International Journal of Sciences:
Basic and Applied Research
(IJSBAR)

ISSN 2307-4531
(Print & Online)

https://gssrr.org/index.php/JournalOfBasicAndApplied/index

International Journal of
Sciences:
Basic and Applied
Research

ISSN 2307-4531
(Print & Online)

Published by:

Mechanical and Antibacterial Properties of Polyester/Chitosan/Ag Nanocomposite

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Abstract

This projects development of a new type of lightweight polymer-based nanocomposite material, utilizing polyester resin as the matrix and incorporating Ag nanoparticles and chitosan as filler materials. Polyester based composites products sometimes suffers from mechanical failure, weather adaption and microbial attack. Our findings demonstrate significant advancements in both mechanical and antibacterial properties of these nanocomposites. Chitosan and Silver (Ag) nanoparticles reinforced polyester based nanocomposites were prepared where chitosan were synthesized from shrimp shell and Ag nanoparticles were synthesized by chemical reduction method. Different percentage of (wt%) chitosan were loaded in polyester matrix and 2.5wt% chitosan loading was found to be optimized. In the optimized polyester-chitosan system different percentage (2.5, 5, 7.5) wt% of Ag nanoparticles were dispersed as reinforcement to fabricate polyester/chitosan/Ag nanocomposites. The resultant polyester based nanocomposites were (i) polyester/chitosan/2.5wt% Ag, (ii) polyester/chitosan/5wt% Ag, (iii) polyester/chitosan/7.5wt% Ag which were compared with the blank polyester resin sheet and optimized polyester/chitosan composite for the investigation of mechanical (tensile strength, young's modulus, flexural strength, flexural strain and hardness), water absorption and antimicrobial activities. Among the entire composite and nanocomposites the polyester/chitosan/2.5wt%Ag showed the best results in an average with respect to mechanical, water absorption and antimicrobial activities.

Keywords: Nanoparticles (Ag); Nanocomposite; Chitosan; Polyester resin; Antimicrobial.

Received: 6/27/2024
Accepted: 8/27/2024
Published: 9/5/2024

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1. Introduction

Polyester based composite materials have an increasing demand in a wide range of applied field due to the high strength to weight ratio [1,2]. Many researchers have taken it as a real challenge to modify the existing polyester matrix composites and develop new types of composites by incorporating fillers and reinforcements such as calcium carbonate, talc organics, wood flour organics, cotton lintels and fiberglass that could show better mechanical performance with long term sustainability [3,5]. Polyester based composites products push a huge application such as sheet molding compound, bulk molding compound, household applications, constructional appliances, electronics, packaging and biomedical applications etc [6,7] but it sometimes suffers from mechanical failure, weather adaption and microbial attack which leads to our research interest to improve these properties by incorporating functional biopolymer and nanoparticles [8,9] that could provide much more mechanical performance and prohibition of bacterial attack [10,11].In this article, we report here the fabrication of polyester based nanocomposites with the dispersion of functional biopolymer (chitosan) synthesized from shrimp shell and silver (Ag) nanoparticles as reinforcements. As chitosan (Cs) is a functional reinforcement which could neutralize the extra hydroxy functional groups of polyester resin by forming cross linking networks between the polymer chains, and increased area to volume ratio, three dimensional stability and high antibacterial properties of silver (Ag) nanoparticles could improve the mechanical ,water absorption and antimicrobial activities.

2. Materials and Methods

2.1. Materials

Sodium hydroxide (Merck, India, 98%), Hydrochloric acid (Merck, India, 98%), Polyester resin (Lucky Acrylic and Fiber, Bangladesh, 98%), Methyl ethyl ketone peroxide (MEKV) (Lucky Acrylic and Fiber, Bangladesh, 98%), Silver nitrate (Merck, India, 98%), Tri sodium citrate (Sigma Aldrich, Germany, 99%), Dimethyl sulfoxide (DMSO) (Merck, India, 99%). All the chemicals and solvents were used as received without any further treatment.

2.2. Synthesis and Fabrication

2.2.1. Synthesis of Chitosan

Shrimp shell was collected from local market then it was cleaned, dried and grinded into small size. Chitosan was extracted from shrimp shell in three steps namely; demineralization, deproteinization and deacetylation References [10,12,13]. Demineralization was done by 4% concentrated HCl with a solid to solvent ratio of 1:13 (w/v) for about 6 hours. Deproteinization was performed by using 4% Sodium hydroxide (NaOH) weight was observed. The dried sample is known as chitin and light brown in color. Deacetylation was done by 40% NaOH solution with a solid solvent ratio of 1:16 (w/v) for about 8 hours with continuous stirring. The acetyl group must be removed in this process, and then the solution was filtered, neutralized and dried to get the white color chitosan.

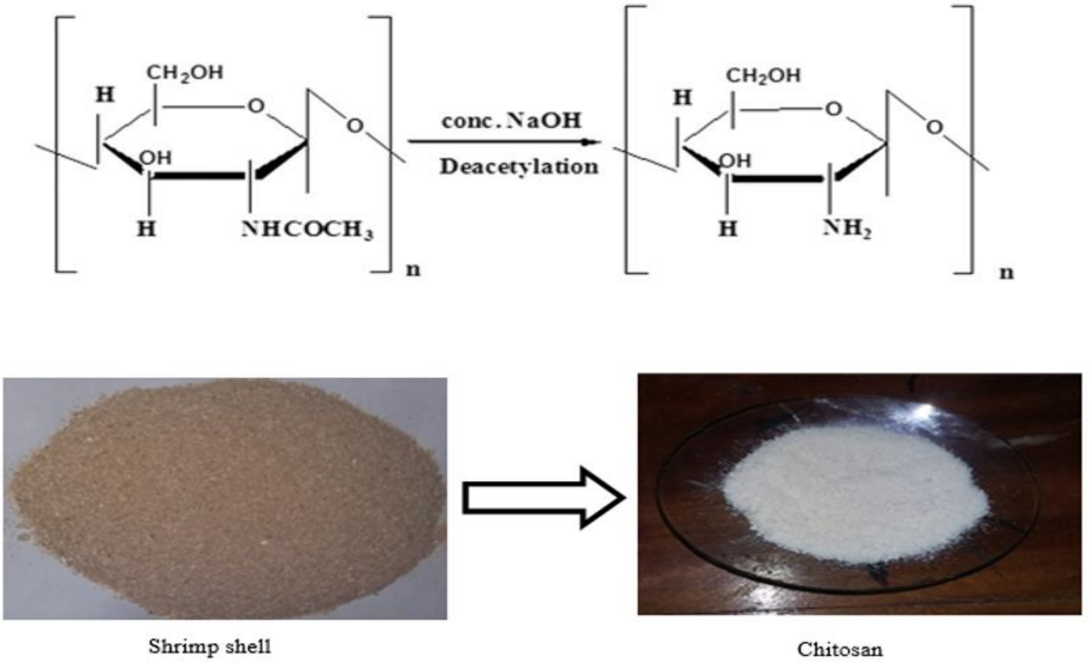


Figure 1: Synthesized chitosan from shrimp shell

2.2.2. Synthesis of Silver Nanoparticles (Ag)

Silver nanoparticles were synthesized by the reduction of silver salt ($AgNO_3$) with tri sodium citrate (TSC) ($C_6H_5O_7Na_3$). All solutions of the reacting materials were prepared in distilled water. In a beaker 300 mL of 0.09 M $AgNO_3$ was prepared and heated to boil with continuous stirring, the beaker was covered with watch glass. When the temperature reached to about 80^{o}C then remove the watch glass and 30mL 0.9M tri-Sodium citrate (TSC) was added to the solution drop by drop with the help of a dropper. Here, TSC acts as both stabilizing and reducing agent. Solution was heated until the color was evident (pale brown). This is the visual identification of successful synthesis of silver nanoparticles [14,15]. Then the solution was cooled at room temperature and stirring was continuing. The solution was filtered and washed repeatedly with distilled water. The filtered powder was then taken in to the oven for drying at 70^{o}C for 4 hours. The different parameter like relative quantities and concentration of reagents, heating time duration and temperature are the factors which affects the successful synthesis of silver nanoparticles.

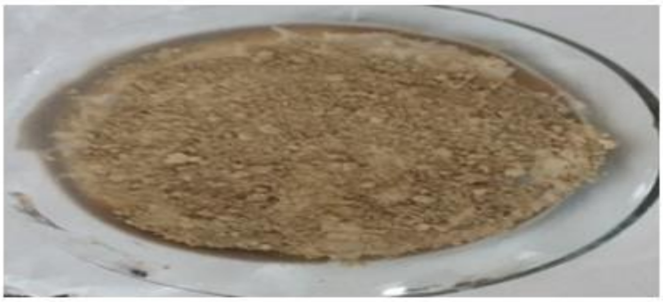


Figure 2: Synthesized silver (Ag) nanoparticles

2.2.3. Fabrication of Composites

(a) Blank polyester resin sheet, (b) polyester/ chitosan composite, (c) polyester/chitosan/Ag nanocomposites were fabricated by solution casting method [16]. The mold for all composites fabricated was same and all samples were fabricated in similar approximate thickness of 2mm.

To fabricate (a) blank polyester resin sheet, 40gm. Of polyester resin was poured into a cup and the hardener Methyl Ethyl Ketone Peroxide (MEKP) **(**1% in accordance with polyester**)** was added to the polyester by a dropper carefully. Both polyester and hardener was mixed thoroughly with a glass rod for 5-6 minutes for homogeneous mixing. The mixture was cast onto a flat mold and outgassed overnight. It takes about 24 hours to completely cure the mixture. After fully cured, the blank epoxy resinsheet was collected diligently.

To fabricate (b) polyester/chitosan composite previously optimized 2.5wt% chitosan was loaded in polyester resin before adding hardener and mixed carefully for about 30 minutes in order to uniform dispersion, Similarly in case of (c) polyester/chitosan/Ag nanocomposite, previously optimized 2.5wt% chitosan and different percentage(2.5,5,7.5)wt% of Ag nanoparticles were loaded before adding hardener and mixed carefully for uniform distribution and then by following the rest of the fabrication process all the composite samples were fabricated and collected carefully for further investigation.

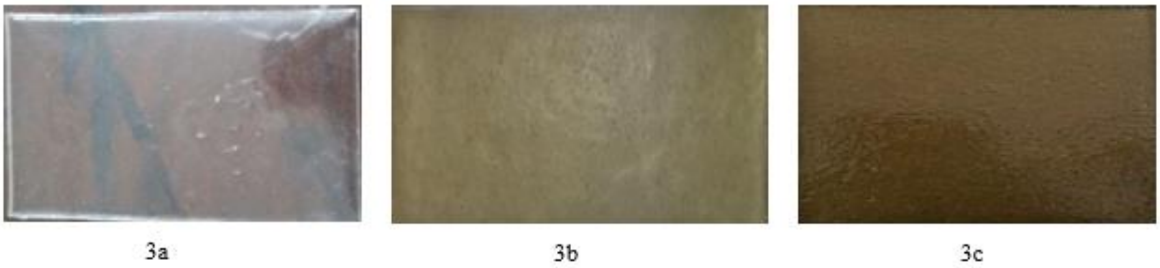

3a 3b 3c

Figure 3: Fabricated (3a) blank polyester resin sheet, (3b) polyester/chitosan composite, (3c) polyester/chitosan/Ag nanocomposite

2.3. Methods

2.3.1. Characterization techniques

The surface morphology of the nanoparticles was observed with HITACHI S-3400N scanning electron microscope (SEM) (operated at 5KV). The average size of the nanoparticles and their size range was calculated from (SEM) this analysis. To examine the bonding and functionalization of chitosan, The FT-IR spectra of chitosan was recorded on a PERKIN-ELMER FRONTIER FTIR/MIR spectrometer. The wavelength was measured from region 225-4000 cm^{-1} at room temperature. To confirm the synthesis of Ag nanoparticles, UV-visible spectrum of the aqueous medium containing nanoparticles samples was measured with a UV-1601 UV–visible spectrophotometer (Shimamdzu Co., Japan). In the absorption mode spectra were taken from 200 to 800 nm wavelengths. Mechanical properties such as tensile strength, flexural strength, young’s modulus, flexural strain of the composites and nanocomposites were measured by computer controlled electromechanical universal testing machine (UTM) (Jinan precision testing equipment co. Ltd, model WOW-50). Parameters of the UTM for tensile properties measurements were, extension range = 100 mm, gauge length = 25 mm, speed = 2 mm/min, approach speed = 2 mm/min, pre-load = 0. For flexural properties measurement, specimens were designed in 120mm length and 20mm in width with around 2mm thickness. Hardness of the composites and nanocomposites was investigated by using Leeb H-1000 Portable Hardness Tester where the test was repeated 20 times for each sample for obtaining the average value and to avoid errors. Water absorption properties of all samples were measured by pouring into water for 24 hours and the specimens were designed in 3cm in length and 1cm in width. Microbial test was done by spectrophotometric (Turbidimetric) analysis where the *E.coli* bacterial population was detected by calculating turbidity or optical density (cloudiness of a suspension) of a broth culture. Increased turbidity in a culture is the indicator of bacterial growth and biomass, as turbidity is directly proportional to the number of bacterial cells. All samples were dissolved in dimethyl sulfoxide (DMSO), supplemented into nutrient broth (NB) medium, an antibiotic namely Ciprofloxacin was also supplemented as standard to compare the microbial activities of the samples. Optical density was measured at every six hours' time period with UV-visible spectrophotometer at 660nm.

3. Results and Discussion

3.1. Characterization of Nanoparticles

3.1.1. SEM Analysis of Ag Nanoparticles

From the SEM image (**figure 4a**) of synthesized Ag nanoparticles, it has observed that the Ag nanoparticles are triangle, tetragon, pentagon and hexagon structures i.e., crystalline in nature at 50,000X magnification. Most of the particles can be identified by nanometer scale, which are defined as primary particles. Diameter of primary particles of Ag nanoparticles is in the range of 35-60nm and the average particle size is 45nm respectively. Typical nanoparticles are agglomerates of several primary particles. The agglomerates are defined as secondary particles. They are formed when primary particles are held together by weak surface forces (soft agglomerates) such as van der walls or capillary forces or by strong chemical bonds (hard agglomerates) [17,18].

3.1.2. UV-visible Spectroscopy of Ag Nanoparticles

To confirm the synthesis of silver (Ag) nanoparticles, UV-visible spectrum of the aqueous medium containing nanoparticles samples were measured. Figure (4b) shows the absorbance of the sample (Ag) in nano range at room temperature. The spectra exhibit between 200nm and 800nm range were studied. It has observed that UV-visible spectra of Ag (figure 4b) shows peak at 435nm wavelength, with absorbance of 0.321 (<1), which means that it exhibits good absorbance at UV region.

The average size of Ag nanoparticles is estimated to be 35nm-60nm using a scanning electron microscope (SEM); this was also confirmed by UV-Vis spectroscopy [19].

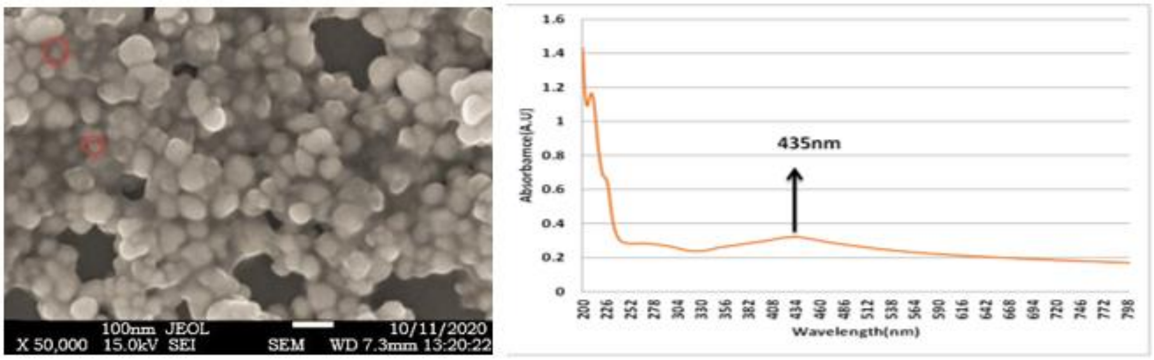


Figure 4: (4a) SEM image of synthesized silver (Ag) nanoparticles, (4b) UV-visible absorption spectrum of Ag nanoparticles

Table 1: Nanoparticles size range and average size

Name of Nanoparticles	Size range, nm (Calculated from SEM image)	Average Size, nm
Silver (Ag)	35-60 nm	45 nm

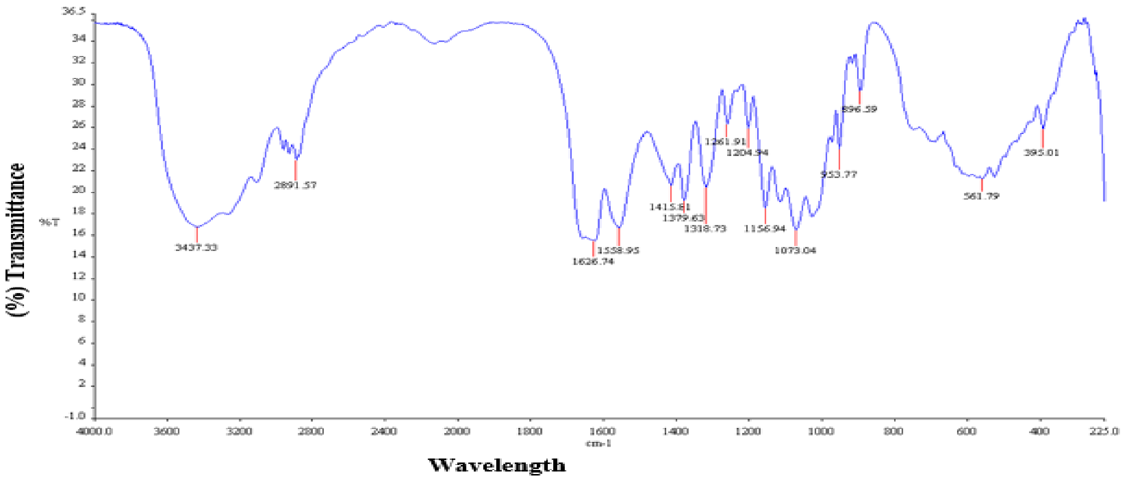


Figure 5: FTIR spectrum for chitosan extracted from shrimp shell

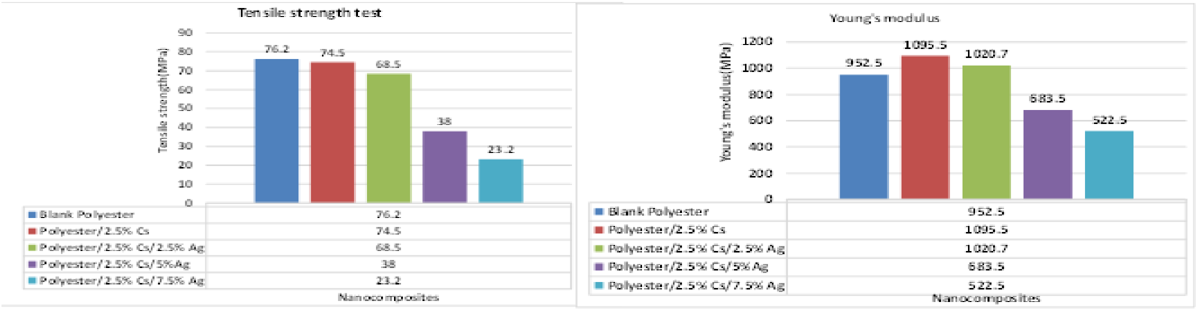
3.2. FTIR Analysis of Chitosan (Cs)

The FTIR test was performed to confirm the identity of chitosan and to assess the functional groups present in the Chitosan (**Figure 5**). We can observe that a strong band in the region 3437.33cm^{-1} corresponds to the stretching vibration −OH, which is superimposed onto −NH stretching vibrations and broadening of band is due to the inner molecular hydrogen bonds of polysaccharides [20].The absorption band at 1156.94cm^{-1} can be assigned to asymmetric stretching of the C-O-C Bridge. The bands at around 1073.04cm^{-1} correspond to C-0 stretching. Fourier Transform Infrared Spectrum shows characteristic picks of carbonyl at 1626.74cm^{-1} and amide at 3437.33cm^{-1} which ensure that this FTIR is of Chitosan.

3.3. Characterization of Fabricated Composite and Nanocomposite

3.3.1. Tensile Strength and Young's Modulus

The tensile strength (**figure 6a**) of blank polyester resin sheet, polyester/2.5wt% chitosan(Cs) composite, polyester/2.5wt%chitosan/2.5wt%Ag, polyester/2.5wt%chitosan/5wt%Ag and polyester/2.5wt%chitosan/ 7.5wt%Ag nanocomposites have found to be 76.2MPa, 74.2MPa, 68.5MPa, 38MPa and 23.2MPa respectively. It has found that loading of optimized 2.5wt% chitosan slightly decreased the tensile strength, where further decrease were observed due to the dispersion of (2.5wt%Ag, 5wt%Ag and 7.5wt%Ag) nanoparticles along with 2.5wt% chitosan. All samples were taken of similar thickness around 2mm. It is difficult to improve all the properties of a composite with the dispersion of nanoparticles, some properties like tensile strength might fall due to certain reasons. One of the prominent reasons is the agglomeration of the nanoparticles. This agglomeration hinders proper dispersion of the nanoparticles into the polymer matrix system resulting weak interfacial adhesion between the matrix and the nanoparticles. Plasticizing effect of the nanoparticles is another effective reason for the decrease of tensile strength. Thus, the decrease in tensile strength can be attributed to the physical properties of this filler and interaction of this filler with the polymer matrix [21,22].Where the young's modulus (figure 6b) has found to be 952.5 MPa, 1095.5MPa, 1020.7MPa, 683.5MPa and 522.5MPa, respectively. It is clear that loading of 2.5wt%chitosan, 2.5wt%Ag nanoparticles along with 2.5wt%chitosan increased the young's modulus but as the percentage of nanoparticles were increased, young modulus of nanocomposites were decreased. As chitosan has -OH functional group which interact with the polymer chain and form bond. The nanoparticles are highly stiff in nature. This stiffness nature causes the enhancement of the young's modulus of the matrices. Lower content of nanoparticles produces large interfacial areas as they have high weight to volume ratio, helping them to interact easily with the matrix material by forming bonds. This ceramic nature bonding does not allow slip under stress. The mobility to slip makes it more difficult for the inorganic filler to absorb stress and increase the young's modulus [23].



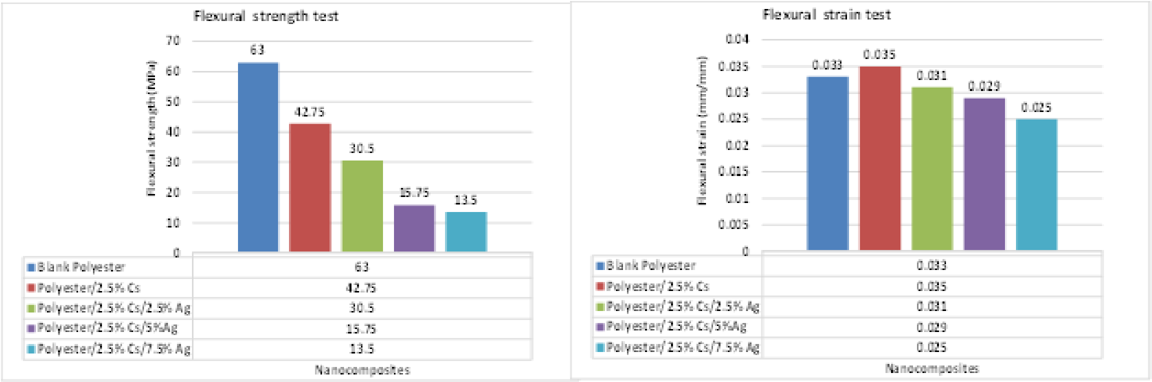
6a

6b

Figure 6: Comparison of tensile strength (6a) and young's modulus (6b) of blank polyester resin sheet, polyester/2.5wt% chitosan, polyester/2.5wt% chitosan/2.5wt% Ag, polyester/2.5wt% chitosan/5wt% Ag and polyester/2.5wt% chitosan/7.5wt% Ag nanocomposites

3.3.2. Flexural Strength and Flexural Strain

The flexural strength (**figure 7a**) measured as 63MPa, 42.75MPa, 30.7MPa, 15.75MPa and 13.5MPa respectively. It has found that loading of optimized amount of chitosan, dispersion of different percentage of nanoparticles along with chitosan in polyester resin decrease the flexural strength. The reasons behind this occurrence are nonhomogeneous distribution of nanoparticles and chitosan within the matrix, agglomeration of the nanoparticles and high-volume percent of nanoparticles [24-25] where the flexural strain (**figure 7b**) of blank polyester resin sheet and the nanocomposites has estimated as 0.033, 0.035, 0.031, 0.029 and 0.025 respectively. The increase of flexural strain for 2.5wt% chitosan loading may be for the plasticizing effect on the polyester resin. Otherwise, the result is very mush expected. As the tensile strength and flexural strength decreases it is easy to understand that the strain property will also decrease for the morphological reasons. The deformation tendency of the composite will decrease due to the presence of ceramic natured nanoparticles, resulting less probability of slip displacement of the composite grains. Newly formed bonds and crosslinks are another reason for the hindrance of deformation, which results lowering of the strain property of the nanoparticle reinforced composites [10,25].



7a

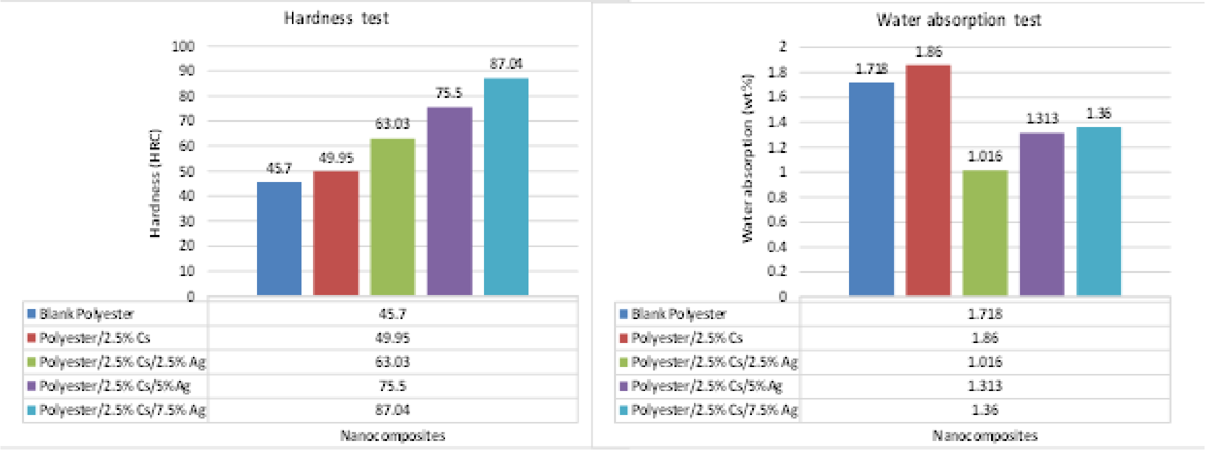
7b

Figure 7: Comparison flexural strength (7a) and flexural strain (7b) of blank polyester resin sheet, polyester/2.5wt% chitosan, polyester/2.5wt% chitosan/2.5wt% Ag, polyester/2.5wt% chitosan/5wt% Ag and polyester/2.5wt% chitosan/7.5wt% Ag nanocomposites

3.3.3. Hardness and Water Absorption

The hardness (**figure 8a**) has measured as 45.7, 49.95, 63.03, 75.5 and 87.04 (HRC) respectively. The loading of chitosan in polyester resin increased the hardness of blank polyester resin sheet due to more cross linking between polymer chains as functional group of chitosan helps to make bonds. The nanocomposites also break the continuity of the polyester ring in order to form new bond with them. This increases the plasticizer content of polyester resin, making it harder than the blank one. The three-dimensional stability of the nanoparticles and because of the nanoparticles penetrating into the nano voids of the composites, the ultimate result is the increase of the hardness of nanocomposites [26].

Where, the wt% water absorption (**figure 8b**) has measured 1.718%, 1.86%, 1.016%, 1.313% and 1.36% respectively. Polyester resin is usually hydrophobic in nature and the water absorption is very poor but the reason of increasing its water absorption content with the loading of chitosan is the inherent hydrophilic nature of -OH and -NH_2 in the chitosan that increases the hydrophilicity of the matrix than the blank polyester resin sheet. The dispersion of silver (Ag) nanoparticles decreases the water absorption percentage due to the hydrophobic nature of Ag nanoparticles. Also, the nanoparticles occupy most of the empty sites of polyester resin but with the increase in nanoparticle contents, agglomeration of nanoparticles occurs that weakens the interface between the nanoparticles and matrix which creates enough pores to absorb water where 2.5wt% of Ag nanoparticles found to be optimized [27].



8a 8b

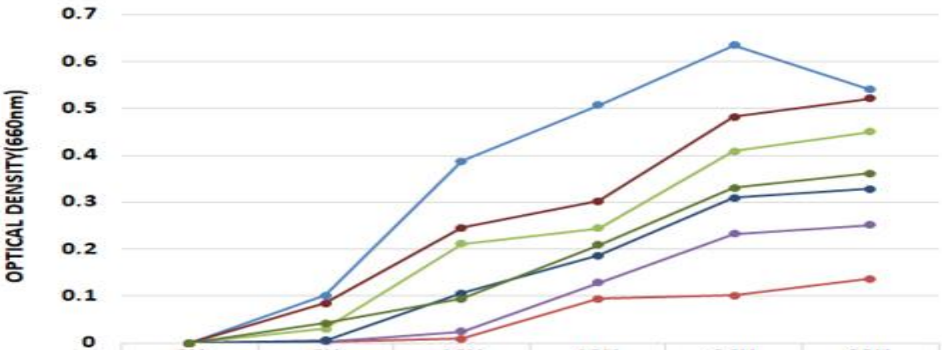
Figure 8: Comparison of hardness (8a) and water absorption (8b) of blank polyester resin sheet

3.3.4. Microbial Activities

Microbial activities of chitosan (Cs), silver (Ag) nanoparticles, Cs+Ag, optimized polyester/chitosan composite and optimized polyester/chitosan/Ag nanocomposite compositions were observed in nutrient broth medium. All the samples were also compared with the nutrient broth (NB) control and standard antibiotic to understand the antibacterial activities where the bacterial (*E.coli*) growth in nutrient broth medium was measured (figure 9) in terms of optical densities as a function of time at 600nm wavelength up to 30 hours (H) periodically.

It has observed that the absorption of NB control is increased up to 24H with a value of .635 and then started to decrease at 30H with a value of .541 which indicates the bacterial growth substantially. In the case of antibiotic, Cs, Ag nanoparticles and Cs+Ag sample, the bacterial growth is slower than the NB control. The lowest value and highest value for antibiotic are 0.003(6H) and 0.137(30H) respectively and continues to increase, the lowest and highest absorption value for Cs, Ag and Cs+Ag are 0.031(6H)& 0.451(30H), 0.003(6H)& 0.252(30H) and 0.006(6H)&0.329(30H) respectively which clearly indicates that chitosan and Silver nanoparticles has a certain degree of antibacterial activities that prohibit the bacterial cell growth. Again, the lowest and highest absorption value in terms of optical density of optimized polyester (PR)/chitosan (Cs) composite and optimized polyester (PR)/chitosan (Cs)/Ag nanocomposite are 0.085(6H) & 0.521(30H) and 0.042(6H) & 0.362(30H) respectively.

It is clear that chitosan (Cs) and Ag nanoparticles both have antibacterial properties but Ag nanoparticles have strong tendency to prohibit bacterial cell growth [28], which is further observed in polyester/chitosan composite and polyester/chitosan/Ag nanocomposite. Thus, incorporation of chitosan and silver nanoparticles in polyester resin nanocomposites can be suggested for best microbial prohibition and longtime sustainability [29].



	0H	6H	12H	18H	24H	30H
NB Control	0	0.102	0.388	0.507	0.635	0.541
NB+ANTIBIOTIC	0	0.003	0.009	0.095	0.101	0.137
CS	0	0.031	0.212	0.245	0.409	0.451
Ag	0	0.003	0.025	0.129	0.233	0.252
CS+Ag	0	0.006	0.106	0.187	0.31	0.329
CS+PR	0	0.085	0.246	0.303	0.482	0.521
CS+Ag+PR	0	0.042	0.094	0.209	0.331	0.362

TIME(HOURS)

Figure 9: Comparison of antibacterial properties of control (NB), antibiotic (standard), and chitosan (Cs), silver (Ag), Cs + Ag, chitosan (Cs) + polyester resin(PR), Cs +Ag + PR in nutrient broth medium (NB)

4. Conclusions

Polyester based chitosan and silver (Ag) nanoparticles reinforced polymer nanocomposites were prepared. The chitosan were synthesized from the shrimp shell and Ag nanoparticles were synthesized by chemical reduction method. Different percentage wt% of chitosan and Ag nanoparticles was loaded and optimized. The optimized loading of reinforcement were 2.5 wt% for chitosan and nanoparticles. The resultant polyester based chitosan, and chitosan & Ag reinforced composite and nanocomposites were (i) polyester/2.5wt% chitosan, (ii) polyester/2.5wt% chitosan/2.5wt% Ag, (iii) polyester/2.5wt% chitosan/5wt% Ag and (iv) polyester/2.5wt% chitosan/7.5wt% Ag which were compared with the blank polyester resin sheet for the characterization of mechanical (tensile strength, young’s modulus, flexural strength, flexural strain and hardness), water absorption, and antimicrobial activities. Among the four composite and nanocomposites the polyester/2.5wt% chitosan/2.5wt% Ag showed the best results in an average with respect to the mechanical, water absorption and antimicrobial activities.

Acknowledgements

The authors are very much grateful to “Rajshahi University Research Project”, Rajshahi University, for their financial support and also grateful to the Atomic Energy Commission (AEC), Bangladesh, and Pharmacy Department of Rajshahi University for giving its support in its laboratory facilities.

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