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Synthesis and characterization of nanoparticles derived from chitosan-based biopolymer; their photocatalytic and anti-termite potential

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Chitosan has been obtained from Labeo rohita fish scales by protonation. The nature of chitosan depends upon the stages of the compound extraction process. Chitosan-based nanoparticles (Ag/chitosan) were synthesized; they were characterized by FTIR and UV-Visible spectroscopy. FTIR analysis confirmed the successful formation of Ag/chitosan hybrid. The synthesized hybrids were subjected to the photocatalytic activity evaluation and anti-termite activity. The results have shown excellent anti-termite activity. The effect of concentration of acetic acid and chitosan was also studied on the composite material for anti-termite activity. Ag/chitosan concentration was more effective than acetic acid concentration. Methylene blue as a standard dye was used to study its photodegradation. The synthesized hybrid material showed much enhanced photodegradation of dye as compared to individual components.

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1. Introduction

Chitin $(C_8H_{13}O_5)_n$ is a naturally occurring mucopolysaccharide that is a hard, white, nitrogenous, and inelastic compound. It is a product of the fishery industry [1]; it's naturally occurring yield is greater than 1000 tons for each year and around 70 % of this originates from marine species. Chitin is the major constituent in the shells of scavengers, for example, shrimp, crab, and lobster, fish scales and is found in exoskeletons of bugs and mollusks [2]. Chitin-based constituents are used for the treatment of industrial pollutants, silver thiosulphate multiplexes, and actinides. Chitin can be used in the form of fibers and films. They are non-allergic, antibacterial and wetness controlling. In the papermaking procedure, chitin fibers are used as binders [3]. Chitin also increases the speed of wound healing. Chitin is generally used for in-activating` enzymes and entire cells. Enzyme inactivation is used in the food industry. Due to its biodegradability, physiological inertness, nontoxicity, antibacterial properties, hydrophilicity, an affinity for proteins, and gel-forming properties chitin has utilization in many areas other than food [4].

Chitosan is obtained from the chitin. Chitosan was introduced in the medical area, afterword's the interest of industrialists and researchers were developed to produce novel therapeutic and effective systems based on chitosan [5, 6]. The flexibility of chitosan is due to its amino groups as the reactive site. The amino groups and their derivatives are known as an amino polysaccharide. Structurally it is composed of (copolymer) N-Acetyl-D-glucosamine and D-

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glucosamine units with one amino (NH_2) group and two hydroxyls (OH) groups in each repeating glycosidic units [7]. At low pH, chitosan acts as a polycationic type, because of protonation of NH_2, which increases its stability. Furthermore, the cationic property of chitosan can be reversed via sulfonation to introduce an anionic character, with water-soluble, better paste fluidity, high water reducing ratio, and anticoagulant property [8]. The cationic property of chitosan can be turned around through sulfonation to present anionic character. Chitosan was used normally in medical fields such as for slimming, tissue engineering, and wound dressing, however by time, chitosan has been an important candidate for drug-carrying [9]. Chitosan has been from a nontoxic sustainable material and react as effective and compatible biomaterial. The other biological uses of chitosan are mucoadhesion, transfection, permeation enhancement, in situ gelations, colon targeting. All the above mentioned uses are endorsed to its amine group. Adhesive nature of chitosan makes it a prominent candidate in dentistry, orthopedics, and ophthalmology [10].

The polymer mixing by common biopolymers (cellulose, starch, chitosan, chitin, etc.) [11-14] with manufactured polymers (polyvinyl alcohols, polystyrene and polylactic corrosive) results the development of new composite materials with unusual properties and applications in various types of areas, as reported by different scientists. Chitosan Si-hybrids were produced using 3-(Glycidoxypropyl)trimethoxy silane which reacts with NH_2 of chitosan [1]. Main aspects of chemical significance are composition, surface area, porosity, and structure of the composites, along with molecular size, degree of acetylation and chemical modification of chitosan [15].

The chitosan nanocomposites were used to eliminate heavy metal ions from wastewater because chitosan is inactive on the surface of the magnetic noncomplex which bind with them. The contact between chitosan and metal ions is reversible, that means those ions can be eliminated from chitosan in weak acidic media with the help of ultrasound radiation [16]. Multiwall carbon/chitosan composite presented a great efficiency for the expulsion of the metal ions from the aqueous solution. The results proposed that this multiwall carbon/chitosan composite could be used in environmental applications [17, 18].

Various productions such as plastics, paper and textile produce waste substance that contains a huge volume of organic dyes [19-21]. When that waste composites are released to the streams and river, they can dangerous for aquatic life and also harm for human life through food. The water treatment processes (adsorption, sedimentation, coagulation, etc.) are now switched by advanced oxidation procedures [22]. TiO_2–Chitosan is proficient of removing the pollutant through the combined effect of photodegradation–adsorption. The reactive –OH, $–NH_2$ and metal oxide substances in the prepared TiO_2–Chitosan responsible for the photodegradation–adsorption [23]. The results show that catalyst plays a vital role in determining the efficiency of methylene orange attributable to both adsorption and photodegradation process. Thus, TiO_2–Chitosan catalyst must be applied to attain maximum absorption of light for the excitation of surface TiO_2. Improved photodegradation effect was selected in place of adsorption effect [24].

Termites are the trickiest pests in plants and buildings. There are 2800 defined types of termites and 185 significant to be pests. They damage the wooden structures having cost three billion dollars annually. Termites are known to destroy agriculture and forestry crops cause damage in buildings. For monitoring termites, artificial termiticides have been recycled for a long time. Continuous use of artificial termiticides for the crop as well as soil treatment has been permitted for the present time because of the absence of any effective replacement.The immobilized embedded in the photoactive chitosan was characterized by Fourier transform infrared spectroscopy, ultraviolet, and fluorescence measurements. The surface structures and biological activity were examined by scanning electron microscopy, atomic force microscope, electron spectroscopy for chemical analysis, and bioassays [25]. It is reported that chitosan or its derivatives are good preservatives in many areas, such as food, medicine, textile and wood. In order to develop novel environmentally friendly wood preservatives, the termite resistance properties of wood treated with chitosan copper complex and modified chitosan copper complex respectively are investigated. Synthetic insecticides are the major method used to avoid termite attack on wood. However, the determined use of chemical termiticides is at current ecological concern and need to research for plant compounds as a substitute for termite resistor [26].

Owing to the growing trend of investigations on nanoparticles [27-29], current studies were made to synthesize nanoparticle derived from chitosan-based biopolymer, characterize them and test their photocatalytic activity and anti-termite potential.

2. Materials and Methods

2.1. Materials

Labeo rohita fish scales were obtained from Kolar fish farm. Laboratory quality distilled water prepared from steam distillation was used for the purpose. HCl, NaOH and methylene blue were purchased from Merck.

2.2. Methods

2.2.1. Extraction of Chitosan

The extraction of chitosan from crude fish scales was carried out in three stages.

i. Demineralization for deduction of calcium carbonate/phosphate.
ii. Deprotonation for the exclusion of protein and then chitin was changed into chitosan
iii. Deacetylation that mostly remove the acetyl group from the polymers chain of chitin.

2.2.2. Demineralization of fish scales

94.6 g of fish scales were taken in beaker of 1L. Then 500mL of 1M HCl solution was addedslowly to the beaker and this solution was kept for 20 hours at room temperature for the removal of natural calcium carbonate. After 20 hours, scales were washed repeatedly with distilled water until pH7 was attained. Then the scales were dried at room temperature for 24 hours.

2.2.3. Deprotonation of fish scales

For deprotonation of fish scales, the dried scales obtained from first step were dispersed with 500mL of 2.5M NaOH solution. This solution was continuously stirred for 5 hours for the expulsion of protein. After that, the product was filtered and washed with distilled water until pH7 was obtained. Then the product was dried in an oven at 80^oC until a constant weight was obtained. The dried product is now chitin.

2.2.4. Deacetylation of chitin

For deacetylation of chitin that obtained from second step was dispersed with 400 mL of 1.56M NaOH solution. This solution was continuously stirred at 80^oC for 3 hours for the expulsion of acetyl group. After that, the slurry was filtered and washed with distilled water until pH7 was obtained. Then the product was dried in an oven at 60^oC until a constant weight was obtained. The dried product is now chitosan.

Fig. 1 is the flow sheet diagram showing extraction of chitosan.

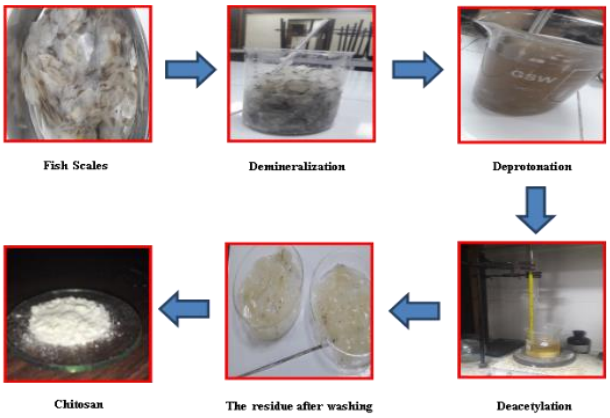

Fig. 1. Flowsheet diagram showing extraction of Chitosan.

2.3. Synthesis of Ag/Chitosan hybrid

The Ag/chitosan hybrid biopolymers were synthesized using wet method. 5g of chitosan was dispersed in 100mL deionized water, to which 5 mL of acetic acid was added. This beaker was marked as beaker A. The mixture in beaker A was continually stirred for 4 hours at 80^{o}C. Then the mixture was allowed to cool at room temperature. 5mL of chitosan solution was taken from beaker A and added it in a beaker in which 0.84g $AgNO_3$ was dissolved in 5mL of deionized water. This beaker was marked as beaker B then 40 mL of methanol was added in this breaker and this beaker was covered with aluminium sheet and was placed it at room temperature for 2 weeks. The solution in beaker B changes its colour from light yellow to yellowish-brown, which indicates the presence of silver particles. The solution was filtered and centrifuged at 3000 rpm for 10 minutes. Finally, the yellowish-brown precipitates obtained were dried in an oven at 80^{o}C until the constant weight was obtained. Similarly, the procedure was repeated to form Ag/chitosan hybrid to examine the effect of concentration of acetic acid and is listed in Table 1.

Table 1. Preparation of Different Ag/Chitosan hybrids.

Sample	**Acetic Acid (Volume)**	**pH**	**Volume of $AgNO_3$**	**pH on adding Methanol**	**pH on adding $AgNO_3$**	**pH changes after 2 week**
R0	5mL	2.76	0mL	0	0	No Change
R1	5mL	2.76	5mL	4.89	4.68	No Change
R2	4mL	2.98	5mL	4.69	4.51	No Change
R3	3mL	3.27	5mL	4.52	4.34	No Change

2.4. Photocatalytic Activity

0.01g of powdered methylene blue was dissolved in 1000mL of distilled water and was stored in dark as stock solution. Then 100mL solution of methylene blue was taken in a separate beaker and 1mL of Ag/chitosan solution from R1 was added. After that, this was kept in dark for 30 minutes. Finally, after every 10 minutes, 10mL of solution was taken out and centrifuged for 2 minutes at 3000 rpm to separate Ag/chitosan hybrid. The UV-Vis spectra were recorded to study photocatalytic degradation of methylene blue. The procedure was repeated for R2 and R3 [30].

2.5. Anti-termite Activity of Chitosan nanoparticles

Simple strips of filter paper were cut in dimension of 6 x 3 square inches. Then added 20 termites into a beaker with filter paper and leave it in lab for 2 weeks.

Weight of filter paper before treating termites = 0.96g

Weight of filter paper after treating termites = 0.87g

Weight loss = 0.96g – 0.87g = 0.09g

2.6. Feed media preparation for termites having Ag/Chitosan

The strips of filter paper were cut of the dimension of 6 x 3 square inches. This filter paper was dipped in Ag/chitosan and kept for 2 hours. Then it was dried in oven at 80^{o}C the paper. Now the filter paper is ready for the termite experiment.

Weight of blank filter paper = 0.90g
Weight of filter paper with chitosan= 0.95g
Weight of chitosan = 0.95g-0.90g = 0.05g
Figure 2 shows the filtter paper coated with Ag/chitosan hybrid.

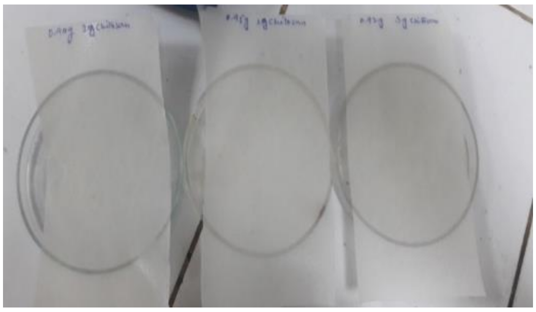

Fig. 2. Filter paper coated with Ag/chitosan hybrid.

2.7. Effectiveness of Ag/Chitosan as Anti-termite

The ability of chitosan nano-particles as anti-termite was determined by the paper weight lose method. Filter paper with a coat of chitosan nano-particles were added into beaker. Twenty termites were added into the beaker and was kept into the lab for 2 weeks. Then the paper was taken out and weighted it for the paper lose measurement.

Initial weight before anti termite activity = W_1 (g)
Final weight after anti termite activity = W_2 (g)
Weight loss of paper = $\frac{w_1 - w_2}{w_1} X\ 100$

Chitosan biopolymer solutions of R1, R2, and R3 were made. These solutions had different concentration of chitosan as described earlier. For 1% solution 1 gram of chitosan biopolymer from R1 solution and 5mL of acetic acid was added in 25mL of distilled water. For a 2% solution, took 2 gram of chitosan biopolymer from R2, 5mL of acetic acid, and added to 25mL of distilled water. For 3% solution took 3 gram of chitosan biopolymer from R3 with 5mL of acetic acid and added 25mL of distilled water.

2.8. Feed media preparation for termites, having acetic acid

The strips of filter paper were cut of the dimension of 6 x 3 square inches. Each filter paper was dipped in Ag/ chitosan and acetic acid and kept for 2 hours. Then it was dried in oven at 80^{o}C. Now the filter paper is ready for the termite experiment.

Weight of blank filter paper = 0.89g
Weight of filter paper with acetic acid= 0.92g
Weight of acetic acid = 0.92g-0.89g = 0.03g
Initial weight before anti termite activity = W1 (g)
Final weight after anti termite activity = W2 (g)
Weight loss of paper = = $\frac{w_1 - w_2}{w_1} X\ 100$

2.9. Effectiveness of Acetic acid as anti-termite

Chitosan biopolymer solutions of R1, R2 and R3 were made. These solutions have different concentrations of acetic acid as described earlier. For 1% solution 1gram of chitosan biopolymer from R1 solution and 1ml of acetic acid was added in 25mL of distilled water. For a

2% solution, 1 gram of chitosan biopolymer was taken from R2 with 2mL of acetic acid, and added in 25mL of distilled water. For 3% solution, 1gram of chitosan biopolymer was taken from R3 with 3mL of acetic acid and was added in 25mL of distilled water. Three pieces of filter paper were taken. Then each paper was dipped into one solution separately. These filter papers were kept in the oven for drying. When the papers were completely dried then weighted on the weighing balance. Above filter papers were placed in beakers. Labelled each beaker as R1, R2 and R3. Now added 20 termites into each beaker and kept these beakers in the dark for 10 days. After 10 days, the filter papers were taken out and noted the weight again.

3. Results and Discussion

3.1. FTIR spectroscopy

FTIR spectra of Chitin, chitosan and Ag/chitosan hybrid were recorded by BRUKER. The spectrum of chitin (Figure 3) shows the peaks at 1653cm^{-1}, 3259cm^{-1},3400cm^{-1} and 2889cm^{-1} respectively that indicates the functional groups having C-O, N-H, O-H and CH_2-CH_3 and respectively.

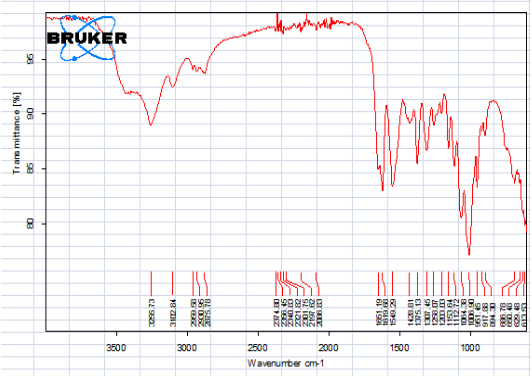

Fig. 3. FTIR spectrum of Chitin.

The spectrum (Figure 4) of chitosan shows the peaks at 3291cm^{-1}, 1028-1066cm^{-1}, 3100cm^{-1}, 1645cm^{-1} and 1423cm^{-1} respectively which indicates the functional groups having N-H, C-O, O-H, C=O and CH_2 respectively.

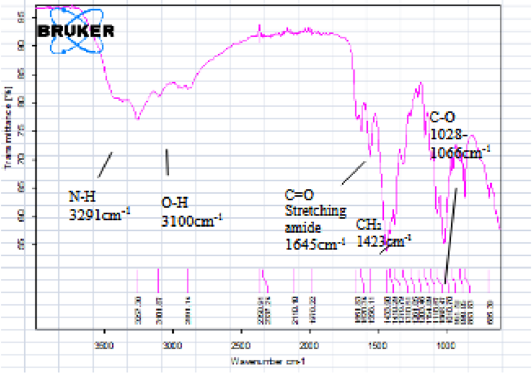

Fig. 4. FTIR spectrum of Chitosan.

The spectrum (Figure 5) of Ag/chitosan hybrid shows the peaks at 3431cm^{-1}, 1653cm^{-1}, 1340cm^{-1}, 1280cm^{-1} and 648cm^{-1}, respectively which indicates the functional groups having O-H, N-H, CH_3 and C-O respectively.

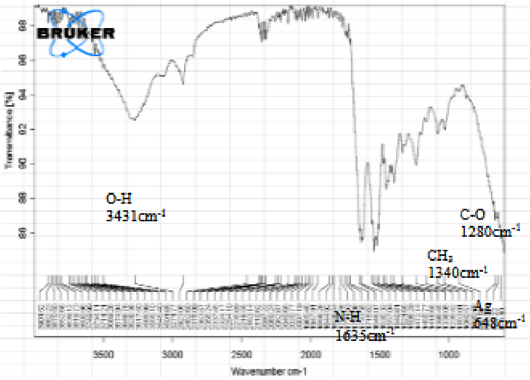

Fig. 5. FTIR spectrum of Ag/Chitosan

3.2. Photocatalytic activity

3.2.1. Photocatalytic activity with 5mL Acetic acid

It was observed that with the passage of time (1 hour 40 minutes) the intensity of absorbance of methylene blue decreases. This result suggests that the decrease in concentration of methylene blue is due to the photodegradation in the presence of sunlight in the intensity 44767 lx. Highest degradation of chitosan with 5 mL acetic acid in sunlight is approximate 93.6 %. The results are displayed in Figure 6.

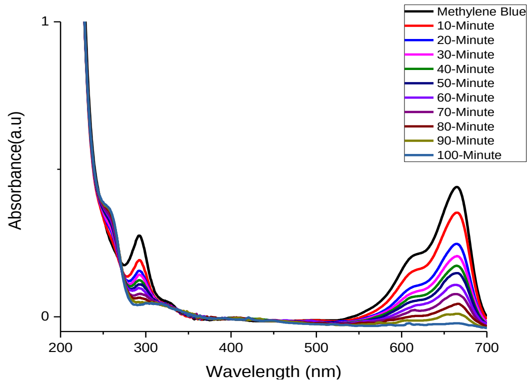

Fig. 6. Photodegradation of chitosan with 5mL acetic acid.

3.2.2. Photocatalytic activity with 4mL Acetic acid

It was observed that after 100 minutes the intensity of absorbance of methylene blue decreases. This result suggests that the decrease in concentration of methylene blue is due to the photodegradation in the presence of sunlight in the intensity 45676 lx. Highest degradation observation for 4 mL solution in sunlight is 92.7 %. The results are displayed in Figure 7.

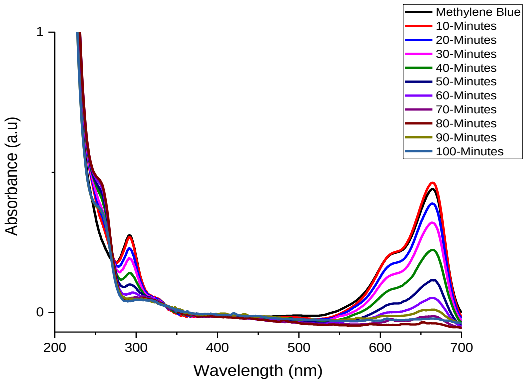

Fig. 7. Photodegradation of chitosan with 4mL Acetic acid.

3.2.3. Photocatalytic activity with 3mL Acetic acid

It was observed that with the passage of time the intensity of absorbance of methylene blue decreases. This result suggests that the decrease in concentration of methylene blue is due to the photodegradation in the presence of sunlight in the intensity 44767 lx. Degradation after 100 minutes was 92.8 %. The results are displayed in Figure 8.

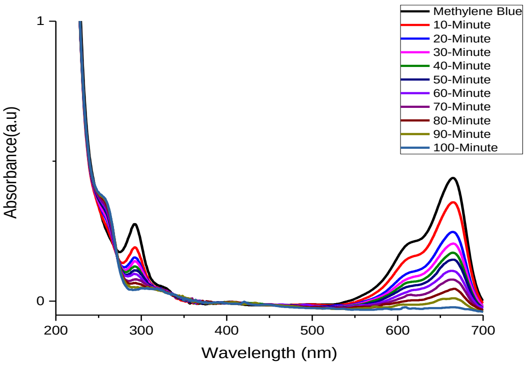

Fig. 8. Photodegradation of chitosan with 3mL Acetic acid.

3.2.4. Photocatalytic Activity of Ag/Chitosan hybrid R1

It was observed that after some time the intensity of absorbance of methylene blue decreases from 0.4 to 0.02. This result suggests that the decrease in concentration of methylene blue is due to the photodegradation in the presence of sunlight in the intensity 44767 lx. Highest photodegradation of methylene blue observed for R1 is 98.7 %. The results are displayed in Figure 9.

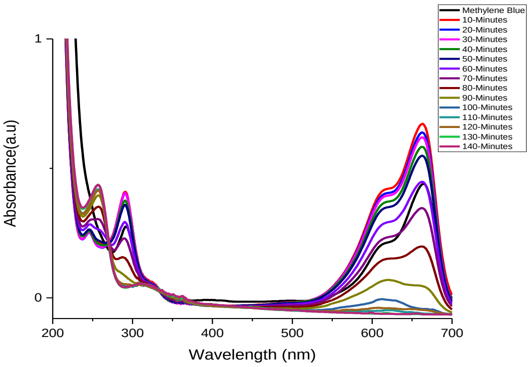

Fig. 9. Photodegradation of Ag/chitosan with 5mL Acetic acid.

3.2.5. Photocatalytic activity of Ag/Chitosan hybrid R2

The spectrum is shown in the following figure 4.8. It was observed that with the passage of time the intensity of absorbance of methylene blue decreases. This result suggests that the decrease in concentration of methylene blue is due to the photo degradation in the presence of sunlight in the intensity 44767 lx. 96.7 % degradation of methylene blue for R2 is observed. The results are displayed in Figure 10.

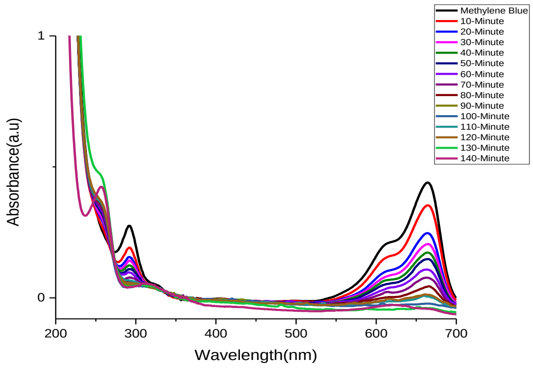

Fig. 10. Photodegradation of Ag/Chitosan with 4mL Acetic acid.

3.2.6. Photocatalytic activity of Ag/Chitosan hybrid R3

It was observed that with the passage of time the intensity of absorbance spectrum of methylene blue decreases. This result suggests that the concentration of methylene blue is decreased due to the photodegradation in the presence of sunlight in the intensity 44767lx. Approximate 95.6 % degradation is observed for R3 solution. The results are displayed in Figure 11.

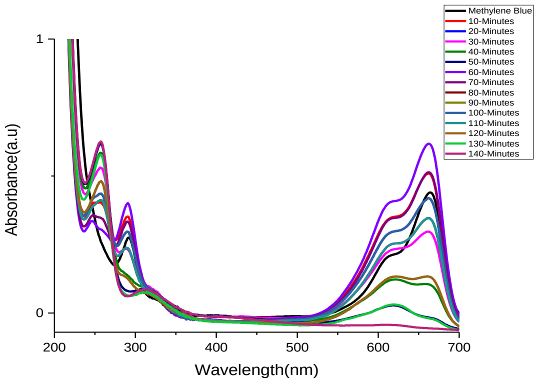

Fig. 11. Photodegradation of Ag/Chitosan with 3mL Acetic acid.

3.3. Effect of Chitosan nanoparticles against termite

Three solutions of different concentrations of Ag/chitosan hybrid were made to check the weight loss activity. At X-axis chitosan concentrations and at Y-axis weight loses % were recorded. Increasing the percentage of Ag/chitosan concentration resulted in decrease of the weight loss % decreased. R1 solution gives 7.57% weight loss. R2 chitosan shows a 5.40% weight reduction rate. R3 chitosan concentration gives 1.44% weight loss. Chitosan concentrations are more effective than Acetic acid concentrations.

Table 2 shows anti-termite activity for various concentrations of Chitosan while Figure 14 displays the weight loss of paper with increasing Chitosan concentration.

Table 2. Anti-termite activity for various concentrations of Chitosan.

Sr. No.	Concentration of chitosan (g)	Weight loss (%)
1	1	6.35
2	2	7.57
3	3	5.70
4	4	1.44

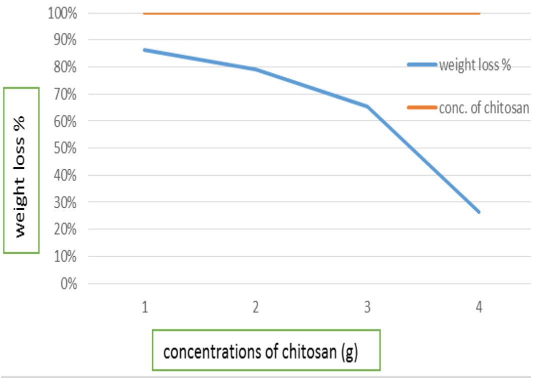

Fig. 12. Weight loss of paper with increasing Chitosan concentration.

3.4. Effect of Acetic Acid concentrations against termite

Three solutions of different concentrations of acetic acid were made to note the weight loss activity. Weight loss of R1, R2, and R3 was 15.94%, 11.04% and 5.97% respectively.

Termite, at a high weight reduction rate, ate filter paper without acetic acid. Without coating weight reduction at a high level, while filter paper coated with acetic acid had less weight reduction. Weight loss level decreased as acetic acid concentration increased. This experiment showed acetic acid could be utilized in the production of paper against termite.

Table 3 displays anti termite activity for various concentrations of acetic acid while Figure 13 shows weight loss of paper with increasing acetic acid concentration.

Table 3. Anti termite activity for various concentrations of Acetic acid.

Sr. No.	Concentration of acetic acid (mL)	Weight loss (%)
1	1	12.96
2	2	15.94
3	3	11.04
4	4	5.97

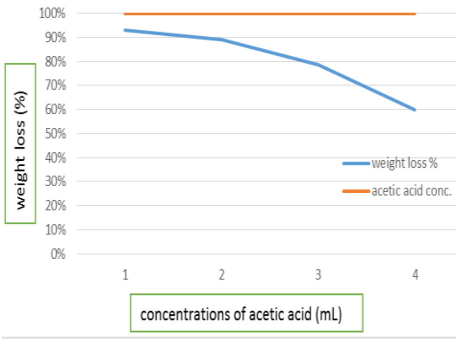

Fig. 13. Weight loss of paper with increasing Acetic acid concentration.

4. Conclusions

Chitin was removed from fish scales (*Labeo Rohita*) by synthetic technique utilizing 2% HCl, 4% NaOH. Chitin was changed over to chitosan by demineralization followed by deprotonation and deacetylation process. The fish scales were widely used for the synthesis of Ag/chitosan hybrid in low cost and ecofriendly manner. The usage of fish scales limits the genuine ecological contamination issues. Chitosan can inhibit termite attack in papermaking. As the amount of Ag/chitosan hybrid is increased, the weight loss is decreased. Ag/chitosan hybrid shows better photocatalytic properties under UV light. Ag/Chitosan concentrations were observed for the photodegradation of methylene blue by $AgNO_3$ were seen as Ro, R1, R2, and R3. R1 (5g Ag/chitosan, 5mL acetic acid) has more degradation because of more Ag/chitosan concentration. As the concentration of Ag/chitosan hybrid is increased, the degradation of methylene blue dye is also increased.

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