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Improving Mechanical and Morphological Characteristics Pva/Sio₂ Nanocomposites for Food Packing Applications

Ohood Hmaizah Sabr, Asra Ali Hussein and Massar Najim Obaid

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College of Materials Engineering, University of Babylon, Iraq

Email: mat.ehood.h@uobabylon.edu.iq

Abstract. This study deals with using nano-SiO₂ to enhance the characteristics of PVA for food packing applications. Thin-film of Polyvinyl alcohol (PVA)/ nano-silica (nano-SiO₂) was prepared by adding different weights of silica (0, 5, 7, 9) wt. %. Differential scanning calorimetry, atomic force microscopy (AFM), FTIR, and mechanical properties such as tensile strength, modulus, elongation at break, and micro-hardness used to examine the characteristics of PVA/nano-SiO₂ films. The results revealed a dramatic improvement in the PVA/nano-SiO₂ mechanical properties, increasing nano-SiO₂ content to 7 wt %, the value of tensile strength increasing from 62 MPa to 143 MPa. While increasing SiO_2 content to 9 wt.%, the tensile strength decreased to 65 MPa. The study also showed that the value of elongation at break and modulus of elasticity of film increment with increases in the nano-SiO₂ content. There was also an increase in the crystallinity from 0.97 (for pure PVA) to 1.154 at 7wt. % nano-SiO₂. Crystallinity, however, decreased to 0.012 with the increase of SiO₂ to 9 wt. %. FTIR results revealed that there was physical interaction between nano-SiO₂ and PVA. AFM result showed less roughness at 7wt. % from nano-SiO₂. Generally, the results showed that the concentration of 7% of the nano –SiO₂ in thin films resulted in a significant improvement in the mechanical properties of the films. These findings suggest that nano-SiO₂ can be useful as food packaging material to maintain food quality.

Keywords. PVA, SiO₂, Thin Film, FTIR, DSC.

1. Introduction

Food-packaging materials crafted from oil-primarily based polymer films show exceptional practical properties. Typically, the non-biodegradability of these materials reasons environmental pollutants and extreme environmental issues [1, 2, 3]. Food packaging materials with adequate mechanical strength, thermal stability, biodegradability, barrier properties, and antioxidant and antibacterial properties are essential for food safety and crucial for extending packaged foods' shelf life. Recently, the packaging industries' materials are relatively cheap and suitable for their good process capability and durability [4]. The development of new packaging materials is increasingly needed because petroleum-based plastics are non-biodegradable, therefore, the environmental pollution caused by traditional plastic packaging is becoming increasingly severe. Nanotechnology can be defined as an applied sciences and technology field that deals with different materials in the nanoscales ranges [5]. Polymer nanocomposite structure has matrix from polymer reinforced with nanofillers such as (nanotubes, nanoparticles, and nanofibers etc.). Significant applications of nanomaterial in physical, chemical, and

Content from this work may be used under the terms of the Creative Commons Attribution 3.0 licence. Any further distribution of this work must maintain attribution to the author(s) and the title of the work, journal citation and DOI. Published under licence by IOP Publishing Ltd 1 biological systems[6], Because of the structure of nanoscale size, it has been improved surface characteristics of nano-fillers, and the interactions between the matrix and nanoparticles, have greatly improved the properties of nanocomposite, such as thermal and mechanical properties. Added many kinds of nanoparticles to the matrix of PVA, such as silica [7], carbon-based nanoparticles [8,9],metal oxides [10, 11], montmorillonite [12,13] metal sulfides [14, 15] and organic nanoparticles [16, 17].

Polyvinyl alcohol is biodegradable, biocompatible, nontoxic, and a water-soluble polymer, due to its outstanding properties, it has been studied as a host matrix for many nano-fillers. [18, 19]. PVA used in biomedical applications such as wound dressing, synthetic vitreous body, drug delivery, artificial organs, and cardiovascular devices, etc. [19,20]

With many properties such as biodegradability and biocompatibility, silica nanoparticles have improved industrial packaged foods' shelf life. Also, silica nanoparticles contain large surfaces and large numbers of silanol groups, which can form a tighter interface with the polymers in the composites, increase the effect of hydrogen bonding, and alter the crystalline behaviors of the polymeric matrix [21].

Liu et al. studied PVA/titanium dioxide nanocomposites with surface-carboxylated nano-TiO₂, the nanocomposite prepared by a solution-blend film-casting method. The result shows that with increasing carboxylated nano-TiO₂ content, loss tangent decreased, and storage modulus increased, significantly enhancing tensile strength on the nanocomposites [22]. Nagalakshmi, N et al. studied preparation nanocomposite from PVA/NiO and the effect of nickel oxide. Due to the reaction between NiO and PVA, the resulting PVA/NiO nanocomposite became amorphous. Increased NiO content resulted in improved thermal stability of PVA/NiO nanocomposite [23].

Mohaddeseh Kariminejad et al. investigated the effects of nano-SiO2 on the physicochemical and structural properties of gelatin/polyvinyl alcohol composite films, the result showed improved properties film through the use of nano-SiO2 could make the films more effective as food packaging [24]. Afifah Iswara Aji et al. studied the effect of SiO2 on the mechanical properties of Semi-Refined Iota Carrageenan for food packaging. The result showed improved tensile strength by adding 0.5% SiO2 to the matrix.[25]. This research aims to prepare samples of PVA with a low content of nano-silica to improve the mechanical properties of the film for use in packaging applications. This study selected nano-SiO₂ because of its unique properties. Additionally, compared with other nanofillers, nano-SiO₂ has a significant effect on improving the mechanical properties of the matrix with lower addition.

2. Experimental part

Polyvinylalcohol (PVA) provided by the Amir Kabir Petrochemical Company for Granular Materials, Iran, and Silica Nanoparticles (SiO₂) obtained from the Central Drug House (P) Ltd.

To prepare the polyvinyl alcohol solution (PVA). Firstly, dissolved 10 g from polyvinyl alcohol in 100 ml of distilled water, and then heating at 90 °C for 1 hr. To prepare the nano-SiO₂ solution by mixing distilled water and nano-SiO₂, using an ultrasonic device at 30% of the total energy (1200w) for 15 min at 40°. The composite film solutions were prepared by mixing 10 g of PVA and various amounts of nano-SiO₂ (0, 5, 7, 9) wt.% and stirred the solutions for 1 hr. at room temperature, then the solutions poured onto glass plates (25 x25) cm and let it to dry for 48 hr. at room temperature. Finally, the prepared sheet peeled off to be characterized.

3. Characterizations

3.1. Differential scanning calorimeter (DSC) result

Differential scanning calorimeter (DSC) measurements were carried out using DSC i-series (CW-05G) under the inert gas atmosphere available at the University of Babylon's Basic Education college laboratory. The prepared $(8-10)\pm 0.5$ mg weight samples were mounted on aluminum pans and heated up from (25 to 250 °C) with a heating rate of 10 °C/min.

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The crystallinity of the sample was calculated as:

$$C (\%) = (\Delta H_{\rm f} / \Delta H^{\circ}_{\rm f}) * 100\%$$
(1)

where: ΔH_{f} : Enthalpy of melting for PVA and ΔH°_{f} : Enthalpy of melting for PVA is 168 J/g at 100% crystallization.

3.2. Fourier-transform infrared (FTIR)

FT-IR is a technique used to obtain an infrared spectrum for the absorption or emission of a solid, liquid, or gaseous substance and used to analyze highly complex mixtures.

3.3. Atomic force microscopy (AFM)

AFM is typically used for determining the morphology and size of nanoparticles in three dimensions. Since the AFM precept is based totally on the mechanical touch among the sample and tip, the dimension of particles in the nanometer scale is much stricken by sample-tip interaction.

3.4. Scanning electron microscope

The microstructural changes and the cross-sectional topography of films studied using SEM inspection.

3.5. Tensile test

Packaging films made from different polymers may be exposed to different kinds of external pressures and stresses during use, so mechanical properties are among the significant factors used to identify the efficiency of the film in packaging [24].

Tensile properties such as tensile strength, elongation, and modulus of elasticity are measured using a microcomputer-controlled electronic universal testing machine model (WDW-5E) according to ASTM commonplace methodology D882-09 (2009) (Standard, 2009). The measure was performed at applied load (5KN) and speed value (2mm/min). The films were cut (50 mm x1 mm) strips. The test is performed using at least three samples, and therefore, the mean results were obtained. As a result of packaging materials, like films product of totally different polymers, could also be exposed to different types of external pressures and stresses throughout use, mechanical properties were among the most factors wont to confirm the potency of packaging (Pawde, and Deshmukh,2008, Tang et al., 2008) [26].

3.6. Microhardness test

The samples' hardness is measured by using a microhardness Vickers device manufactured in China and located in the laboratories of Materials Engineering College/ University of Babylon, the loads ranging from (0.98-1000)N. Microhardness Vickers is a method to measure hardness when test samples are very small or thin.

4. Results and discussion

4.1. Differential scanning calorimeter (DSC) result

Table (1) shows the crystallinity and melting temperature (Tm) for each sample, and they are used to analyze the effect of SiO₂ nanoparticle content on the behavior of crystallization of PVA/SiO₂ nanocomposite. Figure 1 shows an increase in Tm of nanocomposites according to pure PVA having melting temperature 227.12°C were increased with increasing SiO₂ content, this is due to good dispersion of nanoparticles in the polymer matrix. When the silica content increases to 0.09, an agglomeration will occur, leading to a decrease in melting point. By increasing SiO₂ nanoparticle content to (7 wt.%), the crystallinity increases due to dispersion of nanoparticle in PVA matrices uniformly, it was not restricted to the movement of molecular chains of polyvinyl alcohol. At higher SiO₂ content (9wt.%), however, the SiO₂ nanoparticles act as a barrier that may restrict the thermal movement of the molecular chains of PVA. This may noticeably decrease the crystallinity.

results mentioned above, more explanation can be provided on improving PVA/nano-SiO2 films' mechanical properties.

Table 1. Show T _m ,	Enthalpy of melting,	and Crystallinity for
pure PVA and nano	composite.	

Samples	Tm	Enthalpy of melting	Crystallinity
	(°C)	$\Delta H(J/g)$	%
PVA	227.12	-1.64	0.97
PVA+0.05 SiO ₂	230	-1.96	1.16
PVA+0.07 SiO ₂	229.94	-1.94	1.154
PVA+0.09 SiO ₂	207.07	-0.02	0.012



Figure 1. DSC thermos gram of heating cycle for a. Pure PVA, b. PVA+5% SiO₂, c. PVA+7%SiO₂ and d.PVA+9%SiO₂.

4.2. Fourier-transform infrared (FTIR) analysis

According to the values of bands recorded by Fourier transform spectroscopy (FTIR) for the pure PVA and (PVA/SiO_2) nanocomposite prepared by the solvent casting method with various content of SiO_2 are summarized in Table 2 derived from figure 2. FTIR test for polyvinyl alcohol showed

several bands such as (hydroxyl groups) bands at 3541 cm⁻¹, (CH₂ asymmetric stretching) bands at 2767.85 cm⁻¹, (C=O stretching) bands at 1666.5 cm⁻¹, and the band at 1564.27 cm⁻¹ for (CH₂ bending). From Figure 2, FTIR of the PVA/SiO₂ nanocomposite, it can also be seen that broadband near 1047 cm⁻¹ and 918 cm⁻¹ was the result of the Si-Si bonds because the peaks belonged to the Si–O–Si stretching vibration [27]. Shifting spectra to lower wavenumbers was also observed, as shown in Figure 2. FTIR test result showed that there was no reaction between them, this was due to a lack of appearance of bonds. The results, however, showed there were some bonds shifting, which was triggered by physical interaction.



Figure 2. The FTIR for PVA and PVA/SiO₂.

Table 2. Show the absorption bands of the IR spectrum for pure PVA and nanocomposite.

kinds of bond	Standard PVA	Exp. PVA	PVA+ Sio ₂
	[Alireza Kharazmi,et.al] [28]	1	_
OH stretching	3280	3541	3535
CH stretching	2917	2767.85	2899.01
C=0	1690	1666.5	1654.92
CH ₂ bending	1425	1564.27	1560.41
Si-O-Si	-	-	1047.35
			918 12

4.3. Atomic force microscopy (AFM)

Figures 3 shows the PVA topography with (5, 7, 9% wt.) SiO₂ nanoparticles. It is undoubtedly shown that the nanoparticles are totally embedded in the PVA matrix, they are homogenous and do not aggregate out of the system. Strong interaction between the PVA and nanoparticle was produced due to properly dispersing SiO₂ nanoparticles in the PVA matrix. Additionally, yellow tips disappeared

with increased SiO_2 nanoparticles. Besides, nanoparticles' addition has changed the prepared samples' surface roughness, as shown in Table (3). The optimum sample at 0.07 SiO_2 had a minimum surface roughness value, therefore, it clearly obtained good mechanical properties.

Table 3. The average roughness, root mean square for pure PVA and nanocomposite.

Sample	Sa (Average Roughness)	Sq. (Root Mean Square)
PVA	5.02	6.51
PVA+0.05 SiO ₂	5.31	6.58
PVA+0.07 SiO ₂	2.31	2.77
PVA+0.09 SiO ₂	4.07	5.54



(c)

0.00.00

(d)

0.00.00

Figure 3. AFM Topography of (a) pure PVA, (b) PVA+5%wt. SiO₂, (c) PVA+7 %wt. SiO₂ and (d) PVA+9 %wt. SiO₂.

4.4. Scanning electron microscope (SEM) test

Figure 4 shows that the pure membrane's surface is smooth while adding Nano- SiO_2 leads to a firm and compacted surface for the membrane.

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Figure 4. SEM images of (a) pure PVA, (b)) PVA+ 5% $SiO_{2,}(c)$ PVA+ 7% SiO_{2} and (d) PVA+ 9% SiO_{2} .

4.5. Tensile tests results

The effect of SiO₂ nanoparticles on the tensile strength of PVA is shown in Figure (5). The results revealed that there was an increase in the tensile strength with increasing the SiO2 nanoparticle content. The tensile strength is 80 MPa at 0.05 SiO₂, and 143 MPa at 0.07 SiO2 content. As shown in the thermal investigation, the increase in the crystallinity led to improved intermolecular bonding between nanoparticles and matrix, so it led to an increase in tensile strength. While the value of TS at 0.09 from nano-silica is 65 MPa, a decrease in the tensile strength at these percent remains higher than TS of the pure film due to the agglomeration of nano-specks, indicating that PVA cannot prompt silicon dioxide formation. This result agrees with Xiangmin Xu. [29], Mohaddeseh Kariminejad et al. (2018) [24]

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Figure 5. The effect of Sio₂ nanoparticle on the tensile strength of pure PVA.

Figure 6 shows that the increase in the elongation of the nanocomposite with increase nano-SiO₂ content. The most significant value of elongation was observed for 0.07 of nano-SiO₂ (1391%). This result is due to the high resistance of PVA and the presence of nano-SiO₂, this result agrees with Mohaddeseh Kariminejad et al. (2018) [24]. On the other hand, increasing the content of nano-SiO₂ to 0.09 leads to a decrease in the elongation at break. This was resulted due to the agglomeration of nano-SiO₂ [30]. Elastic modulus increases with increasing silica content, but elastic modulus decreases at 0.09 from silica due to the agglomeration of nanoparticles, as shown in Figure 7. This result agrees with Shadpour Mallakpour et al. (2018)[31]



Figure 6. The effect of Sio₂ nanoparticle on the elongation at the break of pure PVA.

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Figure 7. The effect of Sio₂ nanoparticle on the modulus of pure PVA.

4.6. Micro hardness test

With the nano-SiO₂ content increase, hardness increased, as shown in Figure 8. This increase in hardness due to the incorporation of rigid nanoparticle in the matrix. This result agrees with Chao Wang et al. [30].

Figure 8. The effect of Sio₂ nanoparticle on the microhardness of pure PVA.

5. Conclusions

This study examined the PVA/nano-SiO₂ films prepared by the solvent casting method. The results showed that the nanofilms' mechanical properties improved from 60 MPa to 143 when the nano-SiO₂ content increased to 7wt %. With increased nanoparticle content to 9 wt.%, tensile strength decreased

to 65 MPa. Concerning the crystallinity of the nanocomposite with concentration 7 wt. % of nano-SiO₂ increased from 0.97% to 1.154%. Additionally, FTIR figures showed physical interaction between PVA and nano SiO₂ because there was no bond appeared. Resulted of AFM showed that the good dispersion of SiO₂ nanoparticles in the PVA matrix leads to a strong interaction between the SiO₂ nanoparticles and the PVA matrix and that the disappeared of yellow tips obviously with increased SiO₂ nanoparticles. To summarize, the addition of SiO₂ nanoparticle enhances the PVA film's characteristics prepared by the solvent casting method. So, it is a good idea to improve the characteristic of polyvinylalcohol.

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