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Synthesis and characterization of sustainable nano biofilm derived from chitosan nanoparticles and stem starch of *Amorphophallus paeoniifolius*

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Abstract

Packaging one time use plastic garbage possess severe threat to the environment. To address this issue, this study develops and characterizes biodegradable bioplastics films derived from stem starch of *Amorphophallus Paeoniifolius* with chitosan nano particles as filler material, vinegar as stabilizer and glycerol as plasticizer. The wt% of the chitosan nano particles were varied from 1 wt% to 6 wt% in steps of 1 wt% for each bioplastic films and the films were casted by solution casting technique. The casted bioplastic films were tested for its tensile, physical, morphological, water captivation, water vapour transmission, biodegradability and thermal characteristics. The bioplastic film with 5 wt% of chitosan nano particles possessed maximum functional properties with tensile strength of 4.13 MPa, tensile modulus of 18.63 MPa, elongation at break of 61.03%, thickness of 0.55 mm, solubility of 34.83%, water vapour transmission rate of 5.21 g/m^2.h, water absorption of 19.67%, biodegradability of 18.94% in 15 days, density of 1.57 g/cm^3 and thermal stability of 148 °C. The Fourier transform infrared spectroscopy ensures the presence of hydroxyl and carbonyl groups in the bioplastic films. The obtained thermo-mechanical and hydrophilic results of the bioplastic films ensure its usage in packaging applications, offering a viable solution to the global plastic pollution crisis.

Keywords Bio-plastic · Stem starch · Plasticizer · Chitosan filler · Thermo-mechanical characteristics

1 Introduction

The escalating plastic waste crisis necessitates innovative solutions for sustainable material production. The plastic waste crisis poses profound threats to ecosystems, biodiversity, and human well-being [1]. Single-use plastics, such as bottles and bags, contaminate waterways, soil, and oceans, disrupting natural processes and habitats.

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Marine life is particularly vulnerable, with entanglement and ingestion of plastic debris causing devastating consequences. Microplastics, resulting from plastic breakdown, permeate even remote ecosystems, contaminating drinking water, seafood, and agricultural soils [2, 3]. Evidence suggests microplastics act as vectors for harmful chemicals, posing toxicity and bioaccumulation risks in the food chain. Beyond environmental concerns, plastic pollution incurs significant economic burdens, including cleanup efforts, waste management, and lost tourism revenue [4]. The reliance on fossil fuels for plastic production exacerbates climate change.

To mitigate these impacts, concerted efforts are necessary. Reducing plastic consumption, promoting recycling, and innovating sustainable materials are crucial. Bioplastics offer a compelling alternative to traditional petroleum-based plastics, presenting environmental, economic, and social advantages [5]. Derived from renewable resources like plant starches, agricultural by-products, and algae, bioplastics reduce dependence on non-renewable fossil fuels, promoting a sustainable and circular economy. By utilizing biomass as feedstock, bioplastics mitigate environmental impacts associated with fossil fuel extraction and processing, including habitat destruction, pollution, and greenhouse gas emissions [6]. Bioplastics production involves lower carbon emissions due to carbon sequestration during plant growth, and some bioplastics are biodegradable or compostable, eliminating plastic pollution in landfills and marine environments. Bioplastics also drive agricultural innovation, providing new markets for farmers and rural communities. Cultivated crops like corn, sugarcane, and cassava offer additional income opportunities, while valuing agricultural residues and waste streams contributes to resource efficiency and a sustainable bioeconomy [7]. By harnessing renewable resources and reducing plastic pollution, bioplastics pave the way for a more sustainable future.

The widespread use of plastic film mulching in agriculture has raised concerns about its environmental impact. Wang et al. [8] investigated the effects of plastic film mulching on phthalate esters (PAEs) concentrations in soil and plants. Their study revealed that plastic film residues can alter soil physical and chemical properties, impact crop growth, and accumulate PAEs. Understanding the risks associated with plastic film mulching is crucial, especially given the predicted global usage of over 3 million tonnes of mulched plastic film by 2021, primarily in China. Gansu Province, China conducted two experimental studies to examine the effects of plastic film mulching on soil organic carbon content and overall soil health. The first study involved four treatments, including plastic film mulching with maize planting, while the second study simulated residual plastic film in soil after 10, 30, and 60 years of continuous mulching. Additionally, research at the State Key Laboratory of Soil Erosion and Dryland Farming in China investigated the impact of different types of plastic debris on soybean growth [9]. These studies demonstrate the need for sustainable alternatives to traditional plastic film mulching. Biodegradable bioplastics offer a promising solution, reducing plastic pollution and mitigating environmental risks.

Radiani et al. [10] characterized bioplastic nanocomposite films prepared from starch extracted from *Amorphophallus paeoniifolius* to know the influence of concentration of nanocrystalline cellulose on its mechanical and water retention characteristics. Their investigations conclude that *Amorphophallus paeoniifolius* starch-based bioplastic nanocomposite films with 7 wt% of nanocrystalline cellulose filler material exhibited maximum mechanical properties and acceptable water retention properties to be used for packaging applications. Their bioplastic nanocomposite films exhibited a maximum tensile strength of 3.6 MPa, tensile modulus of 22.5 MPa, lesser water vapor transmission rate of 4.2 g/m^2.h and 22.57% biodegradability in 12 days. Arifin et al. [11] studied the influence of plasticizers glycerol and sorbitol with different wt% of filler nano cellulose on the mechanical, solubility and water retention properties of bioplastic film prepared from starch extracted from corn. The studies on corn starch based nano bioplastic films conclude that the prepared bioplastic films with 2 wt% sorbitol and 5 wt% nano cellulose reinforcement exhibited maximum mechanical properties and acceptable water retention properties to be used for packaging applications. Their bioplastic nanocomposite films exhibited a maximum tensile strength of 4.69 MPa, tensile modulus of 0.084 MPa, lesser water vapor transmission rate of 2.259 g/m^2h and water solubility of 30.73%.

The shift toward a circular economy has further sparked interest in bio-based plastics derived from renewable biomass to reduce dependence on fossil fuels. Bio-based plastics play a vital role in this transition, but

their environmental benefits are contingent upon responsible production and end-of-life management [12]. While bio-based plastics promote circularity, their renewable nature does not guarantee biodegradability, and effective recovery strategies are crucial for sustainability. In a circular economy, product recovery at its highest value is essential, with strategies organized into a waste hierarchy. bio-based plastics is categorized into drop-in types, which are chemically identical to petrochemical-based polymers, and dedicated types, which lack a petrochemical-based equivalent [13, 14]. Dedicated bio-based plastics dominate the market and are expected to grow. However, starch extraction for bioplastic applications remains unexplored.

The degradability of bioplastics depends on various physicochemical properties, influencing their breakdown over time. Thickness, hydrophobicity, molecular weight, crystallinity, and melting point all play crucial roles in determining degradation rates [15]. Thinner materials generally degrade faster due to their larger surface area-to-volume ratio, while more hydrophobic bioplastics degrade slower due to reduced water absorption. Higher molecular weight polymers take longer to break down, and increased crystallinity leads to slower degradation rates. Moreover, materials melting at lower temperatures degrade more rapidly as they soften and become more accessible to degrading agents. Environmental conditions, such as temperature, humidity, and the presence of microorganisms, significantly impact degradation [16]. Microorganisms, including bacteria and fungi, produce enzymes that break down bioplastic polymers into simpler compounds. Both abiotic factors (heat, sunlight, and moisture) and biotic factors (microorganisms) contribute to degradation. Researchers employ various methods such as weight reduction, total carbon decrease, visual analyses, adenosine triphosphate measurements, spectroscopic spectrums, clear zone formation, and biochemical methane potential tests to evaluate biodegradability. Understanding the interplay between physico-chemical properties and environmental factors is crucial for designing bioplastics with tailored degradation profiles and developing sustainable waste management strategies. Despite degradation residues, bioplastics offer advantages, such as nontoxicity to living organisms [17]. Researchers can improve bioplastic design and degradation processes by learning more about how bioplastic breaks down in both natural and industrial settings. This will help reduce plastic pollution and make the economy more circular.

Despite significant advancements in bioplastic development, several research gaps persist. The reliance on food crops as feedstocks competes with food production and land use, highlighting the need for non-food biomass alternatives. The lack of standardized regulations and certifications for bioplastics is impeding their widespread adoption [18]. Furthermore, balancing performance, biodegradability, and sustainability remains a challenge. To address these challenges, innovative approaches are necessary. Developing novel bioplastic architectures with enhanced biodegradability and mechanical properties is crucial [19]. Exploring new biocatalysts and enzyme systems can improve bioplastic production efficiency. Life cycle assessments and techno-economic analyses can inform sustainable bioplastic design and production. The potential of non-food biomass feedstocks, such as agricultural waste, algae, or invasive plant species, is particularly promising [20, 21]. We need further research to optimise extraction methods, improve bioplastic properties, and scale up production. By addressing these research gaps, the development of sustainable bioplastics can bridge the gap between performance, biodegradability, and environmental sustainability.

To address these challenges, this study explores the development of biodegradable bioplastics from renewable resources, such as *Amorphophallus paeoniifolius* stems. This study explores the development and characterization of biodegradable bioplastics derived from *Amorphophallus paeoniifolius* stems (APS). The novelty of this research lies in the utilization of *Amorphophallus paeoniifolius* stems above the ground for extraction of starch to prepare bioplastics which is currently dumped as waste. The bioplastic nano films from *Amorphophallus paeoniifolius* Stem Starch (APSS) were synthesized with enhanced mechanical and thermal properties through a multi-stage process that involved starch extraction, gelatinization, and reinforcement of chitosan nano particles and fructose as plasticizer. The synthesized bioplastic nano films were tested for its functional groups, mechanical properties, morphology, biodegradability, solubility, water absorption and thermal characteristics. The resulting bioplastic nano films demonstrated biodegradability, renewability, non-toxicity, food safety, hydrophobic behaviour and sufficient thermomechanical characteristics, making them suitable for packaging applications.

This research contributes to the advancement of sustainable materials science, addressing pressing environmental concerns while promoting socio-economic opportunities for yam-cultivating communities. By harnessing the inherent properties of this tropical crop, this study provides valuable insights into the development of biodegradable bioplastic films, paving the way for industrial-scale production and commercialization. The findings offer a promising alternative to conventional plastics, mitigating plastic pollution and supporting a circular economy.

2 Materials and methods

2.1 Materials

The APSs were collected from the farms in the outskirts of Chennai city, Tamilnadu, India. The chitosan nano particles of size 60–150 nm were obtained from M/s. Aura Biotechnologies Private Limited, Tiruvallur, Tamil Nadu, India. Vinegar and glycerol plasticizer was purchased from M/s. Vairavan Medicals Private Limited, Chennai, Tamil Nadu, India.

2.2 Extraction of starch from APS

The process begins with the collection of harvested APS for starch extraction as shown in Fig. 1. Approximately 1 kg of stem was acquired from two plants, it is weighed using a digital scale to ensure precision. The APS was sliced into small fragments and grind using a mixer grinder to start the starch extraction process [22]. This action transforms the stems into a mush like consistency, facilitating the subsequent extraction stages. The resulting mush was transferred into a muslin cloth, which acts as a filtration medium. Starch was squeezed out from the mush and collected in a container. The process was repeated until maximum amount of starch was extracted from the mush. Then iodine test was performed on the small quantity of extract to confirm the presence of starch [23]. The extract was taken in a test tube and a few drops of iodine solution was added. The presence of starch in the extract was ensured by the change in colour from green to black. The extract solution was left out for 24 h for the starch particles to settle down at the bottom of the container. The settled down APSS was further purified by washing it with water to eliminate any impurities or residual substances. Next, the excess water was carefully drained, leaving behind the purified starch. The purified starch was sundried for 2 days to reduce its moisture content to enhance its shelf stability and quality [24]. The sundried APSS was dry and powdery making it suitable for fabrication of bioplastic films. From the initial 1 kg of APS, approximately 11 g of APSS was successfully extracted through the process.

2.3 Fabrication of bioplastic nano films

APSS based bioplastic nano film fabrication starts with mixing of 9 g of APSS in 200 ml of distilled water to produce starch solution of 4.5% (w/v) in a beaker. On continuous stirring 5 wt% of vinegar was added to the APSS based starch solution as stabilizer [25]. After homogeneous mixing of vinegar stabilizer in the APSS based starch solution chitosan nano particles (CNPs) at different weight% was added to the mixture as shown Table 1. The APSS based starch solution containing vinegar and chitosan nano particles were heated by placing the beaker in a pan as shown in Fig. 2a and stirred continuously using a glass rod. At this stage, the mixture will have a milky white colour and be quite watery [26]. On reaching the gelatinization temperature of 60–65 °C 2 wt% of glycerol was added to the APSS based starch solution mixture as plasticizer. Further the mixture was stirred well and heated to 85–90 °C, at this point the mixture transforms into a viscous substance resembling a glue as shown in Fig. 2b [27]. Then the mixture was degassed and allowed to cooled to 40–45 °C. Now the mixture was poured on a butter paper and allowed to harden for 2 days as shown in Fig. 2c. Figure 2d shows the fabricated APSS based bioplastic nano film.



Fig. 1 Extraction of starch from APS **a***Amorphophallus paeoniifolius* plant **b** Collected APS **c** Sliced and grinded APS **d** Extract from grinded APS **e** Iodine solution test of APS extract **f** APSS in powdered form

Table 1 Nomenclature of APSS bioplastic nano films

Sample code	Material combination in wt%
0 wt% CNPs	93 wt% APSS + 5 wt% Vinegar + 2 wt% glycerol
1 wt% CNPs	92 wt% APSS + 5 wt% Vinegar + 2 wt% glycerol + 1 wt% CNPs
2 wt% CNPs	91 wt% APSS + 5 wt% Vinegar + 2 wt% glycerol + 2 wt% CNPs
3 wt% CNPs	90 wt% APSS + 5 wt% Vinegar + 2 wt% glycerol + 3 wt% CNPs
4 wt% CNPs	89 wt% APSS + 5 wt% Vinegar + 2 wt% glycerol + 4 wt% CNPs
5 wt% CNPs	88 wt% APSS + 5 wt% Vinegar + 2 wt% glycerol + 5 wt% CNPs
6 wt% CNPs	87 wt% APSS + 5 wt% Vinegar + 2 wt% glycerol + 6 wt% CNPs

2.4 Characterization of APSS bioplastic nano films

2.4.1 Film thickness

The MITUTOYO 293-821-30 Digimatic digital micrometer, with an accuracy of 0.001 mm, was used to measure the thickness of APSS bioplastic nano films. Each film's thickness was measured six times at different points on

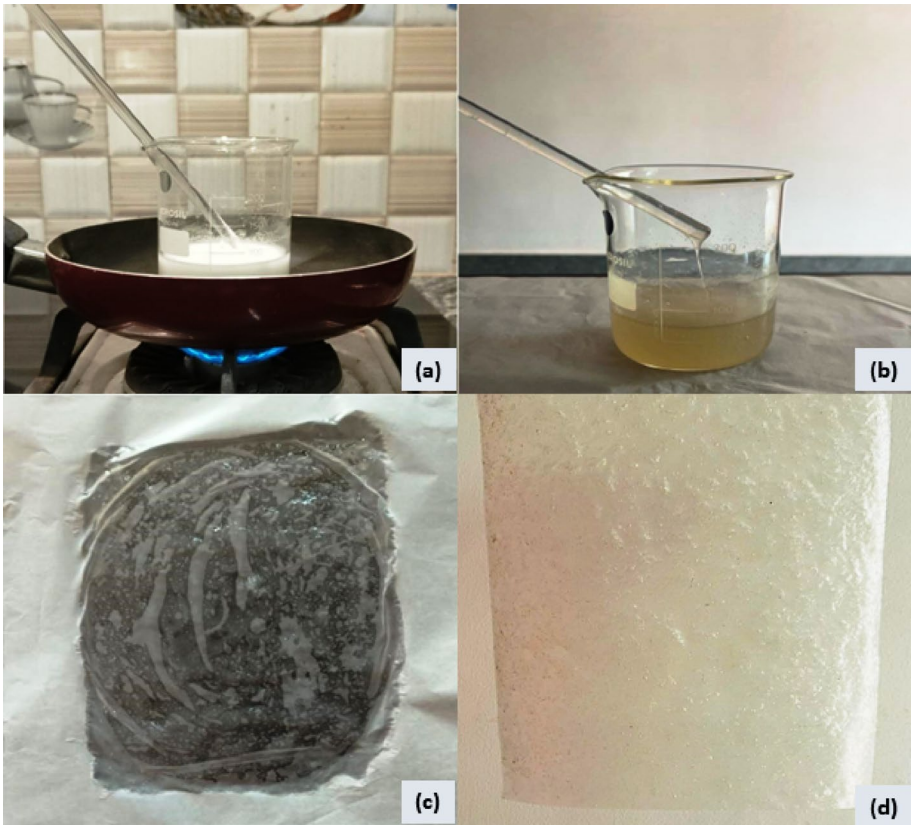


Fig. 2 Fabrication of APSS based bioplastic nano film **a** Heating of APSS solution with vinegar and CNPs **b** APSS solution after adding glycerol plasticizer **c** Poured APSS solution mixture and **d** APSS based bioplastic nano film

the film to guarantee the accuracy of the results [28]. The data obtained were then used to determine the mean thickness.

2.4.2 Tensile properties

In accordance with ASTM D882-88 standard, the mechanical integrity of the different APSS bioplastic nano films was analyzed using an Instron universal testing machine of 5 kN to measure the tensile parameters, such as tensile strength, strain at break, and Young's modulus [29]. The functionality of the APSS bioplastic nano films is mostly determined by its mechanical characteristics, such as tensile strength and strain at break. The machine clamped film strips that were 70 mm × 10 mm width were exerted a pulling force at a crosshead speed of 2 mm/min while maintaining a grip distance of 30 mm. Six trials of the tested samples were used for obtaining the mean for every variable using computer application installed with Bluehill Universal materials testing program.

2.4.3 Scanning electron microscopy

An argon plasma metallizer (sputter coater KK575X) was used to apply a very thin coating of gold to the APSS bioplastic nano films before scanning electron microscopy (SEM) in order to reduce charging impacts during imagery [30]. The APSS bioplastic nano film's surface was then examined with a Hitachi Model S-3400 N SEM running at 10 kV of accelerated voltage.

2.4.4 Density

A densimeter was used to measure the density of the produced APSS bioplastic nano films. To stop the hydrophilic substances from absorbing water, xylene was used as the immersing solution in this investigation. Xylene

successfully assured that the produced bioplastic films were not floating during testing because of its reduced density. Over the course of seven days, the APSS bioplastic nano films were dehydrated in desiccators with silica gel acting as a drying agent. After recording each APSS bioplastic nano film's initial mass (m), the APSS bioplastic nano film was immersed in xylene to determine the amount of liquid evacuated (v). Equation (1) was then used to determine the density (ρ) [30].

$$\rho = \frac{m}{v} \tag{1}$$

2.4.5 Film solubility

Three APSS bioplastic nano film specimens, each with a measurement of 2 cm diameter, were taken from every variety of APSS bioplastic nano film in order to assess its ability to dissolve. The APSS bioplastic nano films were then dried for 24 h at 105 °C in an oven to ascertain their initial mass (m_i). After that, each sample was submerged for 24 h at 24±2 °C in a beaker filled with 40 mL of distilled water, swirling occasionally to aid in solubility. Following the immersion time, the APSS bioplastic nano film's insoluble component was meticulously extracted from the solvent and dried at 105 °C for an additional 24 h. The percentage of soluble substance, which indicates the APSS bioplastic nano film's solubility in water, was then computed using the weight of the dehydrated insoluble leftover (m_f) following Eq. (2) [31].

$$\text{Solubility in } \% = \frac{m_i - m_f}{m_i} \times 100 \tag{2}$$

2.4.6 Water vapor permeability test

The Lyssy L80-5000 Water Vapor Permeability (WVP) analyzer was used to determine the water vapor transmission characteristic for each cast APSS bioplastic nano film. WVP was measured at 25 °C, 20% humidity level, and 700 m/min of air flow. The reduction in weight was tracked until balance was achieved. The following expression (3) [32] was used to determine WVP readings.

$$\text{WVP} = \frac{\text{WVTR} \times L}{\Delta P} \tag{3}$$

2.4.7 Water absorption

The amount of water that one gram of dried substance retains is a measure of the substance's ability to absorb water. ASTM D570-98 was used to evaluate the APSS bioplastic nano film's ability to absorb water. First, a centrifugal tube of known weight (m_I) was filled with 1 g of APSS bioplastic nano film, which was subsequently submerged in 25 ml of water that had been distilled. The centrifugal tube was centrifuged for 30 min at 2500 rpm. Following centrifugation, the residue was desiccated and dried in an oven for 40 min at 55 °C before being weighed again (m_{II}). Until the APSS bioplastic nano film's mass was constant, this procedure was carried out several times. Equation (4) [32] was used to determine the percentage of water absorption in APSS bioplastic nano films.

$$\text{Water absorption in } \% = \frac{m_{\text{I}} - m_{\text{II}}}{m_{\text{I}}} \times 100 \tag{3}$$

2.4.8 Fourier transform infrared spectroscopy

The possible alterations in the functional chains found in the APSS bioplastic nano films were examined using Fourier transform infrared (FTIR) spectroscopy. It aids in comprehending how the various parts interact and

verifies that the bioplastic was successfully grafted. A Nicolet 6750 AEM IR spectrometer from M/s. Thermo Nicolet Corporation was used to investigate each APSS bioplastic nano film. It operated at a spatial resolution of 4 cm^{-1} spanning a wavelength spectrum of 4000–500 cm^{-1}, and 42 scans were performed for each assessment [33].

2.4.9 Thermogravimetric analysis

A Q500 V20.13 Build 39 analyzer has been used to perform a thermal gravimetric analysis (TGA). A 10 × 10 mm piece of APSS bioplastic nano film underwent heating in a nitrogen environment from ambient temperature to 550 °C at a rate of 10 °C per min in platinum crucibles [34]. The reduction in mass as a reflection of temperature was tracked over time in this thermal study to evaluate each APSS bioplastic nano film's thermal endurance.

2.4.10 Biodegradability test

In accordance with ASTM D5988 guidelines, the biodegradable properties of the APSS bioplastic nano film samples were examined. For 15 days, the APSS bioplastic nano film samples were buried 3–4 cm deep in soil with 60% moisture [35]. The temperature of the experimental setup was approximately 28 °C. To determine the APSS bioplastic nano film sample's deteriorating pattern, the mass reduction was measured every five days.

3 Results and discussion

3.1 Film Thickness of APSS bioplastic nano films

The thickness fluctuation of the formed APSS bioplastic nano films is depicted in Fig. 3. The thickness of the APSS bioplastic nano films rose as the concentration of CNPs increased in the bioplastic films. The thickness

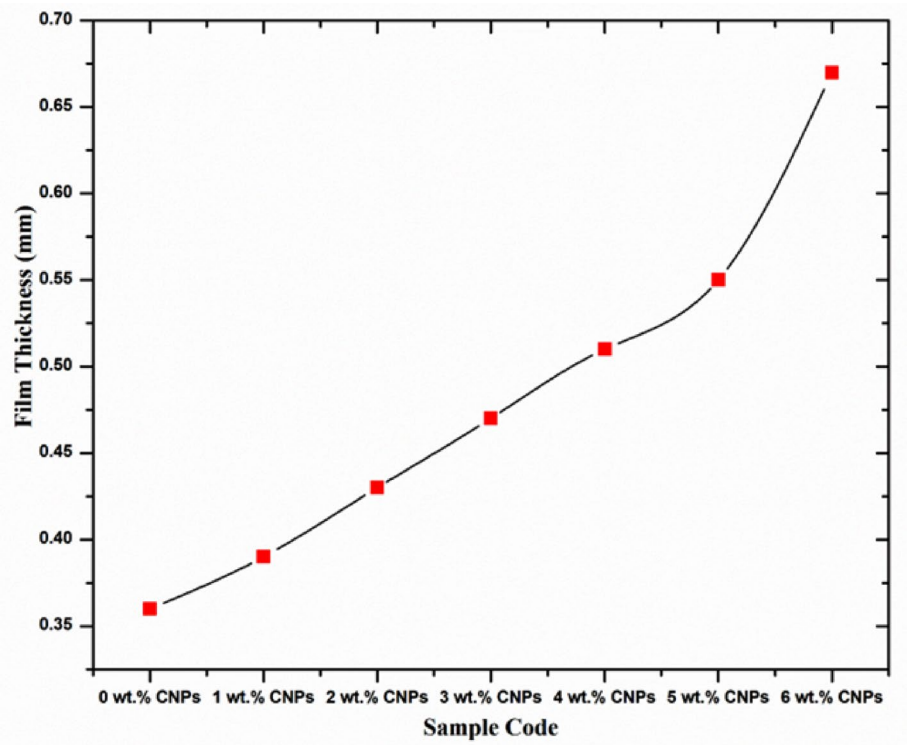


Fig. 3 Average thickness of the APSS bioplastic nano films

of the APSS bioplastic nano films is basically improved by the addition of glycerol plasticizer. The presence of glycerol plasticizer in the APSS bioplastic nano films collapses the intermolecular chains and generates more voids thereby increasing the thickness of the films [36]. The minimum thickness of 0.36 mm is noted for the APSS bioplastic nano film with 0 wt% CNPs whereas maximum value of 0.67 mm is recorded for the APSS bioplastic nano film with 7 wt% CNPs. This is owing to the fact that increasing the wt% of CNPs in the APSS bioplastic nano film increases the solid content of the film resulting in increased film thickness. Similar results of rise in thickness of the bioplastic films with increase wt% of nano filler particles were reported by Haghighi et al. [37]. It is also observed that in the case of APSS bioplastic nano films, the thickness of the films gradually increased with increase in the wt% of CNPs till the APSS bioplastic nano film with 5 wt% CNPs. This indicates the homogeneous distribution of the CNPs in the APSS bioplastic nano films. But a 21.81% increase in thickness is noted for the APSS bioplastic nano film with 6 wt% CNPs compared to the APSS bioplastic nano film with 5 wt% CNPs. This specifies the accumulation and uneven distribution of CNPs in the APSS bioplastic nano film with 6 wt% CNPs leading to a sudden increase in thickness of the bioplastic film.

3.2 Tensile characteristics of APSS bioplastic nano films

Figure 4a and b displays the APSS bioplastic nano films samples before and after tensile test as well as the tensile characteristics were shown in Fig. 4c. The APSS bioplastic nano film with 0 wt% CNPs had a tensile strength of 2.76 MPa. The presence plasticizers such as glycerol and vinegar stabilizer reduce the brittleness of the APSS bioplastic nano films by compromising its tensile strength. This is owing to the fact that the presence of plasticizers in the bioplastic films disrupts the hydrogen connection with the nearer molecules resulting in increased flexibility and reduced tensile strength [38]. But the addition of CNPs as filler in the APSS bioplastic nano films is found to improve its tensile characteristics. The tensile strength of the APSS bioplastic nano films improved till 5 wt% of CNPs addition and recorded a maximum tensile strength of 4.13 MPa. The high aspect ratio of the CNPs when reinforced in APSS interacted well with the matrix forming a stronger bioplastic film. Being that CNPs are uniformly distributed throughout the APSS matrix, stress is effectively transferred from the APSS substrate to the CNP filler through the interface when axial load is applied, improving the APSS bioplastic nano film's tensile characteristics.

The CNP-APSS coupling may also limit the substrate macromolecule's mobility, increase the APSS bioplastic nano film's strength and decrease elongation at break, which ranges from 96.78 to 66.97%. The flexible to fragile phase transition that happens when the CNPs wt% in the APSS substrate surpasses 5 wt% reduces the mechanical strength and flexibility. This is owing to the nonhomogeneous distribution and agglomeration of CNPs in the APSS bioplastic nano film with 6 wt% CNPs as filler material [39]. The APSS bioplastic nano film's modulus of elasticity, which ranged from 7.82 to 18.63 MPa, was greatly increased by the addition of CNPs. The increase stiffness of the APSS bioplastic nano films was owing to the uniform distribution of the CNPs and hydrogen bond formation in the bioplastic films. These alterations in properties under tensile stress imply that APSS bioplastic nano films containing 5 wt% CNPs have appropriate mechanical characteristics and are likely to be used in wrapping applications.

3.3 Surface morphology of APSS bioplastic nano films

The SEM images of control sample APSS bioplastic film and APSS bioplastic nano film with 5 wt% CNPs are shown in Fig. 5a and b. The surface of the control sample APSS bioplastic film is extremely smooth and uniform in texture, with no discernible bands or clusters, as seen in Fig. 5a. Few white spots noticed is the non-plasticized starch in the control sample APSS bioplastic film. On the other hand, Fig. 5b shows that the APSS bioplastic nano film with 5 wt% CNPs have no apparent interfacial layer and small bands with good firmness. This homogeneous dispersion is mostly caused by hydrogen bonding between the functional chains in the bioplastic film [39]. Being that CNPs are crystalline, there are slight bands on the surface of the APSS bioplastic nano film with 5 wt%

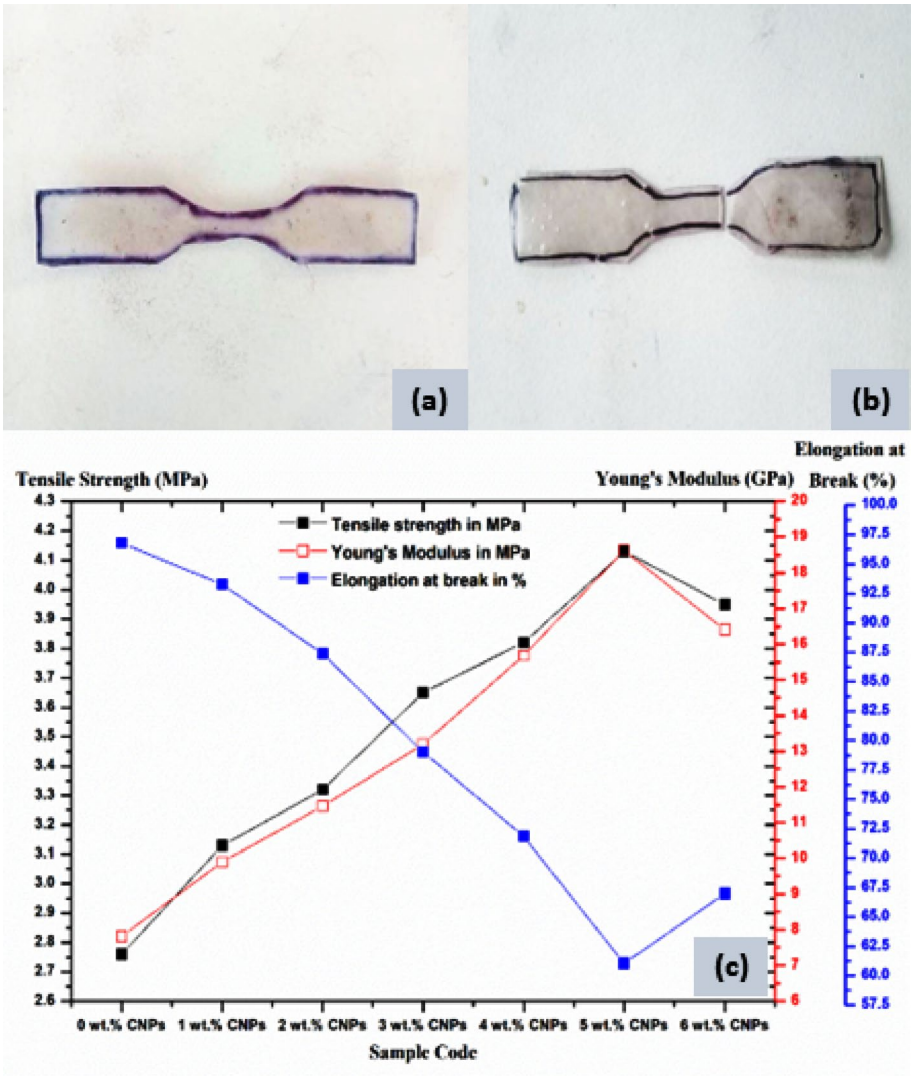


Fig. 4 Tensile characteristics of the APSS bioplastic nano films **a** Tensile specimen before test **b** Tensile specimen after test **c** Tensile properties and Elongation at break

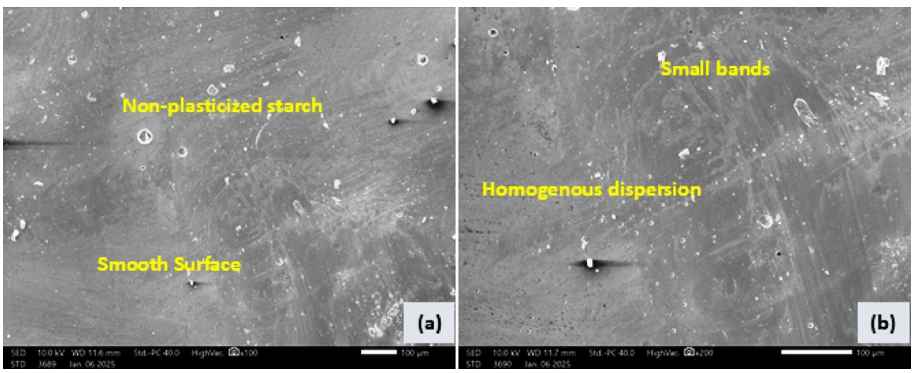


Fig. 5 Surface morphology of the TS bioplastic films **a** 0 wt% CNPs and **b** 5 wt% CNPs

CNPs. Moreover, the presence of CNPs in the APSS bioplastic nano films favoured the formation of hydrogen bonds within the APSS molecules as well as with the glycerol plasticizer and vinegar stabilizer molecules making the APSS bioplastic nano film with 5 wt% CNPs denser [40]. The homogeneous distribution of the CNPs in the APSS bioplastic nano film with 5 wt% CNPs is owing to the better aspect ratio of the CNPs. The particle size of

the CNPs were determined using the SEM images by randomly selecting 100 particles. The measurements give the average diameter of CNPs as 105.3 ± 31.3 nm.

3.4 Density of the APSS bioplastic nano films

The density of the APSS bioplastic nano films ranges from 1.49 to 1.57 g/cm³, as opposed to the control APSS bioplastic film's density of 1.46 g/cm³, as seen in Fig. 6. The density of all the APSS bioplastic nano films was higher than that of the APSS bioplastic film without CNPs. The APSS bioplastic nano film with 5 wt% CNPs had the highest density of 1.57 g/cm³ which is 7.53% higher compared to APSS bioplastic film without CNPs. The increase in density of APSS bioplastic nano films with CNPs as filler is due to the fact that the presence of CNPs in the bioplastic film accelerates the formation of hydrogen bonding among the APSS molecules as well as among the APSS, glycerol plasticizer and vinegar stabilizer molecules [41]. This makes the APSS bioplastic nano films with CNPs as filler material denser. The homogeneous distribution of the CNPs in the APSS bioplastic nano films were also found to favour the increase in density of the APSS bioplastic nano films. The reduction in density by 1.91% for the APSS bioplastic nano film with 6 wt% CNPs compared to the APSS bioplastic nano film with 5 wt% CNPs is due to the agglomeration of CNPs leading to more voids and increased thickness resulting in higher overall volume.

3.5 Solubility of APSS bioplastic nano films

A crucial property of APSS bioplastic nano films is their water solubility, especially in situations where resistant to water is frequently required, such as food that contains liquid packing. The addition of CNPs in APSS bioplastic nano films decreased its solubility as shown in Fig. 7. The solubility of the APSS bioplastic film without CNPs is 46.45%. The solubility of the APSS bioplastic film without CNPs is relatively higher due to the presence of glycerol plasticizer. Basically, the starch-based bioplastic films are soluble in nature due to the hydrophilic nature of the starch molecules [42]. Further, the presence of glycerol plasticizer being hydrophilic in nature weakens the

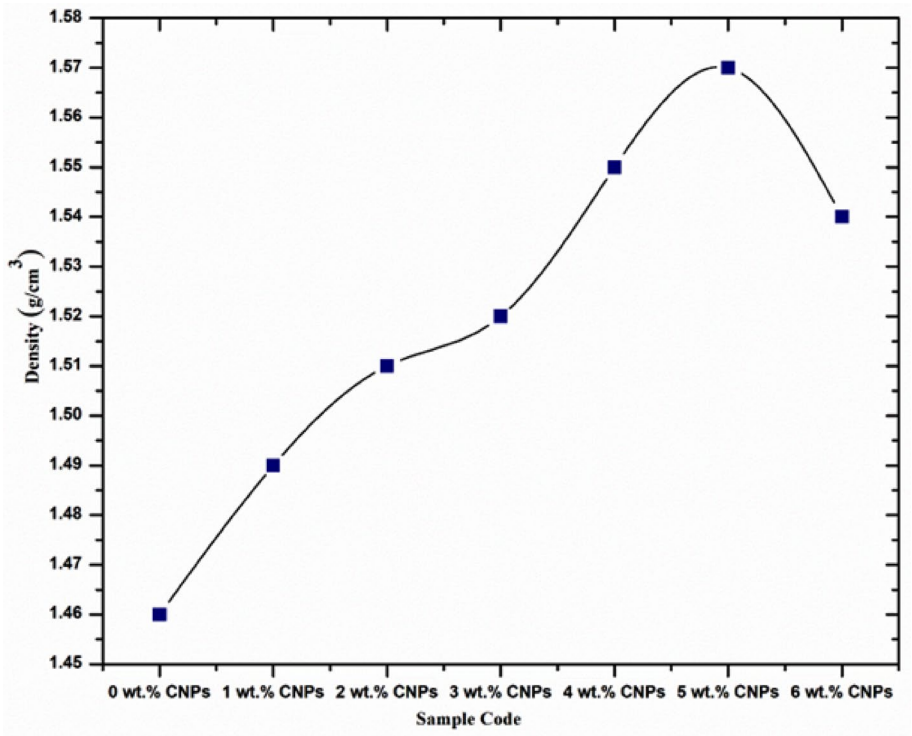


Fig. 6 Density of the APSS bioplastic nano films

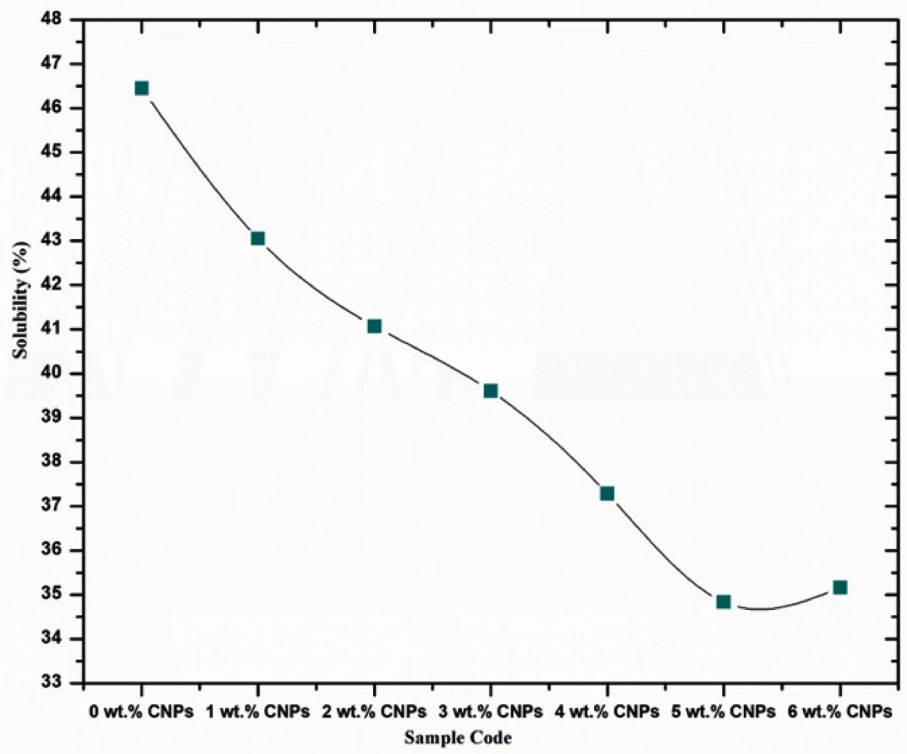


Fig. 7 Solubility of the APSS bioplastic nano films

bonding among the starch molecules and creates more voids between the molecular chains making the bioplastic film's surface more porous thus allowing the penetration of water molecules and favouring the solubility of the APSS bioplastic film without CNPs.

The solubility of the APSS bioplastic nano films with 1 wt% CNPs decreased by 7.31% compared to the APSS bioplastic film without CNPs. Moreover, the solubility of the APSS bioplastic nano films decreased with increase in wt% of the CNPs. Minimum solubility of 34.83% is achieved for the APSS bioplastic nano films with 5 wt% CNPs which is 25.01% lesser compared to the APSS bioplastic film without CNPs. This decrease in solubility of the APSS bioplastic nano films with addition of CNPs is due to the fact that the presence of CNPs in the APSS substrate favours the hydrogen bonding among the molecules of CNPs and APSS [43]. The CNPs interact through hydrogen bonding with carboxyl and hydroxyl clusters of the APSS thereby increasing the density decreasing the hydrophilic nature of the APSS bioplastic nano films with CNPs as filler material. In the case of APSS bioplastic nano films with 6 wt% CNPs the increase in solubility was due to the agglomeration of CNPs leading to more voids and permitting the water particles to penetrate easily thus favouring solubility.

3.6 Water vapor permeability of APSS bioplastic nano films

The WVP of APSS bioplastic nano films were presented in Fig. 8. The WVP of APSS bioplastic nano film with 0 wt% CNPs was 9.64 g/m^2. h. The higher WVP of APSS bioplastic nano film with 0 wt% CNPs is due to the influence of glycerol plasticizer and vinegar stabilizer in the bioplastic film. The presence of glycerol plasticizer and vinegar stabilizer in the APSS bioplastic nano film with 0 wt% CNPs in addition to providing flexibility to the bioplastic films weakens the bond between the APSS molecules creates space between the molecular chains promoting water vapor transmission through the bioplastic films [44]. The addition of CNPs as filler material is found to reduce the WVP of APSS bioplastic nano films. The WVP of the APSS bioplastic nano film with 1 wt% CNPs reduced by 14.31% compared to the APSS bioplastic nano film with 0 wt% CNPs. Moreover, the observations also reveal that the WVP of APSS bioplastic nano films were found to reduce with increase in wt% of CNPs.

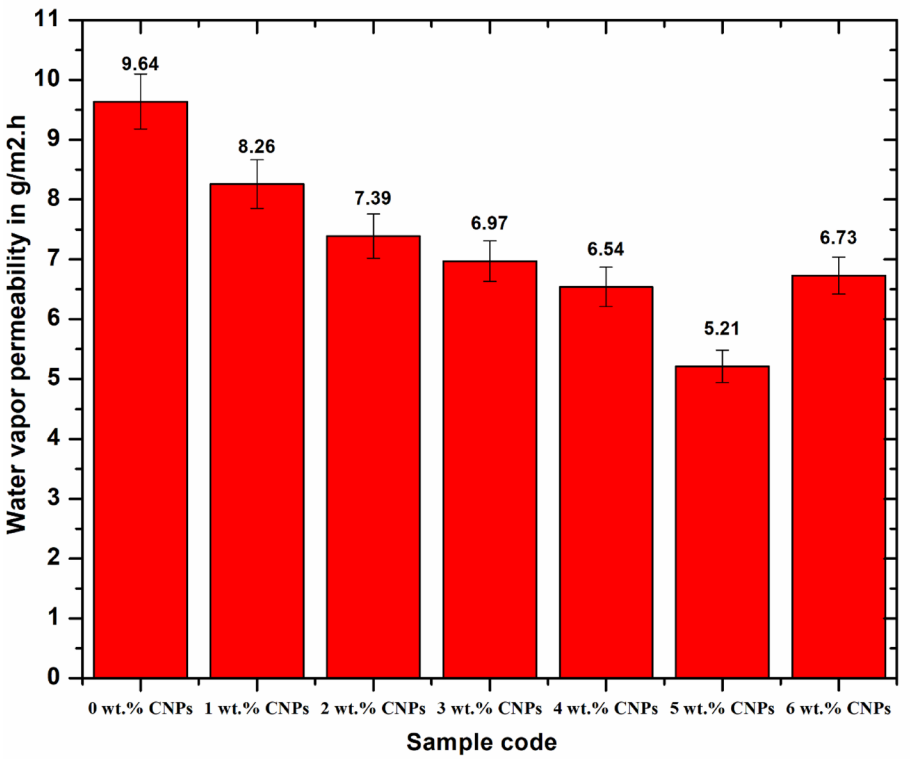


Fig. 8 Water vapor permeability of the APSS bioplastic nano films

The minimum WVP of 5.21 g/m^2. h is obtained for the APSS bioplastic nano film with 5 wt% CNPs. This is owing to the fact that higher wt% of CNPs in APSS bioplastic nano films tend to form more twisted structures in the APSS matrix which makes the water vapours more difficult to pass through the bioplastic films [45]. Further, the presence of CNPs in APSS bioplastic nano films favour the formation of stronger hydrogen bonds with the APSS substrate in the bioplastic films thus reducing the pores and hindering the transmission of water vapours through the bioplastic films. The APSS bioplastic nano film with 6 wt% CNPs showed an increase in WVP by 29.17% compared to APSS bioplastic nano film with 5 wt% CNPs. This is due to the agglomeration of CNPs leading to more voids and permitting the water vapor particles to penetrate easily thus favouring WVP.

3.7 Water absorption of APSS bioplastic nano films

The hydrophilic nature of APSS bioplastic nano films was presented in Fig. 9. The water holding capacity of APSS bioplastic nano film with 0 wt% CNPs was 31.84%. The higher water holding capacity of APSS bioplastic nano film with 0 wt% CNPs is due to the APSS's hydrophilic nature and influence of glycerol plasticizer and vinegar stabilizer in the bioplastic film [45]. The presence of glycerol plasticizer and vinegar stabilizer in the APSS bioplastic nano film with 0 wt% CNPs in addition to the hydrophilic nature of APSS further weakens the bond between the APSS molecules and creates more space between the molecular chains promoting water absorption characteristics of the bioplastic films. The addition of CNPs as filler material is found to reduce the water absorption characteristics of APSS bioplastic nano films. The water holding capacity of the APSS bioplastic nano film with 1 wt% CNPs reduced by 10.23% compared to the APSS bioplastic nano film with 0 wt% CNPs. Moreover, the observations also reveal that the water holding capacity of APSS bioplastic nano films were found to reduce with increase in wt% of CNPs. The minimum water holding capacity of 19.67% is obtained for the APSS bioplastic nano film with 5 wt% CNPs. This decrease in water holding capacity of the APSS bioplastic nano films with addition of CNPs is due to the fact that the presence of CNPs in the APSS substrate favours the hydrogen bonding among the molecules of CNPs and APSS [46]. The CNPs interact through hydrogen bonding with carboxyl and

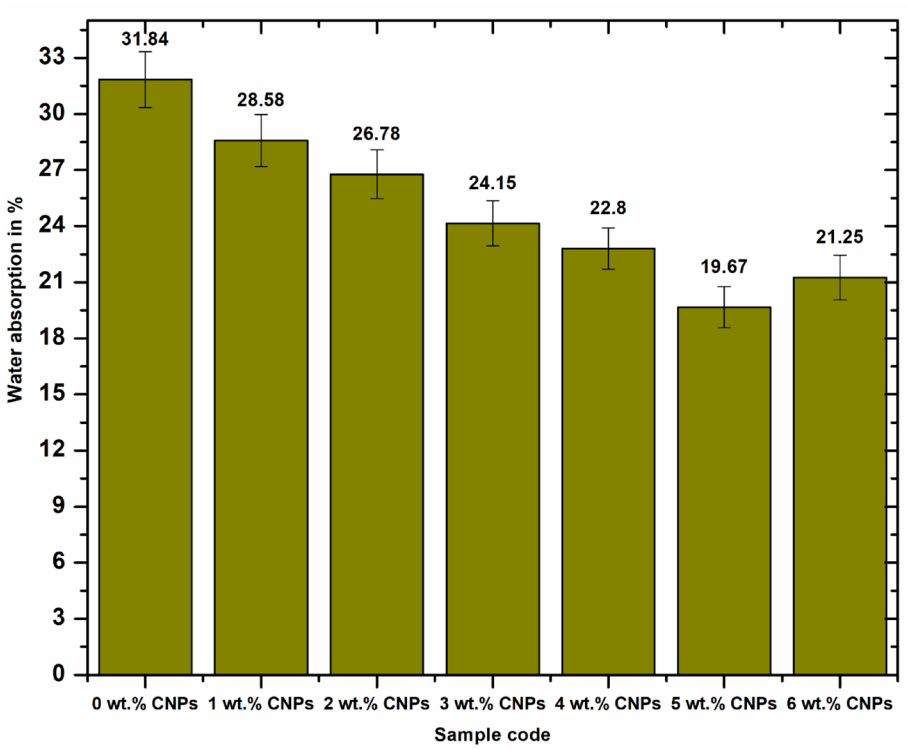


Fig. 9 Water absorption of the APSS bioplastic nano films

hydroxyl clusters of the APSS thereby increasing the density and reducing the voids decreasing the hydrophilic nature of the APSS bioplastic nano films with CNPs as filler material. Further, the presence of CNPs in APSS bioplastic nano films favour the formation of stronger hydrogen bonds with the APSS substrate in the bioplastic films thus reducing the pores and hindering the penetration of water particles through the bioplastic films. The APSS bioplastic nano film with 6 wt% CNPs showed an increase in water absorption behaviour by 7.87% compared to APSS bioplastic nano film with 5 wt% CNPs. This is due to the agglomeration of CNPs leading to more voids and permitting the water particles to penetrate easily thus favouring water absorption characteristics.

3.8 FTIR spectroscopy of APSS bioplastic nano films

Figure 10 presents the FTIR spectra of the APSS bioplastic nano films with 0 wt% CNPs and 5 wt% CNPs. The characteristic peaks along with its functional groups were listed in Table 2. During the processing of APSS bioplastic nano films the interaction of hydroxyl groups between the APSS, glycerol plasticizer, vinegar stabilizer and CNPs have generated stronger hydrogen bonds leading to a functionally stable bioplastic film. Chitosan is a cellulose derived hydroxyl (−OH) and carbonyl (C=O) functional group. The −OH group has the strong ability to form hydrogen bond with other molecules in the substrate of the APSS bioplastic nano films [47]. This phenomenon leads to variation in functionalization of the APSS bioplastic nano films with 5 wt% CNPs compared to APSS bioplastic nano films with 0 wt% CNPs. The hydrogen coupling that occurred when the APSS and CNP elements were combined during the APSS bioplastic nano film manufacturing method was the cause of the slightly higher peak in absorbency at the O−H binding in the frequency range of 3200–3700 cm^{-1} [48]. Amylose's couplings with amylopectin, glycerol plasticizer, vinegar stabilizer and CNPs comprises of the hydrogen bonding. The carbonyl group of CNPs react with the APSS, glycerol plasticizer and vinegar stabilizer leading to the formation of widened peak in the frequency range of 2800–2950 cm^{-1} and 1500–1650 cm^{-1} respectively for C-H and C=C bonds with alkene compounds for APSS bioplastic nano films with 5 wt% CNPs [49]. The minor shift in frequency of the O−H, C−H and C=C bindings of APSS bioplastic nano films with 5 wt% CNPs compared

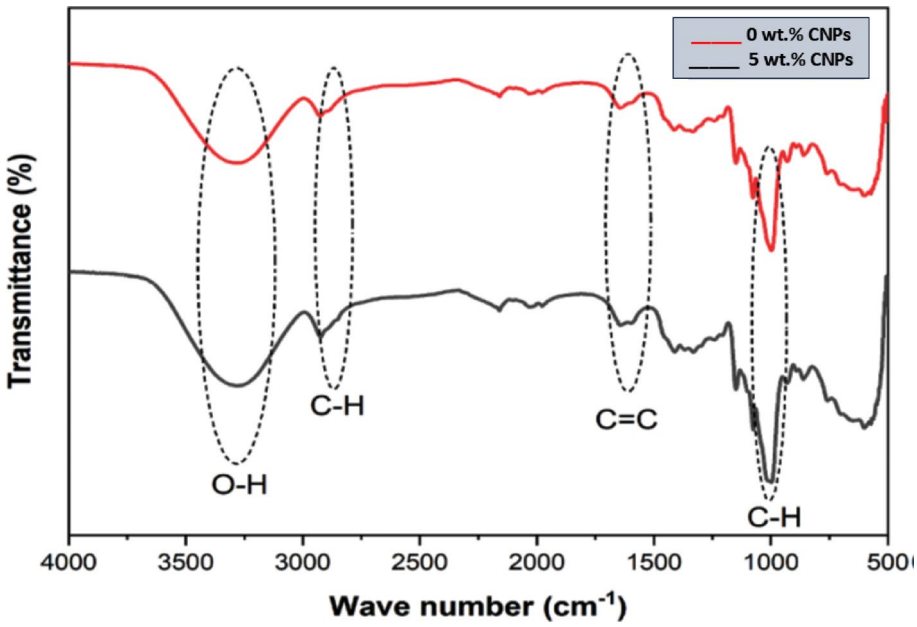


Fig. 10 FTIR spectra of the APSS bioplastic nano films

Table 2 Characteristic peak with its functional group

Wavenumber range in cm^{-1}	Functional group	Reference
3200–3700	O–H	[47]
2800–2950	C–H	[47]
1500–1650	C=C	[48]
985	C–H	[49]

to APSS bioplastic nano films with 0 wt% CNPs is owing to the decrease in molecular mass in the bioplastic film and an indicator for new molecular couplings in the APSS bioplastic nano films with 5 wt% CNPs.

3.9 Thermogravimetric analysis of APSS bioplastic nano films

The thermogravimetric plot of APSS bioplastic nano films with 0 wt% CNPs and 5 wt% CNPs were presented in Fig. 11. The APSS bioplastic nano films with 0 wt% CNPs exhibit two phases of thermal decomposition. Both the APSS bioplastic films exhibit an initial weight loss at around 108 °C which relates to its dehydration process of water molecules and acetic acid which amounts to a weight loss of 9% of bioplastic films [50]. The second phase of degradation of APSS bioplastic nano films with 0 wt% CNPs from 110 °C relates to the damage caused to amylose in the APSS made up of glucose molecules. The third phase of degradation of APSS bioplastic nano films with 0 wt% CNPs from 168 °C corresponds to the deterioration of amylopectin in the APSS which is a polysaccharide and glycerol plasticizer. The APSS bioplastic nano films with 5 wt% CNPs exhibit a weight loss of 21% between 118 °C and 214 °C relating to the deprivation of part of amylose, amylopectin glucose molecules and glycerol plasticizer in the APSS [51]. In the temperature range between 215 °C and 324 °C the glucosamine units of chitosan and remaining parts of amylose and amylopectin glucose molecules degrade amounting to a weight loss of 48%. The derivative thermogram plot of APSS bioplastic nano films with 0 wt% CNPs and 5 wt% CNPs show an infliction point at 289 °C and 293 °C respectively which indicate a shift in degradation pattern for both the bioplastic films. The initial peak in the DTG curve at around 95 °C corresponds to the evaporation of water with the degradation rate of −0.19 to −0.21%min^{-1}. The maximum degradation occurs at around 270–320 °C with the degradation rate of −0.80%min^{-1} and −0.83% min^{-1} for bioplastics with and without CNPs respectively. The residual char at 549.8 °C for both APSS bioplastic nano films with 0 wt% CNPs and 5 wt% CNPs are 8%

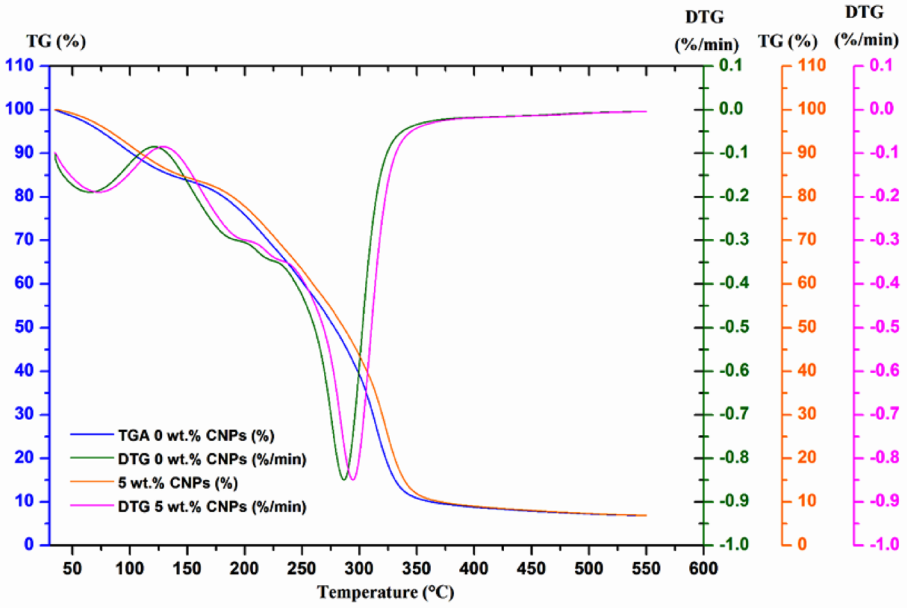


Fig. 11 Thermal stability of the APSS bioplastic nano films

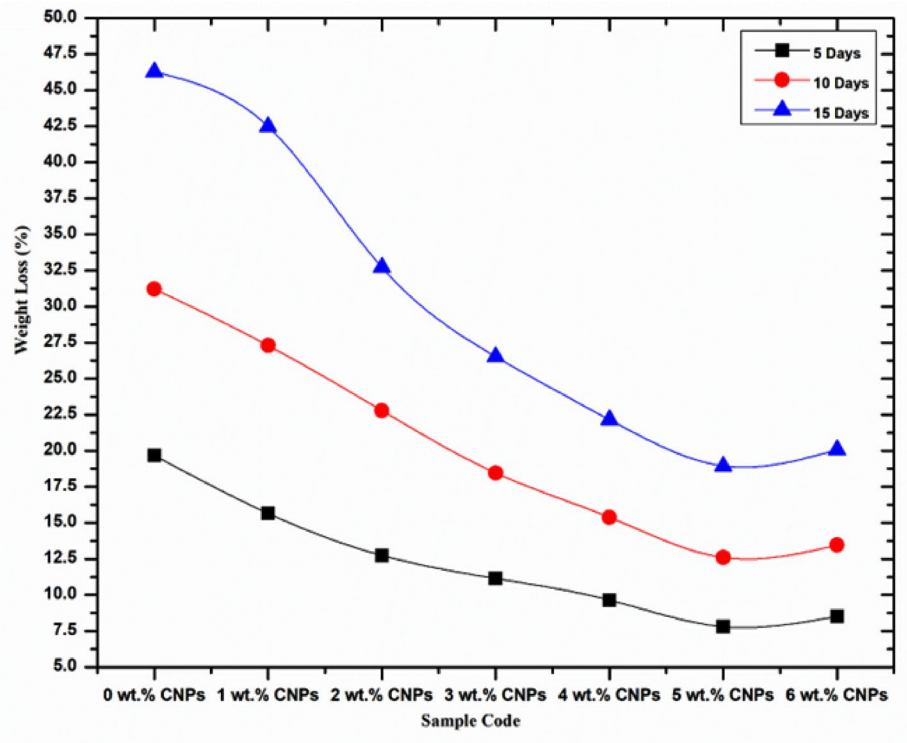


Fig. 12 Biodegradability of the APSS bioplastic nano films

respectively [52]. The percentage of residual char for APSS bioplastic nano films shows its better thermal resistance for packaging applications.

3.10 Biodegradability of APSS bioplastic nano films

The biodegradability of APSS bioplastic nano films was presented in Fig. 12. The biodegradability of APSS bioplastic nano films was observed over a period of 15 days with every 5-day duration. And the weight loss in

percentage was noted. The biodegradability of APSS bioplastic nano film with 0 wt% CNPs in terms of weight loss % at 15th day was 46.28%. The higher biodegradability of APSS bioplastic nano film with 0 wt% CNPs is due to the influence of glycerol plasticizer and vinegar stabilizer in the bioplastic film. The presence of glycerol plasticizer and vinegar stabilizer in the APSS bioplastic nano film with 0 wt% CNPs weakens the bond between the APSS molecules creates space between the molecular chains promoting water absorption from the soil and favour the entry of microbes into the APSS chain thus accelerating hydrolysis and deterioration [53]. The addition of CNPs as filler material is found to reduce the biodegradability of APSS bioplastic nano films. The biodegradability of the APSS bioplastic nano film with 1 wt% CNPs reduced by 8.21% compared to the APSS bioplastic nano film with 0 wt% CNPs. Moreover, the observations also reveal that the biodegradability of APSS bioplastic nano films were found to reduce with increase in wt% of CNPs. The minimum biodegradability of 18.94% is obtained for the APSS bioplastic nano film with 5 wt% CNPs. This is owing to the fact that higher wt% of CNPs in APSS bioplastic nano films favour the formation of stronger hydrogen bonds with the APSS substrate in the bioplastic films thus reducing the pores and hindering the entry of water particles and microbes from the soil into the bioplastic films thus delaying the biodegradation process [54]. The APSS bioplastic nano film with 6 wt% CNPs showed an increase in biodegradability by 5.96% compared to APSS bioplastic nano film with 5 wt% CNPs. This is due to the agglomeration of CNPs leading to more voids and permitting the water particles and microbes to penetrate easily thus favouring the biodegradation process of APSS bioplastic nano film with 6 wt% CNPs.

4 Conclusion

This study demonstrates the potential of *Amorphophallus paeoniifolius* stem starch-based bioplastic films with CNPs as filler material, vinegar as stabilizer and glycerol as plasticizer as a sustainable solution to the escalating plastic waste crisis. Influence of various CNP filler concentration on the tensile, physical, morphological, water captivation, water vapour transmission, biodegradability and thermal characteristics of the casted bioplastic films were observed. The findings reveal that the bioplastic film with 5 wt% of CNPs possessed maximum improved functional characteristics among the tested specimens. The bioplastic film with 5 wt% of CNPs showed an improvement in tensile strength by 49.63%, tensile modulus by 138.23%, thickness by 52.78%, density by 7.53% and thermal stability by 3.1% compared to the bioplastic film with 0 wt% of CNPs. On the other hand, certain properties of the bioplastic film with 5 wt% of CNPs like elongation at break, solubility, water vapour transmission rate, water absorption and biodegradability decreased by 36.93%, 25.01%, 45.95%, 38.23% and 59.07% respectively compared to the bioplastic film with 0 wt% of CNPs. The surface SEM images and FTIR spectroscopy of the bioplastic film with 5 wt% of CNPs ensure the homogeneous dispersion of CNPs along the matrix and ensures the presence of hydroxyl and carbonyl groups respectively. The bioplastic film with 5 wt% of CNPs demonstrates resilience, adaptability and commercial viability making it suitable for flexible packaging and disposable utensils. This study contributes to the development of sustainable bioplastics, offering a viable solution to the global plastic pollution crisis.

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Author contributions Justin Abraham Sajin: Investigation (Lead); Writing—original draft of manuscript (Equal); Raveendra Kurup Anish: Investigation (Supporting); Writing—original draft of manuscript (Equal); Bright Brailson Mansingh: Investigation (Supporting); Writing—original draft of manuscript (Equal); Mathew Jude Joseph: Supervision (Equal); Writing—review and editing (Equal); Vishnu Nandan Harikumar: Investigation (supporting); Writing—review and editing (supporting); Murugan Sundaram Senthil Saravanan: Validation (supporting); Writing—review and editing (supporting); Inès Hammami: Validation (supporting); Writing—review and editing (supporting); Ahmed Mellouli: Validation (supporting); Writing—review and editing (supporting); Joseph Selvi Binoj: Supervision (Equal); Writing—review and editing (Equal). Haiter Lenin Allasi: Validation, Review & Editing.

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Data availability The datasets used and/or analysed during the current study available from the corresponding author on reasonable request.

Declarations

Ethics approval and consent to participate The plant material (Amorphophallus paeoniifolius) used in this study was obtained from an authorized commercial/local supplier and was not collected from wild populations. Therefore, no collection permits or licences were required. Consequently, compliance with local or national guidelines for wild plant collection is not applicable.

Consent for publication Not applicable.

Competing interests No potential conflict of interest was reported by the authors.

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