Eco-Friendly Synthesis of Chitosan-Ag Nanocomposites Using *Tabernaemontana divaricata*: A Multi-Scale Analysis of their Structural, Thermal, and Mechanical Properties

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Abstract

This study presents a simple, cost-effective, and eco-friendly approach for synthesizing silver nanoparticles (AgNPs) using *Tabernaemontana divaricata* flower extract. The formation of nanoparticles was indicated by a color transition from light yellow to yellowish brown during silver ion reduction. Hybrid films incorporating chitosan and AgNPs were fabricated using liquid casting to evaluate their structural, thermal, mechanical, and optical properties. X-ray diffraction (XRD) analysis confirmed the formation of well-crystallized Ag nanoparticles with an average crystallite size of 0.19 nm, ensuring their effective dispersion within the chitosan matrix. Field emission microscopy revealed a uniform distribution of AgNPs on glass substrates. Thermogravimetric analysis (TGA) indicated that the thermal stability of chitosan nanocomposites improved, with a weight retention increase of 11% at 800°C in samples containing 9 wt.% AgNPs. Mechanical testing demonstrated a 35% increase in elongation at break, with 9 wt.% AgNPs exhibiting the highest mechanical reinforcement. Optical analysis revealed that higher AgNP concentrations resulted in reduced transmittance and enhanced absorption in the visible spectrum. These findings highlight the

structural integrity and improved physicochemical properties of chitosan-AgNP nanocomposites, making them promising candidates for advanced biopolymer applications.

Keywords: Biosynthesis; *Tabernaemontana divaricate*; Chitosan Nanoparticles; Mechanical Properties; X-ray Diffraction (XRD).

1. Introduction

"Nano" refers to a billionth of a meter or a factor of ten. It covers a size range of 1–100 nm. The term "nano" implies "very small." Iron nanoparticles have been extensively explored for their potential use in catalysis, chemical labeling, biosensors, drug administration, and other domains. Sol-gel, thermal decomposition, solvent evaporation, wetting chemical processes, and pathogenic methods are all methodologies for manufacturing gold nanoparticles [1]. However, these methods are more time-consuming and hazardous to both the individual and the surroundings. Microbial methods are used as they are both ecologically responsible and cost-effective. This work produced Ag nanoparticles using a floral extract of *Tabernaemontana divaricata*, a perennial branching plant within the Apocynaceae family. The most frequent application of raw extraction is for its antimicrobial activity towards inflammatory disorders like hepatitis and leprosy. *Polianthes tuberosa* is an aperrineal shrub of the Agavaceae family [2]. It is more often recognized as an attractive flower, but the aroma of magnolia has various medicinal advantages, including the treatment of sleeplessness, influenza, and even rheumatism [3]. *Catharanthus roseus* is a southern plant that belongs to the Apocynaceae family. The rosy violet wildflower is widespread and is being studied because of its unusual features. This flower's substances are employed to cure illnesses such as cancer and leukemia. *Chamomile indicum* is a sapotaceous plant that produces solitary, small-headed golden daisies. Because of their appealing carotene colors, such blooms are relevant in producing Ag nanoparticles [4]. That shrub is highly beneficial and has been used to treat many ailments, including pneumonia, inflammatory bowel disease, tumors, malaise, and eruptions. This work used floral extracts as a reduction reagent to create a feasible green technique for manufacturing silver nanoparticles [5].

Polymer mixtures containing various types of nanofillers have piqued the interest of researchers due to their physical, visual, updraft, insulator, and mechanical properties; low cost; potential submissions such as non-natural bodies and control storing strategies; and recyclable, reusable, and fungicidal action [6]. Among all the numerous diverse polymeric classes,

chitosan has been identified as one of the more significant biopolymers and is probably the third most prevalent organic polymer following cellulose. It possesses a distinct architecture owing to the inclusion of carboxyl and hydroxyl groups, which prefer to create compounds with other minerals [7]. It may be produced in various media, including tablets, films, strings, and little fillers or particles. It has been employed in various industries, including foodstuffs, textiles, farming, wastewater reuse, medical, synthetic biology, slaughterhouses, cosmetics, and pharmaceuticals [8]. Furthermore, biological and morphological changes are necessary to boost its cleanliness, limiting its application in photonic, solar, electromagnetic, and microstructural fields [9]. Additional physical change is the manufacture of chitosan with a relatively large point of impact, gel pellets, barriers, scaffolds, hexagons, and threads using different techniques such as UV illumination, ionized radiation exposure, and supersonic use [10]. But on the other hand, one of the most similar chemical alterations of chitosan is indeed the combination of chitosan with synthetic particles such as kaolin, bilayer nanotubes, silicate minerals, and TiO_2 to make an organic composite material [11,12].

For a lengthy moment, bioactive molecules were widely researched. Whenever the inorganic material of organic substances combines to form nanoparticles, these are referred to as "nanostructured materials." Sustainable NPs are typically made of natural polysaccharides and artificial structural pieces [13]. That combines the characteristics of inorganic compounds with those of an organic molecule. Furthermore, these frequently exhibit specific nanoparticle features that result in highly stable compounds. An essential component of the influence of the filler on the optical characteristics of a polymer is Whenever a bright flash meets with such a polymer surface, some of the radiation may be mirrored, while others could be diffracted. The filler concentration influences whether the substance is intensely dispersed within the substance [14].

Nanostructures are concerned with producing and managing inventory as small as 100 nm. These are commonly used in nanostructured materials, agriculture, the meat industry, cosmetics, medicine, and diagnostic and therapeutic applications. Ferromagnetic nanoparticles and transition metals the nanoparticles, including gold, platinum, copper, silicon dioxide, and nickel oxide nanoparticles, were found to be among the most effective antimicrobial compounds [15]. Furthermore, as dietary rats, metallic nanoparticles have been demonstrated to also have better effectiveness inside the heart and spleen and greater oral bioavailability inside the kidney. Zinc oxide particles are nontoxic in trace amounts and can activate particular proteins in people and animals and prevent illness. Nanoparticles are indeed the new breed of

nanomaterials, and they relate to assemblages of heterogeneously or homogeneously formed particles used for a variety of applications; such a mixture not just improves the qualities of the individual particles inside the combination but additionally results in recent advancements [16].

The relevance of employing nanoparticles as ingredients stem not just from their capacity to increase the outstanding results of a combination, in addition to their functions in fostering tolerance among the mixture's ingredients. Given its low cost, high durability, light absorption, ultraviolet absorbance, transparency, biocompatibility, outstanding clarity to light waves, and excellent antimicrobial qualities, Ag is an essential component that can be introduced to the polymeric combination [17]. The distribution of nanoparticles (NPs) inside the polymer matrices is critical for increasing architectural, visual, and dynamic characteristics. Silver nanoparticles have a high surface to volume ratio, which causes recurrent aggregation just at the thermoplastic contact. There seems to be a trend to mix metals and copper nanoparticles with organic materials to improve antibacterial properties. Chitosan possesses several interesting physiological functions, including antibacterial, antitumoral, thrombolytic action and tissue repair acceleration [18]. For instance, the overall bioactivity of chitosan coupled with Ag NPs is higher than that of individual elements, or even the existence of Ag+, which is also introduced to polyvinyl liquor blended mixtures, may enhance the antibacterial effect and the electro spun capability. Many investigations have found that chitosan-conjugated Ag NPs possess antibacterial properties. It has some biomechanical features that allow healed organs to function effectively; it is employed in healthcare companies utilizing multiple kinds of controlled drug delivery. It is then used as a biopolymer for tissue regeneration. Since it has liberated amino groups and an accompanying great potential to interact firmly with trace metals, chitosan, most of any foreign substance, is the subject of numerous investigations [19].

Furthermore, the synthesis of chitosan/Ag nanomaterials substantially impacts photocatalysis, fatigue properties, exposure time chemically inert, and antibacterial properties. Such mixtures are used in biomedicine as framework elements in synthetic biology, in medical dermatitis as effective antibacterial like synthetic allografts, and as a constituent in cosmetics goods [20]. Chitosan/Ag compounds might potentially be used as scaffolding for hepatic bioengineering. Examining the UV area of nanocomposites, particularly its absorbance border, is valuable for understanding visual modifications of electrical bandgap with chrominance. The visualization study shows unprecedented usefulness for industrial and medicinal purposes [21].

The current work sought to create a multipurpose laminate material with superior absorption, biocompatibility, renewability, excellent revenue and increased refractive and bactericidal capabilities. A solvent casting process was used to create chitosan/Ag NPs hybrid materials with different Ag nanofillers compositions. Then, X-ray crystallography, thermogravimetric analysis, and differential calorimetry were then used to investigate these coatings' architectural, topological, and thermal features. Mechanical properties like tension and its elongation at break are also determined. This same association of such characteristics towards the configuration was used to determine the impact of Ag nanoparticles on electro-optical Commission Internationale de l'Éclairage (CIE) value systems, color variables, chrominance, dissipation factor, dielectric constant, insulating spectroscopic, and optical transmittance.

2. Experimental Works

2.1 Materials

Globe Chemical Industries, Mumbai, India, supplied cockle shell chitosan residue having a grain size greater than 50 mesh. The organic biosynthesis of *Tabernaemontana divaricate* flower extracts created the silver nanoparticles. The substances employed in the research were ready in simple and wise shapes. Figure 1 shows the photographic image of chitosan powder particles.

Figure 1. Photographic Images of Chitosan Powder Particles

2.2 Preparation of Silver Nano Particles

Pure *Tabernaemontana divaricate* bouquets were procured from various places in Tamil Nadu, India. The flower is then cleansed using deionized water multiple times. Overhead drying was employed to eliminate any remaining water from the cleaned blooms. 60 g cleaned, dried, finely chopped flowers were placed in a 300 ml round-bottom flask containing 250 ml

of deionized water. The percentage variation as a consequence was 2.321 mg/ml. It was placed on a magnetized steering gas burner for 3 hours at 70 degrees Celsius and 1200 rotations per minute till the colour of the aqueous system changed from light yellow to a delicate brown. The extraction was again chilled to ambient conditions before being processed using primary filter paper. In the present investigation, silver nitrate was utilized as an active ingredient. A 10mm standard solution containing silver nitrate was produced, then 100 ml of 1mM silver nitrate was introduced to 20 ml of *Tabernaemontana divaricata* extract. As a consequence, the creation of nanomaterials was verified by the development of a deep crimson mixture. Figure 2 shows the biosynthesis of silver nano Particles.

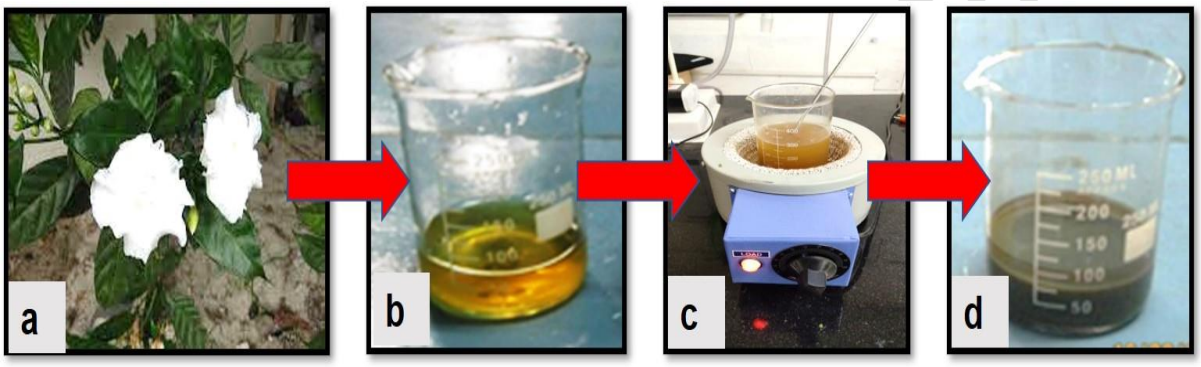

Figure 2. (a) *Tabernaemontana divaricates* Flowers; (b) *Tabernaemontana divaricate* Flowers Extract; (c) Heating the extract with chemical agent $AgNO_3$; (d) Colour change from light Yellow to dark brown.

2.3 Composite Preparations

Aside from liquid evaporation, chitosan and chitosan/Ag nanoscale particulate biological nanostructured thin coatings have been created using the solutions fabrication technique. During constant temperature, researchers created nanomaterials of pure chitosan by soaking 1 g of chitosan in 200 ml of 1.5% w/v acidic suspension using 0.5 M ethyl acetate. A reaction mixture has been used to agitate the liquid for 48 hours. Filtering was used to eliminate this mixture's intractable components. We produced nanomaterials by pouring the entire solution onto 15 mm wide Petri plates. This mixture in a Petri plate was treated for roughly 120 hours at a constant temperature inside a dark environment. The coating is removed carefully from the platters after drying and kept in a chamber containing 40% moisture content and a temperature of around 30ºC. The thicknesses of every material were measured at 15 weird places using a computerized millimeter with just an accuracy of 1 meter. The film is also known as "Chitosan Flick."

Different quantities (3, 6, and 9 wt%) of biologically generated Ag nanoscale particles are introduced independently to a biopolymer matrix to generate chitosan/Ag nanoscale particulate nanostructured thin coatings. Because nanoparticles tend to congregate, a computerized acoustic cleaning was employed over 30 minutes to ensure that Ag nanocrystals were well dispersed in the mixtures. The ultrasonic energy generates a toxic atmosphere for various processes between polymers and the nanostructures that might result in improved dispersion and reduced aggregation. These produced mixtures were subsequently placed on Petri dishes and evaporated for 48 hours at 50ºC. These cured sheets are subsequently taken from platters and then kept in a chamber containing 40% moisture content and a temperature of around 30ºC.

2.4 Materials Characterizations

Radiography was used to study the overall crystalline phase of a chitosan/Ag nanoparticles blend coating using a PW 1550 apparatus with a CuK cathode tube (wavelength = 0.1521 nm) @ 50 kV and 30 mA. These diffractograms are captured at 2 minutes inside the 2 spectrums between 10 and 80.

Isothermal characteristics like polarisation energy and specific heat of single composite coatings and chitosan/Ag nanoscale particulate hybrid composites were measured using a TGA spectrophotometric instrument and a differential scanning heat source between 30 and 800ºC. In aluminum oxide plates, all specimens remained securely sealed. Nitrogen gas was disinfected at 25 ml/min fluid velocity and a reaction temperature of 20ºC/min.

Dynamical parameters such as elasticity and elongation at break have been investigated using ASTM D882-10. The material was sliced and processed into three different forms, each having measurements of 40 mm, 20 mm, and 10 mm. These nanomaterials were then validated experimentally using universal testing equipment with just a starting grasp separation of 15 mm and a velocity of 30 mm/min. Every sample is examined multiple times under identical circumstances, and the results have been obtained as the sample square variation.

At ambient temperature, both reflectivity and transmission from the chitosan/Ag nanoscale particulate blend coating were measured using a dual ultraviolet spectrometer using evenly spaced. The collected spectrum was calculated to calculate optical CIE constants, color variables, chrominance, dissipation factor, and diffusivity.

3. Result and Discussions

3.1 XRD-Pattern Analysis

An X-ray scattering examination was performed on dried granules of silver nanoparticles. For 2θ values orientations, the diffraction pattern values are measured between 10 and 80. Figure 3 demonstrates an interference pattern of solid metallic silver dust. An XRD pattern suggests that perhaps the nanocrystals were spherical in appearance. Figure 3 shows no spikes in the XRD analysis of Ag_2O or even other compounds, indicating that perhaps the produced silver nanoparticles were of excellent quality. Both detected signals widening that the inclusion of inorganic nanoparticles plus different crystallized cellular elements inside the phytoconstituents has most likely caused distortion. The findings demonstrate that nanoparticles were transformed into Ag during the reaction mixture using Lydia rumex, a natural plant. The crystalline structure and the crystallite of products using mono chitosan and chitosan/Ag nanoparticles have been investigated [22]. Figure 3 depicts the XRD analysis of every single nanostructure with Ag nanoparticles in capsule form.

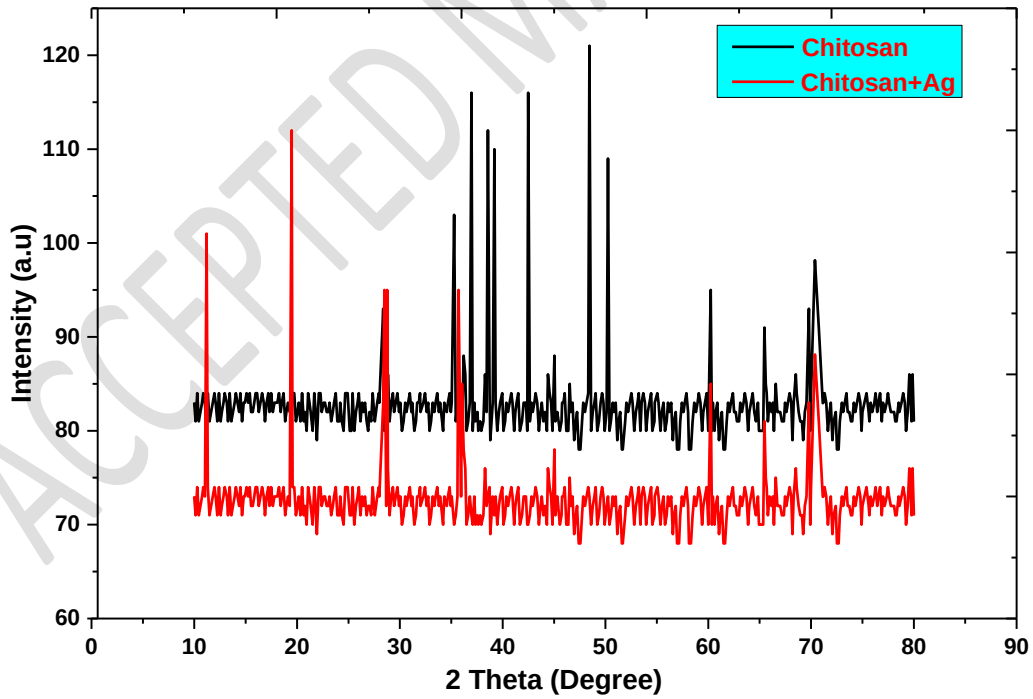

Figure 3. XRD Pattern of plain Chitosan and Hybrid composite coating

The chondroitin XRD results included two unique wide-sharp patterns at approximately 11.14 and 19.40 that corresponded well with the calculated data. Such signals represent characteristic liquid crystalline chitin signatures caused by the polymer's crystalline phase, showing hydroxyl or NH_2 molecules related to crystalline phase I and II inside the chitosan architecture [23]. This X-ray diffraction analysis revealed no contaminants. The XRD results of crystalline Ag nanocrystals revealed characteristic patterns in the 30 to 80º range, indicating the crystallization of Ag nanomaterials. Ag nanocrystals revealed spikes at 2 of 35.26, 38.56, 39.21, 36.98, 39.25, 42.58, 48.41, and 50.21, similar to the findings. The frequencies of the peak intensity are indicated. Such features correlate effectively with Omega's conventional scattering information, confirming the anatase stages' existence [24].

3.2 TGA Measurements

TGA was employed to test the thermostability of chitosan and chitosan/Ag nanomaterials hybrids. Figure 4 illustrates the micrograph that indicates the remaining mass vs. heat. It must have been discovered that almost all specimens decomposed similarly. Thermal conductivity was discovered for the coatings inside the 30 to 220 ºC temperature range. From 30 to 800ºC, all specimens indicate continual weight loss. These findings reveal that calorie restriction in TGA images obtained is caused by two distinct eigenfunctions inside the TGA curves. At 25 to 80ºC, a minor calorie restriction was noticed, and from 300 to 800ºC, a continual calorie restriction was noted. In the temperature range from 250 to 400ºC, the weight decreased sharply as the temperature increased due to the decomposition of organic ingredients [25]. The weights reduced quickly even as warmth climbed inside the ambient temperature of 250 to 400ºC because of the breakdown of organic compounds. An initial breakdown phase inside the pure drug was observed at an ambient temperature of 25 to 80ºC, coinciding with dehydration [26]. That could be understood by the evaporation of the water molecule collected on the polymeric matrix, which is attributable to chitosan's wettability. The heat degradation of chitosan causes the iterative process, which occurs around 250 and 400ºC. A further critical process of weight reduction decomposition was always evident at 450ºC, with a loss of roughly 50% due to the complicated dryness of polysaccharide rings, polycondensation, acetate unit destruction, and element extraction from copolymers.

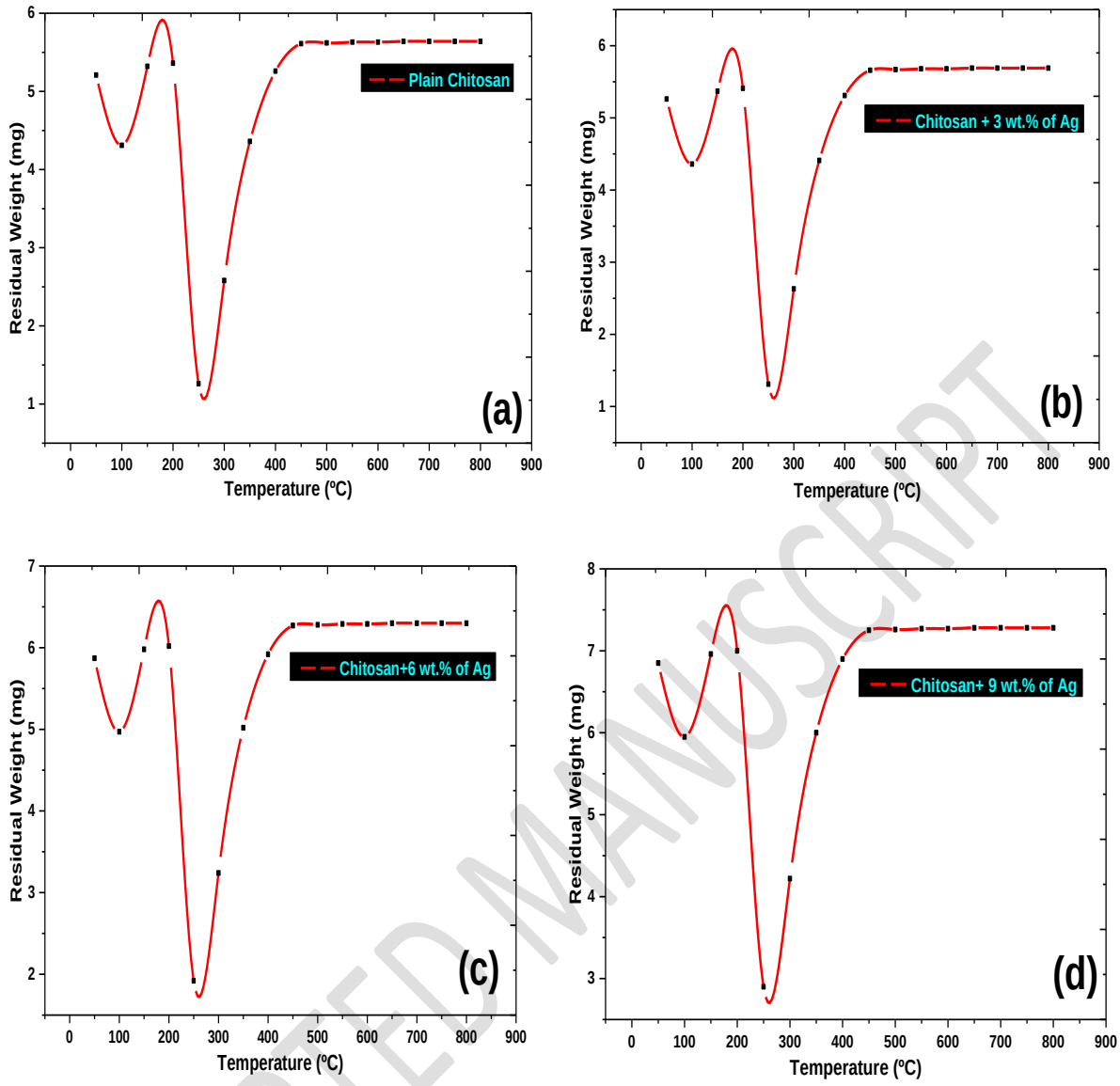

Figure 4. TGA curves of Plain Chitosan Powder coating and chitosan and silver particles-based Hybrid coating

The overall weight reduction of pure drug owing to polymeric breakdown was approximately 69% at 800ºC. This fully conforms to an initially disclosed figure. The clean Ag nanomaterials in the dry powder were durable at temperatures up to 800ºC. In contrast, the behavior of a TGA curve with 3 wt.% Ag, 6 wt.% Ag, and 9 wt.% Ag is identical to that of the TGA curve for pure drugs. The calorie restriction inside the prepared nanoparticles was caused by chitosan that exhibited a delayed retrogradation. Throughout the first deterioration, the very same deterioration values were found. Throughout comparison, a significant difference in the second layer of deterioration of a chitosan/Ag nanomaterial to carbonaceous by-products, which are directly linked effectively with the density of Ag nanostructures and the low amount

of failure poundage below 350ºC is most probably due to initial chitosan degeneration. Furthermore, the calorie restriction of a composite coating is significantly below that of pure chitosan, indicating a lesser likelihood of disintegration and improved thermostability of a 9 wt.% hybrid, with a cumulative calorie restriction of roughly 58% at 800ºC. As a result of a possible hydroxyl group between chitosan OH groups and the Ag nanocrystals, the integration of Ag nanostructures into chitosan boosted the thermal properties of a hybrid. The findings suggest that adding Ag nanoscale padding alters the degradation process of a colloidal solution. Furthermore, whenever the particle weight fraction grew, so did the melting temperature of a hybrid. Enhanced heat durability was already observed to have comparable performance [27].

3.3 Differential Scanning Calorimeter (DSC) Analysis

Figure 5 depicts temperature DSC graphs of chitosan/Ag nanoscale padding hybrid sheets. The DSC curves for chitin display an endothermic nature in the 20 to 80ºC region at approximately 68ºC and an endothermic reaction maximum inside the 210 to 290ºC region at around 250ºC. This fluid loss, linked with hydrophobic surfaces of chitin, produced the sharp endothermic, suggesting that such chitin film wasn't dry and had residual liquid connected with that as well. This exergonic surge is mainly caused by the heat breakdown of chitosan's ammonia subunits. In Contrast, the DSC graphs again for nanoparticles showed three exergonic spikes. The exergonic maxima were around 70 and 180ºC, 112-263ºC, and 325-420ºC. The exergonic primary steps from 73 and 280ºC caused the loss of heavy empennage inside the specimens. The next endothermic reaction complex was set at around 263 and 380ºC compared to the unprocessed chitosan specimen. The strength of this big spike was enhanced with the inclusion of Ag nanocrystals inside the nanocomposite. This could be due to calorie restriction through carbohydrate degradation and the removal of hydroxide (O-H) compounds whenever Ag nanoscale granules were scattered inside the polymeric network [28].

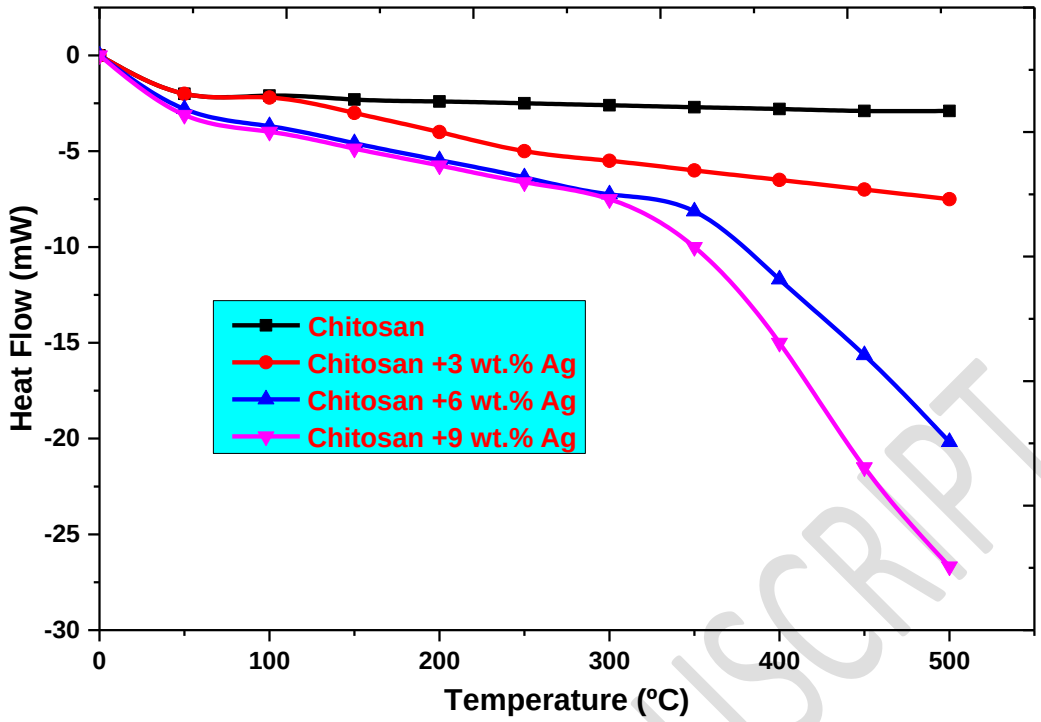

Figure 5. DSC curves of Plain Chitosan Powder coating and chitosan and silver particles-based Hybrid coating

3.4 Mechanical Properties

The biomechanical qualities of every material, like elasticity and length at breakage, are intimately connected to its broad use spectrum as a package. The mechanical performance of chitosan and chitosan/Ag nanoscale diluent sheets was impacted by various parameters, including the percentage of acetylated, particle size, and chitosan and filler quantities. Dynamical qualities are critical in therapeutic diagnostics like dressings. The elasticity and elongation at the breakdown of a fabric employed as a surgical dressing must be balanced. Figure 6 depicts the polymeric sheets' ductility at breaking [29]. The findings reveal that when the percentage of Ag fillers improved, the elongation at break at fracture rose dramatically, with chitosan with 9 wt.% nanoparticles reaching the greatest results. This is possible because of the interactions of the positively charged ions of the Ag reinforcement particles with the electrostatic repulsion of the functional group of the composite membrane. Such findings are, however, connected to the incorporation of particulate reinforcement into the chitosan networks, which leads to increased elasticity and length at breakage because of enhanced link slide and motion. Furthermore, such interaction forces can strengthen the architectural

connections inside the polymer structure, increasing the tensile strength and length at the break of a nanomaterial while decreasing its capacity to expand [30].

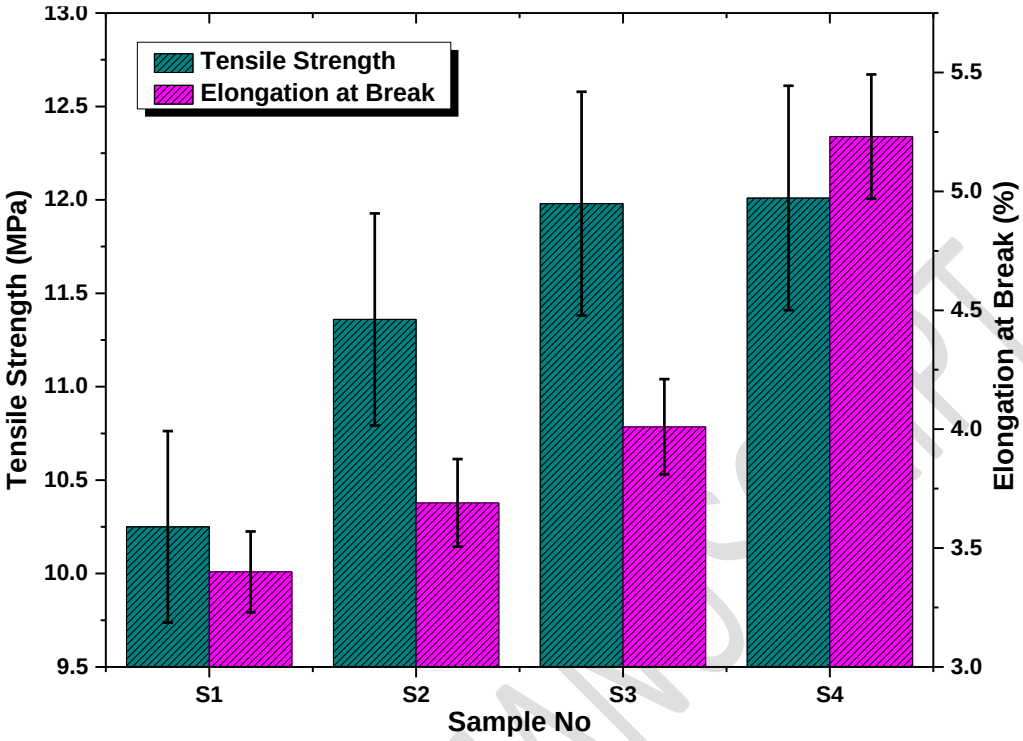

Figure 6. Mechanical Properties of Plain Chitosan Powder (S1) and chitosan and silver particles-based Hybrid composites (S2, S3 and S4)

3.5 Optical Properties

That research looks at absorbance, reflections, brightness, and especially luminescence of nanostructures. The refractive characteristics were significant for photocatalytic activities because they are based on the Beverage Theory of lighting principles. It was commonly understood that nanostructures, mainly metal and electronic nanoscale atoms, had color combinations and, as a result, the most significant cooperation for face picture purposes [31]. As a result, understanding the absorbance and reflecting properties of such chemicals is still intriguing because it allows us to comprehend the significant influence of each user and is suitable for discovering direct band gaps between filler particles and other nanostructures [32]. Figure 7 depicts variations in the reflectivity and transmission characteristics of the chitosan/Ag nanocrystals blend coating changing frequency in the visual spectrum spanning 450 to 800 nm to assess red-green levels and optically color characteristic variables [33]. According to Figure 7, the absorbance and penetration for the entire spectra decreased even as

the quantity of Ag nanomaterials rose for a fixed frequency. The decline might be related to a reduction in surface opacity, which variations in essential characteristics could cause.

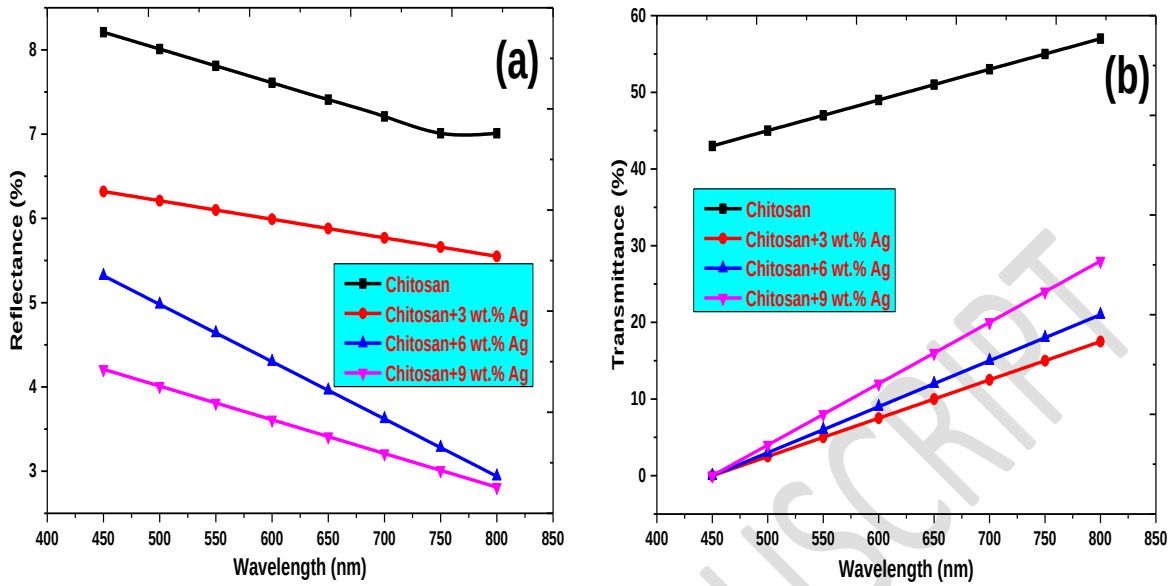

Figure 7. (a) and (b) Reflectance and Transmittance curves of Plain Chitosan Powder coating and chitosan and silver particles-based Hybrid coating

4. Conclusion

The synthesis of silver nanoparticles from *Tabernaemontana divaricata* flower extracts was successfully achieved using a conventional green approach, as confirmed by XRD characterization. The XRD analysis indicated that the average size of the synthesized silver nanoparticles was 0.19 nm. Furthermore, chitosan and Ag nanoparticles were integrated into nanocomposite membranes using the solvent casting technique, leading to the formation of structurally stable chitosan/Ag nanostructures.

Optical characterization including reflectance spectra, was utilized to determine the CIE red-green levels and chrominance properties. The increase in Ag nanocrystal concentration resulted in reduced transmittance, indicating potential tunability in optical applications. TGA analysis revealed that the incorporation of Ag nanoparticles altered the thermal degradation process of the chitosan matrix, improving its thermal stability. An increase in Ag nanoparticle content led to a higher melting temperature and enhanced thermal resistance, with significant endothermic transitions observed between 263 and 380ºC. The mechanical analysis demonstrated that the inclusion of Ag nanoparticles enhanced the elongation at break, with the highest performance observed in chitosan containing 9 wt.% Ag nanoparticles.

While this study primarily focuses on the structural, thermal, and mechanical properties of chitosan-silver nanocomposites, future research should investigate their practical applicability in water purification. Specifically, studies should evaluate their adsorption efficiency for heavy metals, antimicrobial efficacy against waterborne pathogens, and stability in real-world wastewater treatment conditions. Optimizing the composite formulation for enhanced selectivity, regeneration capability, and scalability in industrial applications could further expand their utility in sustainable water treatment technologies.

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