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Hybrid Nanocomposite of Chitosan/Polyvinyl Alcohol and Green Synthesized Al2O3-SiO2 Nanoparticles for Food Packaging Application

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Abstract

In the context of emerging global concerns with synthetic plastic packaging, alternative natural biodegradable packaging materials are gaining increasing attention for food packaging applications. In this study, Chitosan/Polyvinyl Alcohol nanocomposite hybrid films containing green synthesized Al2O3-SiO2 nanoparticles (NPs) were developed and microstructural properties were studied. Antimicrobial activity of the developed films was evaluated using both gram positive bacteria (Staphylococcus aureus) and gram negative (Escherichia coli). Green synthesis protocol was used for the precipitation of Al2O3-SiO2 NPs using

extract of leaves of fimbristylis tetragona plant as capping agent. The as-synthesized crystalline Al2O3-SiO2 NPs average crystallite size was in the range of 39 nm. Reinforcement with Al2O3-SiO2 NPs in the hybrid films leads to improved thermal stability, elongation-at-break (EAB), and compactness properties. The developed films with 2% and 4% Al2O3-SiO2 NPs showed a smooth, compact, and heterogeneous surface morphology compared to the control (chitosan-gelatin hybrid) films. Disc discussion assays showed that the nanocomposite film had significant antimicrobial activity against E. coli. The developed hybrid nanocomposite films have the potential to be developed as a biodegradable alternative for post-harvest packaging of fresh fruits and vegetables.

Introduction

In these decades fast food or readymade food to eat or cook food is more popular. So, there is a need to increase the production of food. Al_2O_3-SiO_2 nanoparticles are gaining attention in food packaging applications due to their unique properties, which enhance the performance of packaging materials. These nanoparticles, when integrated into packaging films, significantly improve the material's barrier properties, mechanical strength, and thermal stability. The combination of aluminum oxide (Al_2O_3) and silicon dioxide (SiO_2) form a hybrid structure that offers excellent resistance to oxygen and moisture permeability, which is crucial in extending the shelf life of food products.

Chitosan is an important biopolymer with a wide range of applications, particularly in fields like food packaging, pharmaceuticals, agriculture, and biomedical sciences, due to its unique properties. Derived from chitin, which is found in the shells of crustaceans such as shrimp, crabs, and lobsters, chitosan is biodegradable, biocompatible, and non-toxic, making it a sustainable and environmentally friendly material.

One of the most significant aspects of chitosan is its antimicrobial activity. It has the ability to inhibit the growth of a wide range of bacteria, fungi, and other microorganisms, which is particularly useful in food packaging. When used in packaging materials, chitosan helps extend the shelf life of food by preventing microbial contamination and spoilage. This property is critical for preserving perishable items like fruits, vegetables, meat, and dairy products.

Chitosan also offers excellent film-forming capabilities. It can form thin, flexible films that are useful in various applications, particularly in edible coatings for food products and biodegradable packaging materials. These films act as barriers to gases like oxygen and moisture, which can spoil food by promoting oxidation and microbial growth. By incorporating chitosan into food packaging, the shelf life of products can be significantly extended while maintaining food quality.

Another important property of chitosan is its biodegradability. As environmental concerns regarding plastic waste continue to rise, chitosan presents an eco-friendly

alternative to synthetic polymers in packaging. It decomposes naturally without leaving harmful residues, contributing to more sustainable packaging solutions.

Chitosan is also biocompatible, meaning it is safe for use in contact with living tissues and can be applied in areas such as wound healing and drug delivery systems. Its ability to bind with proteins and cells has been utilized in various biomedical and pharmaceutical applications, ranging from tissue engineering to targeted drug delivery.

Additionally, chitosan exhibits bioadhesive properties, which makes it an ideal candidate for applications where adherence to surfaces, such as biological tissues, is required. This feature is particularly valuable in developing controlled-release drug delivery systems or functional coatings for biomedical devices.

In agriculture, chitosan is used as a plant growth promoter and a natural pesticide. It stimulates plant growth and strengthens the plant's immune system, helping crops resist diseases and environmental stresses.

Overall, chitosan's antimicrobial, biodegradable, biocompatible, and film-forming properties make it an invaluable material for a wide variety of applications, with its importance growing as industries seek more sustainable and eco-friendly alternatives to conventional materials.

In particular, Al_2O_3-SiO_2 nanoparticles create a more compact and organized structure within the packaging material, reducing the transmission of gases and water vapor that typically lead to spoilage. This barrier function is especially important for packaging fresh and processed foods, where maintaining an optimal internal environment can prevent oxidation and microbial growth. For instance, in meat or dairy packaging, the use of Al_2O_3-SiO_2 nanoparticles can help preserve freshness by limiting oxygen exposure and retaining moisture.

Additionally, these nanoparticles contribute to the mechanical strength of the packaging films, making them more resistant to tearing, punctures, or deformations. This enhances the durability of the packaging during handling and transport, ensuring that the food inside remains protected throughout its journey from producer to consumer. The inclusion of Al_2O_3-SiO_2 nanoparticles also improves the thermal stability of the packaging materials, allowing them to withstand higher temperatures without degrading, which is beneficial for packaging foods that undergo heat treatment or need to be stored in varying environmental conditions.

Moreover, Al_2O_3-SiO_2 nanoparticles can impart antimicrobial properties to the packaging, helping to inhibit the growth of harmful bacteria or fungi on the food surface. This is particularly important for perishable goods, as microbial contamination is one of the main factors that reduce food shelf life. The antimicrobial effect, combined with improved barrier and mechanical properties, makes packaging materials with Al_2O_3-SiO_2 nanoparticles highly effective for preserving food quality and safety.

In summary, the application of Al_2O_3-SiO_2 nanoparticles in food packaging enhances the protective functions of packaging films by providing superior barrier properties, mechanical robustness, and antimicrobial activity. These improvements contribute to the extended shelf life of food products while also ensuring the safety and integrity of the packaging material.

These can be prepared using a certain hygienic way. One of the important parameters is to protect the food against foodborne diseases. Nowadays, food borne diseases are widely increasing very rapidly [1-2]. Mostly foodborne diseases are increasing in various counties like the US around 76 million peoples a sufferer from this, 3,25,000 people can get affected and around 5000 die due to such diseases. It became necessary to develop antimicrobial and stable packing material. This packing material improves the quality of the food. They protect the food from microbes [3-4]. A hybrid nanocomposite of chitosan, polyvinyl alcohol (PVA), and green-synthesized Al_2O_3-SiO_2 nanoparticles represents an innovative approach to sustainable food packaging. Chitosan, a biodegradable and antimicrobial biopolymer, is blended with PVA, a synthetic polymer known for its excellent film-forming capabilities and mechanical strength. This combination improves the flexibility and durability of the material, making it suitable for food packaging applications.

Aluminum-silicon oxide (Al_2O_3-SiO_2) nanocomposites are advanced materials combining aluminum oxide (Al_2O_3) and silicon dioxide (SiO_2) nanoparticles. These nanocomposites offer enhanced mechanical strength, thermal stability, and barrier properties, making them valuable for a variety of applications.

Al_2O_3 provides hardness, wear resistance, and thermal conductivity, while SiO_2 adds toughness and electrical insulation. Together, they form a nanostructured material that is more durable, heat-resistant, and impermeable to gases and moisture.

In food packaging, Al_2O_3-SiO_2 nanocomposites improve the shelf life of products by preventing oxidation and moisture ingress. Their lightweight nature, combined with thermal and mechanical resilience, also makes them useful in aerospace, electronics, and catalysis.

Synthesis methods like sol-gel, co-precipitation, and hydrothermal techniques ensure uniform nanoparticle dispersion, which is critical to achieving the desired properties. These nanocomposites are also used in biomedical devices due to their biocompatibility and antimicrobial properties.

In summary, Al_2O_3-SiO_2 nanocomposites are versatile materials with applications in packaging, aerospace, electronics, catalysis, and healthcare, offering a balance of strength, heat resistance, and barrier efficiency.

Green synthesis of Al_2O_3-SiO_2 nanoparticles involves using biological methods such as plant extracts to reduce and stabilize the nanoparticles. This eco-friendly method avoids the use of harmful chemicals typically involved in conventional nanoparticle synthesis. The Al_2O_3-SiO_2 nanoparticles are introduced into the

chitosan/PVA matrix to enhance the nanocomposite's mechanical, thermal, and barrier properties.

The resulting hybrid nanocomposite exhibits improved mechanical strength, elasticity, and gas barrier properties, making it more resistant to oxygen and moisture permeation. This is particularly important for extending the shelf life of food products. The antimicrobial properties of chitosan are further enhanced by the nanoparticles, providing an additional layer of protection against spoilage and microbial contamination.

Due to its biodegradable nature, this nanocomposite provides a sustainable alternative to conventional plastic packaging, reducing environmental waste while ensuring food safety and preservation. Its potential applications include packaging for perishable foods like fresh produce, meat, and dairy products, where both preservation and sustainability are critical concerns.

Most of the materials are inorganic which cannot react with the metal oxides or polymers or enzymes, the organic acids, enzymes and polymers and various metal oxides. The metal oxides have high temperatures [5-6]. Nanomaterials are newly developed materials, due to their good properties and features, they are used widely [7]. Such as nano-catalysts derived from plants are used for the synthesis of Benzimidazole derivatives [8-9]. In the last decades certain nanomaterials is used for food packing [10]. If a higher concentration of silver or other nanomaterial is present, they are not allowed for food packing. There are two types of nanomaterial packing was used such as packing in which the gases are trapped, in such case nanomaterials mixed with the polymer composites while in the second type the nanomaterial directly mixed with the food to get better antibacterial activity [11]. Metal nanoparticles with their potent antimicrobial properties are used for active packing. These are nanoparticles such as Ag, Au, Ti, Zn, Cu [12]. AgNPs and other nanoparticles can be hosted in different matrices and polymers with the agent as citrate and long chain polymeric alcohol. Among the many nanoparticles, the silver and aluminium oxide nanoparticles have a wide range of activity against fungi, viruses, yeasts and including bacteria [13]. These nanoparticles show very low volatility and good stability at high temperatures [14]. For such polymeric material, various strategies are used where the material was absorbed or directly incorporated in a synthetic process [15]. Furthermore, these nanoparticles have good properties. There is a certain risk associated with this due to the migration of the nanomaterial [16]. The food safety authority from Europe added the safety of the silver and aluminium nanoparticles [17]. According to the ESFA did provide the highest limit for the migration of the nanomaterial 0.05 mg/L in water and 0.05 mg/kg in food [18]. US FDA also gives certain rules for the packing of the food material, their metabolism and their mode of action [19]. Chitosan is a characteristic highly polysaccharide learned from the shells of shellfish by the deacetylation of chitin [20]. They have natural activity due to different functionality present. These may

bring a surge of different proteins and microbial cells [21]. This chitosan is a nonlethal polymer with good antibacterial activity is a good choice for packing material [22]. Moreover, this study describes a promising method to enhance packaging material presently used in the food industry. Moreover, there is mammoth potential for this method in other food safeguarding applications. In the present work, plant assisted Aluminum oxide nanocomposite shows real antibacterial properties that can be used to augment the mechanical and block properties of chitosan aluminium oxide nanofilms. These can be easily fabricated. This may increase the capacity of the packing material. They are used in industrial food packing.

The plant used for the nanomaterial coating Fimbristylis tetragona R.Br., belongs to the family Cyperaceae (Sedge family) Fimbristylis tetragona R.Br., Prodr. Fl. Nov. Holl. 226. 1810; C. B. Clarke in Hook. F. Fl. Brit. India 6: 631. 1893; T. Cooke, Fl. Bombay 3: 393. 1958 (Repr. ed.). Perennial with the short rhizome. Phenology-Flowering: August–September; Fruiting: September–December. Voucher specimen: ANC 1727.

Collection localities: Abasaheb Marathe College, Vikhare Gothane, Rajapur tehsil of Ratnagiri District (Maharashtra). Distribution: Fimbristylis tetragona is distributed in tropical Asia and Australia. In India, it occurs in Bihar, Gujarat, Goa, Karnataka, Kerala, Maharashtra, Meghalaya, Orissa, Rajasthan, Tamil Nadu, Uttar Pradesh, Madhya Pradesh, Andhra Pradesh and West Bengal. Illustrations: K. M. Matthew, Further Illus. Fl. Tamilnadu Carnatic 4: t. 675.1988; C. B. Clarke, Illus. Cyp. t. 40, f. 1-4. 1909 [23-24].

Experimental Material and Methods

Materials

Chitosan is a natural polymer having an average molecular weight 92,700 g mol−1, and it can be deacylated at 82.5 purchased from S. D. fine limited. Polyvinyl alcohol and acetic acid, ammonium hydroxide, aluminium sulfate and SiO2 Powder were also purchased from S. D. Fine limited. Leaves obtained from the plant were dried in shade for 3 weeks. Then dried in the oven for 300C till constant weight was obtained. These are grinding into fine powder; the powder is dried at 400C for 6h or till constant weight was obtained and then stored in an air tight container.

Preparation of Aluminium-Si oxide Nanocomposites

The aluminum nanoparticles were prepared by the coprecipitation method aluminium sulfate, 0.1M was taken in distilled water. 0.2M of NaOH solution was added with stirring over the sonication. We have added water extract from the leaves of the plant as a capping agent, 1 g of the SiO2 NPs. The obtained creamy solution was kept for 6h for settling down and upper solution remove easily. The precipitated washed several times with water then dried at 900C for 12h till constant

weight was obtained. The precipitated heated at the 500 0C for 3h to obtain alumina silica nanocomposites. Chitosan was placed in 5 mL of the acetic acid (2%) at temperature to get a homogenous solution. The synthesis of the aqueous PVA solution at 800C by mixing PVA polymer in distilled water. The PVA/CS (70:30 wt/wt) was blended and the solution was stirred. Bio-nanocomposites Al2O3-SiO2 loading at 0.5, 1, 3, 5 % w/v created through the Al2O3-SiO2 suspension of 1mg/mL in water to the PVA/CS and sonicated for 4 h. The homogeneous suspension of PVA/ Al2O3-SiO2 was taken in the Teflon plate and kept for 85 h at room temperature to get the film. The film of PVA/CS/Al2O3-SiO2 was taken out of the mould. Then it was characterized and used for packing material. All measurements were carried out by the standard protocol.

Results and discussion

Infrared Spectral Analysis

The IR Spectra Fig.1 was recorded on the Shimadzu IR Infinity from 400-4000cm-1 on the IR spectra. Si-O-Si bond occurs at 1196 cm-1, Al-O-Al bond occurs at 1116 cm-1, Si-O-Al bond occurs at 596 cm-1, Al-O bond occurs at 596 cm-1 and Al-O bond occurs at 596 cm-1.

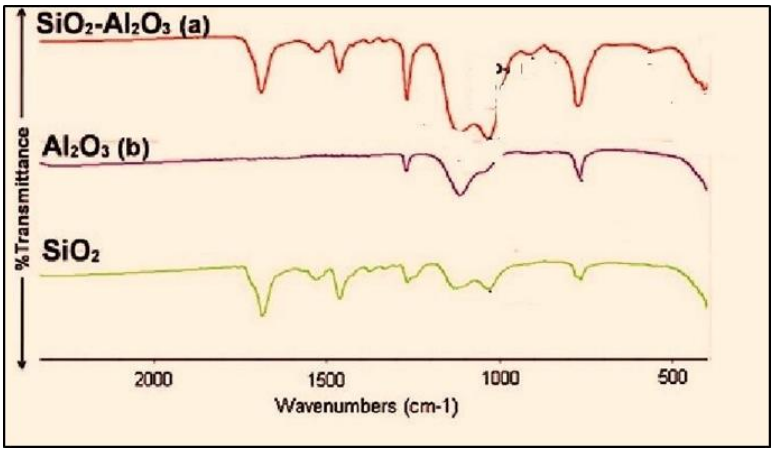


Fig.1. FTIR Spectra of SiO2, Al2O3 and SiO2-Al2O3.

X-ray Diffraction (XRD)

X-ray diffraction (XRD) is an analytical technique used to study the crystallographic structure of materials. When X-rays are directed at a crystalline sample, they interact with the crystal lattice and are diffracted in specific directions based on the atomic arrangement. By measuring the angles and intensities of the diffracted beams, the crystal structure, phase composition, and grain size of the material can be determined. XRD is widely used in material science to identify compounds, analyze crystalline phases, and assess the purity and structural properties of solids. The x-ray diffraction Fig.2 was measured using an X-ray Philips diffract meter fixed with radiation. Fixed with radiation of Cu lamp Kα

(45kV, 40mA, with λ=0.15418nm). In 2 theta degree of the scanning. The Al2O3-SiO2 nanocomposites were studied using the XRD. The XRD pattern of synthesized Al2O3-SiO2 nanocomposites is carried at 2 theta values for Al2O3 32.00, 34.89, 36.67, 47.87, 56.78, 62.90, 67.90, 70. The peak of the planes agree with (100), (002), (101), (102), (110), (103), (112) and (201) 2 theta values for SiO2 22.00, 39.00, 45.00, 77.67, 76.67 and planes occur at (100), (110), (111), (220) and (221).

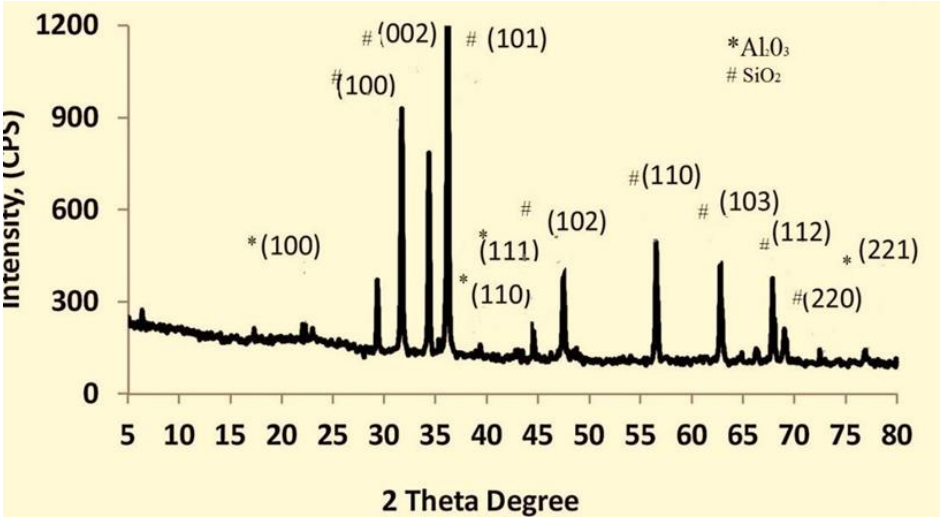


Fig.2. X-ray diffraction (XRD)

Barrier Properties

The barrier properties Table2 can be measured using the GBI W303(B) and the water permeability analyzer water vapours transmission rate was measured using the CUP procedures. The amount of the water vapours coming out of the film was tested for the test film. The water vapour permeability as well as the gas transmission rate of the prepared nano biocomposites.

Table1: Barrier properties of various synthesized biopolymers.

Samples	Al2O3-SiO2	OTR, g/(cc/m^2.day)	WVTR, g/(m^2.day)
PVA/CS	0.0 Al2O3-SiO2	150	900
PVA/CS	0.5% Al2O3-SiO2	140	970
PVA/CS	1% Al2O3-SiO2	90	1010
PVA/CS	3% Al2O3-SiO2	70	1050
PVA/CS	5% ZnAl2O3-SiO2	50	1110

Scanning Electron Microscopy (SEM) and EDX Measurement

SEM Fig.3 was recorded with SEM, JSM6360. This was used to study the morphology of the filmed dispersive energy spectrometer (EDX) to which

microscope carried at 10-15 kv as accelerating voltage. This was carried out using the low deposition rate.

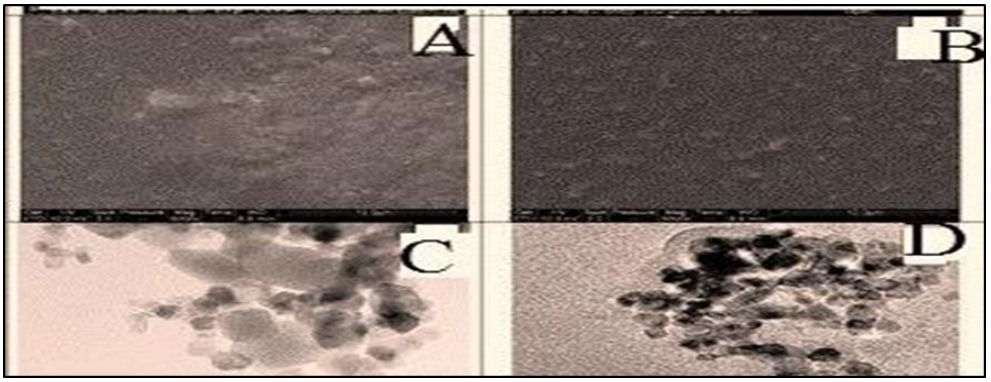


Fig 3. SEM images of the prepared PVA/CS blend, a) PVA/CS Al2O3SiO3 bio-nanocomposites, b) at concentration 0.5%, c) at concentration 1%, d) at concentration 5%.

The morphological analysis of the various Bio-nanocomposites film was evaluated using the scanning electron microscope. This was also carried out for 0.5, 1.0 and 5% indicate that the good particle size of the material.

Antibacterial Activity

Antimicrobial Activity Table2 of the polyvinylalcohol/chitosan (PVA/CS) was carried out with the different concentration Al2O3-SiO2 nanoparticles. As (0.5, 1,3 and5%) was studied using the zone of inhibition method. Certain gram-positive and Gram-negative bacteria were used. For this Specific protocol was used.

Table 2: Concentrations of Al2O3-SiO2 against Common Bacteria.

Sample	**Sta9**	**Escherichia coli**
PVA/CS	0	0
0.5% Al2O3-SiO2	23	28
1%Al2O3-SiO2	25	31
3%Al2O3-SiO2	32	37
5%ZnAl2O3-SiO2	39	41

The result of the antibacterial activity shown was better and had the good activity against the different compositions of the Al2O3-SiO2 nanocomposites.

Table 3: Mechanical Properties of the synthesized biopolymers.

Samples	**% Al2O3-SiO2**	**Tensile strength Pa**	**Elongation (%)**
PVA/CS	0.00	2.71x107	60.00
CS	0.00	1.58x107	95.00
Al2O3-SiO2	0.5%	7.68x107	78.00
Al2O3-SiO2	1%	6.61x107	110.00

Al2O3-SiO2	3%	5.71x107	120.00
Al2O3-SiO2	5%	4.81x107	130.00

The results of the mechanical properties in Table 3 indicate that various Nanocomposites have good tensile strength and the% of elongation. We have synthesized the biopolymer using the nanocomposite of the Al2O3-SiO2. During the synthesis of this nanocomposite, we used the leaves of the Fimbristylis tetragona plant. The various natural products present in leaf powder act as a capping agent for this nanocomposite instead of the chemical regent. The formation of this nanocomposite has been proved using IR spectroscopy. The nature of the nanocomposite is proved by the XRD. The morphology is given by the SEM and EDX analysis. The synthesized biocomposites are converted to biopolymer using various natural polymeric material. The synthesized biopolymer contains various % of the nanocomposites and shows good antimicrobial activity. It shows all properties required for the biopolymer.

Conclusion

The study demonstrates that various bionanocomposite films of PVA/CS/Al_2O_3-SiO_2 exhibit satisfactory tensile strength, along with excellent gas and water vapor barrier properties. These films, synthesized using chitosan and polyvinyl alcohol with varying percentages of Al_2O_3-SiO_2, show strong antibacterial activity against common bacterial strains. The findings suggest that these nanocomposites provide an improved material for food packaging. A green synthesis approach was employed, using plant leaves as a natural coating material for the production of nanomaterials. This eco-friendly method highlights the potential of sustainable, green chemistry in developing high-performance packaging solutions.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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