
anhydride were fed into the twin-screw extruder with the help of hopper. Barrel temperatures were set to 190, 200, 220, 230 and 240 °C for zone 1, 2, 3, 4 and 5 respectively and die temperature was set at 230 °C during the process [23]. A water bath was used to cool the continuous hot semi-solid filament of the composite. After a water bath, the semi-solid filament of nylon 6-boric anhydride was converted into an almost solid form which converted into pellets or grains again using pelletizer. 2, 4 and 8 wt% boric anhydride used to fabricate different nylon 6 composites. The reinforcement percentage was limited to less than 10 wt% to analyze the effect of small wt% of boric anhydride on the mechanical behavior, abrasive weight loss and characterization of nylon 6. After the extrusion process, pellets were dried again at 70 °C for 5 h for injection molding of specimens.

Figure 3(a) represents imported granules of pure nylon 6 and figure 3(b) represents 2 wt% boric anhydride reinforced nylon 6 composite granules after the extrusion process. Compounding of composite granules and injection molding of specimens were done at room temperatures at CIPET, Ahmedabad. Figure 4 represents SEM images of nylon 6 composites at x100, x200 and x400 magnifications. SEM images shows the well dispersed particles of boric anhydride in nylon 6 matrix as well as it shows the temperature effect of extrusion and injection molding process on the composite material samples as dark spots in figures 4(b) and (c).

2.3. Mechanical testing

The H50KL Tinius Olsen L-series, 5KN-50KN force measurement capacity universal tensile testing machine was used to perform the tensile strength test of nylon 6 and its composites. Tensile properties such as strength, elongation, modulus of elasticity and maximum force carried before fracture were measured at a speed of 50 mm min⁻¹ on UTM. Tests were carried out according to the ASTM D 638 and Type-1 sample specifications were used to carry out the test.

To measure the hardness of materials, Rockwell hardness tester, make Saroj Engineering with maximum load capacity 60 kg. was used. Rockwell hardness tester can measure the hardness of a material by indentation and comparing major load with a minor one. It can show the results directly on the dial which makes it more convenient. All tests were performed according to ASTM D 785 for hardness measurement. All results were measured on R-scale and 100 mm * 3 mm were the sample specifications for the test. The steel ball of diameter 12.7 mm was used for indentation.

Impact tests were performed on the Ceast Resil impactor, 0 to 25 J capacity Izod tester. A pivoted arm raised to a height and then released to hit a sample at its notch to break it. The energy absorbed by the sample is then calculated. Impact test is used to measure the toughness of materials which is an essential mechanical property. In this test, results were shown in energy lost per unit of thickness in J/m at the notch of a sample. Impact tests were performed according to the standard ASTM D 256. Dimensions of specimens were taken according to the standard and were 63.5 * 12.7 * 3.2 mm. Rockwell hardness and Izod impact tests were performed at Central Institute of Plastics Engineering and Technology-(CIPET), Ahmedabad. All mechanical tests were carried out at room temperature.

2.4. Abrasion resistance testing

The abrasion resistance tests were carried out on TABER abrasion resistance tester at Central Institute of Plastics Engineering and Technology-(CIPET), Ahmedabad. 100 mm * 3 mm were the specifications of disc samples for nylon 6 and its composites. Abrasion resistance for 1000 cycles and with 1 kg (9.81 N) load was measured for all the samples of nylon 6 and nylon 6 composites at room temperature. H-18 abrasive wheel was used to perform the abrasive action on materials. All tests of abrasion resistance were performed according to ASTM D 1044.
3. Results

3.1. Tensile strength, rockwell hardness and impact toughness

Tensile strength, modulus of elasticity and elongation were measured of 5 samples and the average of the results were plotted for nylon 6 and nylon 6–boric anhydride composite. Figure 5(a) represents average tensile strength in MPa and was noted 52.7, 62.9, 55 and 43.9 MPa for nylon 6 and 2, 4 and 8 wt% boric anhydride reinforced nylon 6 composites, respectively. The highest tensile strength found for 2 wt% boric anhydride reinforcement and was 19.35% more than pure nylon 6. Tensile strength increased up to 4 wt% reinforcement thereafter it decreased and found lowest at 8 wt% boric anhydride reinforcement and was noted 16.7% less than pure nylon 6.

Modulus of elasticity was noted 420, 569, 621 and 743 MPa for nylon 6 and 2, 4, 8 wt% boric anhydride reinforced composites respectively as shown in figure 5(b). Measurement of Modulus of elasticity is necessary because it measures the material’s resistance when deformed elastically. Modulus of elasticity continuously increased and found maximum for 8 wt% boric anhydride reinforcement and was noted 76.9% more than pure nylon 6.

Elongation of materials were noted in % and were 266, 327, 204 and 197% for 0, 2, 4 and 8 wt% boric anhydride reinforced nylon 6 composites respectively as shown in figure 5(c). Maximum percentage elongation was noticed for 2 wt% boric anhydride reinforcement and was 22.93% more than pure nylon 6. After 2 wt% reinforcement it decreased and found minimum at 8 wt% boric anhydride reinforcement and was 25.94% less than pure nylon 6. The maximum extension was 306 mm and the maximum load carried was 2140 N for nylon 6 before fracture. For 2 wt% boric anhydride reinforced nylon 6 composite, maximum extension was 376 mm and load-carrying capacity was 2387 N. For 4 and 8 wt% reinforced nylon 6 composites, maximum extensions were 234 and 226 mm and maximum load-carrying capacities were 1975 and 1770 N respectively.

Rockwell hardness results on R-scale is represented in figure 6. Average hardness after five sample experimentations of nylon 6 was 116 and of nylon 6 composites, the average Rockwell hardness was 119, 120 and 123 for 2, 4 and 8 wt% boric anhydride reinforced nylon 6 composites, respectively. Rockwell hardness continuously increased and found maximum for the 8 wt% boric anhydride reinforcement and was 6.03% more than pure nylon 6.

Impact strength results of nylon 6 and composites are shown in figure 7. The average impact strength for nylon 6 was 48.4 J m\(^{-1}\) and for 2, 4 and 8 wt% boric anhydride nylon 6 composites, it was 32.4, 45.7 and 30 J m\(^{-1}\), respectively. Impact strength results show an uneven pattern and found maximum for pure nylon 6.
while the minimum for 8 wt% boric anhydride reinforcement. Impact strength found 38.02% less for 8 wt% boric anhydride reinforced nylon 6 composites compared to pure nylon 6.

3.2. Abrasion resistance
Weight loss in percentage during abrasion test of nylon 6 and composites is shown in figure 8. In the case of nylon 6, total 0.14% weight loss was observed after 1000 cycles rotation of H-18 abrasive wheel. For 2 wt% boric anhydride reinforced nylon 6 composite it was observed 0.12% and for 4 and 8 wt% reinforcement, weight loss was observed 0.7 and 1.2% respectively.

Weight loss due to abrasive action of H-18 abrasive wheel was found minimum for the 2 wt% reinforcement and was noted 14.29% less than pure nylon 6. After 2 wt% reinforcement it increased and found maximum at 8 wt% boric anhydride reinforcement and was 8.6 times more than pure nylon 6.
3.3. XRD and TGA

XRD study was carried out on Bruker D2 Phaser x-ray diffractometer at a scanning speed of 1° per minute with Cu-Ka radiation (λ = 0.154 nm) at room temperature. Tests were carried out in scanning range of 10° to 50° two thetas. Figure 9 represents the XRD plots of nylon 6 and nylon 6 composites. x-axis represents 2 theta values and the y-axis represents counts per second (CPS) or intensity of peaks. Pure nylon 6 peaks are ranging from 21–24 two thetas as represented in figure 8 with black color and having the intensity of about 90–100 CPS. Nylon 6 composites are having the same peak range as nylon 6 with different intensities but with additional peaks ranging from 13–15 and 27–29 two thetas due to boric anhydride reinforcement [24]. The intensity of peak for 8 wt% reinforcement in 13–15 two thetas was about 260 CPS while for the range 27–29 two thetas it was about 450 CPS. Nylon 6 can take two crystallographic forms, α and ϒ. The crystal density and fusion heat of ϒ pseudo-hexagonal form are less than α monoclinic form at weaker interactions between the chains. The α and ϒ XRD peaks of nylon 6 lies at approximately 24 and 21–22 two thetas, respectively. XRD peaks can be identified easily by higher intensities up to 4 wt% boric anhydride reinforced nylon 6 composites majorly near 21–22 two thetas. For 8 wt% boric anhydride reinforced nylon 6 composite XRD peaks are found near 24 two thetas with very low intensity and broader hump probably because of change in the crystallinity of nylon 6. [25–27].

As nylon 6 is a semi-crystalline polymer material, its degree of crystallinity (DOC) was found 54.72% and rest are the amorphous content. For 2 wt% boric anhydride reinforcement, DOC was found 41.54% and for 4 and 8 wt% reinforcement it found 30.68 and 44.75% respectively. As shown in figure 10, nylon 6’s crystallinity was little affected by the boric anhydride reinforcement [28]. Lowest crystallinity found for 4 wt% boric anhydride
reinforcement and it decreased the crystallinity of pure nylon 6 about 24.04%. XRD analysis was carried out at PD Patel Institute of Applied Sciences, CHARUSAT. Figure 11 represents TGA plots for nylon 6 and nylon 6 composites. Perkin Elmer, pyris-1-TGA apparatus with 1000°C temperature capacity was used to carry out thermo-gravimetric analysis and samples were taken in the form of granules. Heat was given from 50°C to 850°C at 20 °C min⁻¹ rate. The x-axis represents temperature (°C) while the y-axis represents the sample weight (%). At 506 °C nylon 6 curve reached its almost lowest position as shown by black color in figure 10. 2 and 4 wt% boric anhydride reinforced nylon 6 composites curves reached their lowest position at approx. 520 °C as shown by red and blue colors respectively. While 8 wt% boric anhydride-nylon 6 composites reached its lowest position at 555 °C and is shown by pink color in the figure. At 570 °C nylon 6 was totally degraded and the sample weight was observed 0 mg (0%). In the case of 2, 4 and 8 wt% boric anhydride-nylon 6 composites 0.6%, 1.12% and 2.41% residuals were still there at 850 °C which ensures the reinforcement presence in nylon 6 composites. As the sample weight was measured 5.061 mg for 2 wt%, 7.027 mg for 4 wt% and 5.176 mg for 8 wt% boric anhydride reinforced nylon 6 composites for TGA testing it can be understood that in this small quantity of sample weight reinforcement was still there which is enough evidence of reinforcement presence in the composites [29]. TGA analysis of materials were carried out at CIPET, Ahmedabad at room temperatures.
4. Discussion

Due to moisture absorption from the atmosphere, tensile strength of nylon 6 was found decreased while elongation found increased compare to dry nylon 6. Moisture acts as a plasticizer in nylon therefore it increased the elongation up to several percentages and reduced the tensile strength of nylon 6 [30, 31]. By closely analysing the plots of elastic modulus and Rockwell hardness, figures 5(b) and 6, it was found that the modulus of elasticity and Rockwell hardness are having the same pattern. As the elastic modulus of material increased, the hardness also followed [32]. Regression analysis of modulus of elasticity and Rockwell hardness of materials shows the value of multiple-R 0.998 which indicates a strong linear relationship between them. Standard error found was only 9.98 which shows that the regression model is quite good.

Abrasion resistance and tensile strength of the materials also show good correlations. As the tensile strength decreased, abrasion resistance of materials followed. It means that, as the tensile strength of nylon 6 and composites decreased, the weight loss due to abrasion increased. Regression analysis of tensile strength and abrasion resistance shows good correlation and shown in table 2(b). Multiple-R value of regression was 0.8 which indicates a strong correlation between both while the standard error is just 0.38 which shows a precision of regression model.

Most of the mechanical tests are done by gradual loading of specimens while impact test include fracture by application of sudden load at the notch. Fracture of materials depends on weak spots, imperfection of material, and cracks in the material and because of these reasons it is hard to interpret the exact reasons behind increasing or decreasing the impact strength [33]. By comparing and analysing tensile & impact strength data, it was found that the both properties show inverse co-relation [34, 35]. As the tensile strength of 2 wt% boric anhydride reinforced nylon 6 increased, the impact strength of the same was found decreased. In table 2(c), regression analysis results of up to 4 wt% filler reinforcement is shown. The multiple-R value found 0.9982 which indicates strong correlations while standard error was found only 0.44 which indicates precision of the regression model.

Overall, tensile strength found maximum for 2 wt% boric anhydride reinforced nylon 6 and hardness found maximum for 8 wt% boric anhydride reinforced nylon 6. Impact strength found maximum for pure nylon 6. Boric anhydride reinforcement was not found effective in enhancing the impact strength of nylon 6. Weight loss due to abrasion found minimum in 2 wt% boric anhydride reinforcement thereafter it increased linearly, and crystallinity found minimum in 4 wt% boric anhydride reinforcement.

![Figure 11. TGA plots of nylon 6 and nylon 6–boric anhydride composites.](image)

| Table 2. Regression analysis (a) elastic modulus versus Rockwell hardness (b) tensile strength versus abrasion resistance (c) tensile strength versus impact strength. |
|----------------|----------------|----------------|
| (a) Regression statistics | (b) Regression statistics | (c) Regression statistics |
| Multiple R | 0.9981 | Multiple R | 0.8041 | Multiple R | 0.9982 |
| R Square | 0.9962 | R Square | 0.6465 | R Square | 0.9965 |
| Adjusted R Square | 0.9944 | Adjusted R Square | 0.4698 | Adjusted R Square | 0.9931 |
| Standard Error | 9.9764 | Standard Error | 0.3754 | Standard Error | 0.4413 |
| Observations | 4 | Observations | 4 | Observations | 3 |
5. Conclusion

Boric anhydride reinforcement increased the tensile strength and abrasion resistance when used up to 2 wt% in the nylon 6 matrix. Hardness found to be continually increased while impact strength was found decreased by the boric anhydride reinforcement. The crystallinity of nylon 6 was little affected due to fillers. Elastic modulus and Rockwell hardness found linearly co-relative to each other. Similarly, tensile strength and abrasion resistance were also found co-relative. Thermal stability was improved of nylon 6 composites due to fillers. In summary, Boric anhydride reinforcement in nylon 6 matrix improved few of the mechanical properties, abrasion resistance in few amount and thermal stability of nylon 6.

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