

Mechanical and Thermal Stability Studies of PVB/SiO₂ Nanocomposite and Their Application on Polyester Non-woven

Ahmed A. Mousa^{*1,2}, Ahmed H. Moustafa², Hassan A. El-Sayed², Manal G. Mohamed³.

¹MATCO Chemicals SAE, 10th of Ramadan city, Al-Sharqia, Egypt

²Department of Chemistry, Faculty of Science, Zagazig University, Zagazig, Egypt

³ Polymer Lab, Petrochemical Department, Egyptian Petroleum Research Institute, Cairo, Egypt

*Corresponding author: ahmedamoussa38@gmail.com

ABSTRACT: Polyvinyl butyral (PVB) exhibits exceptional characteristics, flexibility, and recyclability. Composite materials can be created with different inorganic species. Both hydrophilic and hydrophobic vinyl butyral groups were found in PVB which act as binders for organic moieties and promoters of polymer sticky. A coupling agent was used to prevent film defects and improve compatibility between the inorganic and organic phases. In this study, the nanocomposites prepared from PVB and silicon dioxide (SiO₂) with different ratios 1%, 3%, 5%, and 7% using solution casting method with and without coupling agent 3-glycidyloxypropyltrimethoxysilane were used as sustainable binding agent for nonwoven polyester. The prepared films were characterized by FTIR and XRD. The morphology was studied using SEM. Thermal stability TGA was studied, and it was found that the coupling agent made the silica well dispersed into the PVB, but it did not affect the increase in the SiO₂ ratio because the silica and the coupling agent formed strong linkages and aggregates inside the PVB. The mechanical properties (tensile strength and hardness) of the composites were also investigated. The main challenge is improving the non-woven durability and mechanical properties of the prepared nanocomposites. The results show the best ratio of SiO₂, which improved the thermal stability of PVB was 3% in the presence of a coupling agent with a weight residue 53.35%. Thermogravimetric analysis (TGA) was performed for the treated non-woven polyester. The most thermally stable sample was PVB/1% SiO₂.

KEYWORDS: Poly (vinyl butyral), Silicon dioxide, coupling agent, mechanical properties, and non-woven polyester.

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I. INTRODUCTION

Polyvinyl butyral (PVB)-based nanocomposites have recently received considerable attention owing to their distinct optical and adhesion characteristics. These nanocomposites also exhibit characteristics that make them resistant to heat, water, and oxidation. Compared to micro composites, the (mechanical, electrical, thermal, rheological, magnetic, electrochemical, and optical) characteristics of the resulting nanocomposites can be drastically changed at lower loadings related to the larger specific surface area in these systems. The primary goal is to produce high-performance polymer nanocomposites that maintain the unique features of nanoparticles while exhibiting excellent nanoparticle dispersion and homogeneous distribution across the polymer matrix [1]. Polyvinyl butyral is rapidly becoming a material of choice in the industry and meets many essential requirements owing to its excellent flexibility, adherence to various surfaces, enhanced optical clarity, high water resistance, high compatibility with other polymers, and good processing capabilities. PVB, a non-toxic, odorless, and ecologically friendly polymer, is commonly used to produce organic and inorganic composite materials [2, 3]. Nanoparticles were added to the polyvinyl butyral matrix to enhance their thermal, mechanical, and barrier capabilities. Surface-modified mesostructured silica particles were used as fillers to significantly improve the thermal and mechanical properties of the composites by increasing the interfacial interaction between the polymer matrix and nanoparticles [4]. Silica nanoparticles (NPs) are currently a hot research topic. Nano-silica has many advantages over other nanoparticles, such as high specific surface area, economic viability, simplicity in industrial manufacturing, and competitive cost [5]. Fibrous nanocomposites made of polymers and silica can improve their mechanical and thermal properties. Additionally, silica can alter hydrophobicity or catalytic activity by increasing or decreasing it. Fumed silica is an amorphous silicon dioxide that can readily be blended with a spinnable polymer solution and can contain particles as small as 8 to 20 nm.

For instance, polyvinyl butyral (PVB) and fumed silica have created nanofibers with regulated wettability investigations and characterization of PVB/silica nanofibers [6].

For example, silica and alumina particles can be added to and mixed with a polymer matrix to significantly improve the electrical, mechanical, and thermal properties of polymer matrix composites. PVB, a thermoplastic polymer, is created when poly (vinyl alcohol) (PVA) and n-butyraldehyde are combined in an acidic environment. At low temperatures, the polymer has a good impact strength and excellent adhesive characteristics with various materials, making this polymer notable (glass, metals, and plastics) [16]. PVB is also used in protective apparel, adhesives, printing inks, coatings, and healthcare. The mechanical characteristics of the PVB matrix are enhanced by adding nanoparticles and nanotubes [7]. According to Chang et al., SiO₂ sol and PVB-based inorganic/organic thin films displayed exceptional hydrophobicity and superior mechanical and optical features. Another study combined a silica sol made from tetraethyl orthosilicate with a PVB ethanol solution to create plasticized PVB-silica nanocomposite films. It was established that films made from PVB-SiO₂ nanocomposite materials <5 wt percent silica were suitable for use as the interlayer for high-performance laminated glass [8,9].

Non-woven textiles, which are fibrous constructions with randomly arranged fibers, exhibit significant qualities and traits that make them suitable for various applications. Therefore, there is a need to enhance the mechanical properties of non-woven fabrics owing to growing interest in personal health and comfort. Surface modification of non-wovens is a creative technique for improving their quality. Textile antennas have recently gained much attention, especially in medicine, military, space exploration, and other applications [10].

The main goal of this research is to enhance the non-woven polyester textile properties by preparing nanocomposites of polyvinyl butyral (PVB) in the presence of different percentages of fumed silica. The nanocomposites were also prepared in the presence of coupling agent 3-glycidyloxypropyltrimethoxysilane.

II. MATERIALS AND METHODS

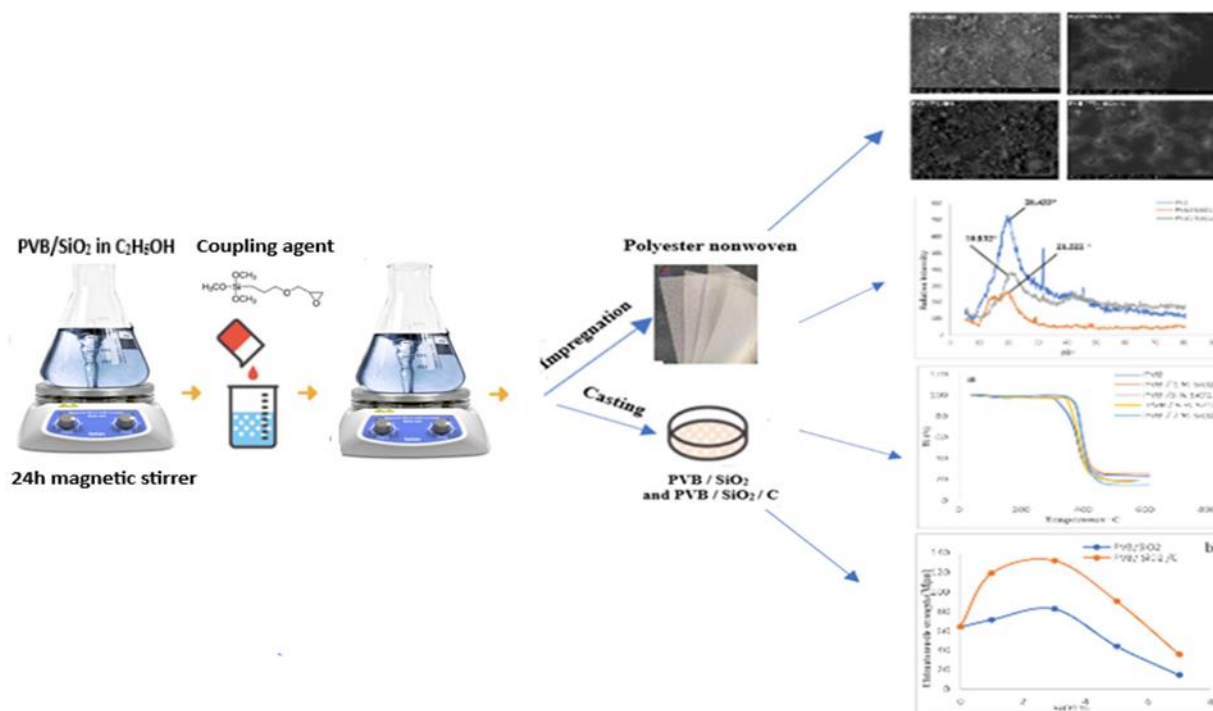
Reagents and chemicals

Polyvinyl butyral PVB (Mowital B60H-from Kuraray Company with the following character contents of vinyl butyral, vinyl alcohol, and vinyl acetate, herein at 75%–81%, 18%–21%, and 1%–4%, respectively), absolute ethanol (El Nasr chemical company Egypt). SiO₂ (Neosyl® GP) was obtained from East Java, Indonesia. The coupling agent, 3-glycidyloxypropyltrimethoxysilane (Dynasylan GLYMO), was obtained from EVONIC. Polyester non-woven (NW) fabric was supplied by El-Mahalla Co., Egypt.

Procedures

Preparation of polyvinyl butyral PVB/SiO₂ nanocomposites

In a flask of 250 mL, 3 g of polyvinyl butyral PVB was dissolved in absolute ethanol (30 mL) by magnetic stirring at room temperature to create a clear viscous solution. Then, Silicon dioxide SiO₂ with different ratios of 1%, 3%, 5%, and 7% were added using a magnetic stirring for 24 h, pure PVB solutions and PVB/silica blends were homogenized. Using the casting method, the mixtures were poured into Petri dishes to evaporate the solvent, and films were obtained. All ingredients were used as obtained and combined in one step without further purification. The quantity of silica nanoparticles was always proportional to the weight of PVB. The prepared nanocomposites were labeled with PVB, PVB/ 1% SiO₂, PVB/ 3% SiO₂, PVB/ 5% SiO₂ and PVB/ 7% SiO₂. The same procedure was repeated in the presence of coupling agent 3-glycidyloxypropyltrimethoxysilane with symbols PVB/ 1% SiO₂/C, PVB/ 3% SiO₂/C, PVB/ 5% SiO₂/C and PVB/ 7% SiO₂/C (Scheme 1).



Scheme 1. Steps of preparation of PVB/SiO₂ Compound and application of fiber.

Characterization of the polyvinyl butyral PVB / SiO₂

ALPHA FTIR, a Bruker spectrophotometer, was used for Fourier transform infrared spectroscopy (FTIR) in the 4000-400 cm⁻¹ range. FESEM QUANTA FEG 250 MADE in the Netherlands examined images taken using a scanning electron microscope (SEM). Thermogravimetric analysis (TGA) was performed using a Setaram detector (LABSYS evo TGA STA DTA / DSC, France). Universal Testing Machine, 10ST Series, Constructed 2019, serial #. ST-AF-01264GB was used to conduct the tensile test, which was practical with 0.2 mm for adhering to two steel strips and fixing them with clamps, following ASTM D1002-01. Tinius Olsen, Made in England; Country of Origin: GB; Serial Number: AP61601 (10000 N). The average of the three prepared samples was used to determine each data point, along with sample evaluation and observational analysis. In addition, the hardness of the composites was evaluated using a Model 470 Type D Durometer following ASTM D2240. Each sample was tested with a minimum 6-mm thickness were recorded. The enduring device used in this project was a QUV cyclic UV enduring device (120 V, 60-Hz model made by the Q Panel Company, United States). This device uses eight UVA 340 fluorescent lamps (UV range, 365–295 nm). As per the ASTM G154-00 recommended procedure, the test boards were left exposed in the QUV Bureau for 120 h. A weathering test was conducted to determine how exposure to these elements affects the weight loss and mechanical characteristics of the HDPE/ESP composites. Equation 1 was used to determine the weight reduction caused by weathering. Percentage of weight loss (%) = $(W_1 - W_2) / W_1 \times 100\%$ (1)

where W_1 and W_2 are the weights of the samples before and after weathering, respectively.

After the sample was weathered, the tensile strength, modulus, and elongation at break were measured.

Processing polyester non-woven samples

The polyester non-woven (NW) samples were cut into equal rectangular pieces of 3 cm × 15 cm and then separately impregnated in a different prepared solution PVB/SiO₂ for 5 min. The samples were air-dried at room temperature to completely dry with a fixed weight before examination.

III. RESULTS AND DISCUSSION

Fourier transform infrared spectroscopy

Figure 1 displays the FTIR spectra of pure PVB and PVB/ SiO₂ nanocomposites with and without coupling agents. The broad peak at 3458 cm⁻¹, which results from the stretching of the hydroxyl group in the polymer, shows the basic backbone structure of the PVB polymer. The peaks at 1433 cm⁻¹ were attributed to the vibrations of CH₂ bending, whereas the peaks at 2921 and 2870 cm⁻¹ were caused by the asymmetric and symmetric stretching of -CH₂, respectively. The peaks observed at 1736 and 1053 cm⁻¹ were caused by the carbonyl stretching of the acetate fragments and the C-O-C group, respectively, in the PVB polymer. The peaks of the SiO₂ groups are ascribed to the asymmetric and symmetric stretching modes detected at 1103 and 810 cm⁻¹.

¹, and the bending mode at 483 cm^{-1} as a weak band appeared to have little impact on the morphologies of the PVB/SiO₂ samples [11]. According to the semi-similarity of the effective groups of the coupling agent in the samples labeled PVB/SiO₂/C, the peaks showed little shift.

The FT-IR spectra of polyester non-woven before and after treatment with the PVB/SiO₂/C nanocomposites are displayed in Figure 2. The bands of non-woven polyester without treatment at 3428 cm^{-1} and 1440 cm^{-1} are attributed to the -OH group and the C-H stretching vibration, respectively. The stretching vibration of the C=O bond was thought to be responsible for the band at 1711 cm^{-1} . The stretching of C-O causes bands at 1239 and 1092 cm^{-1} [12]. In addition, the peaks of the treated non-wovens with the prepared nanocomposites were confirmed by the appearance of the bands belonging to PVB.

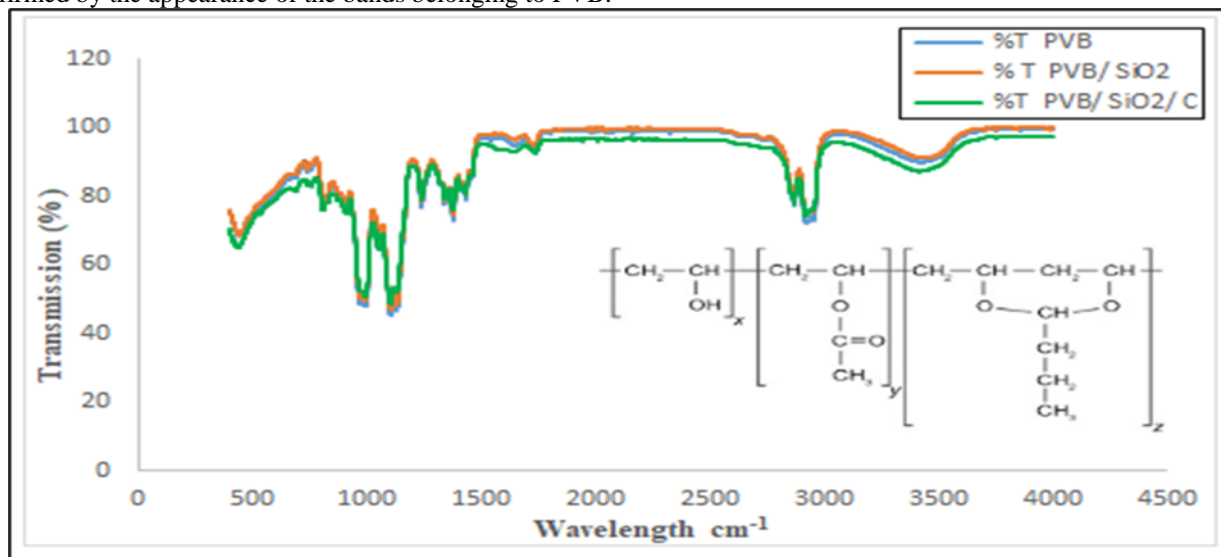


Figure 1. FTIR of PVB and PVB/ SiO₂ with and without coupling agent.

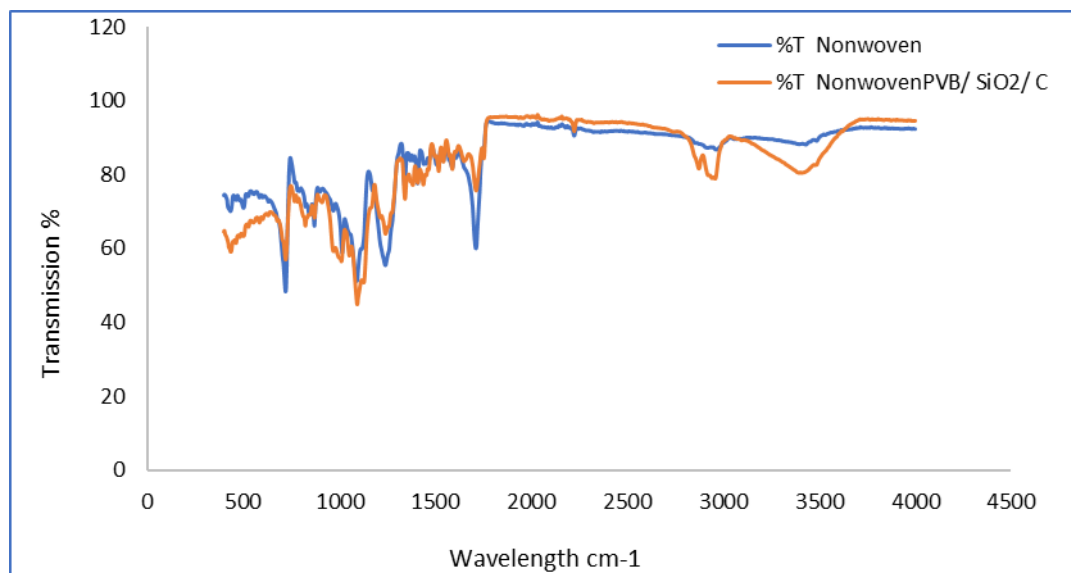


Figure 2. FTIR of Non-woven and Non-woven/ PVB/ SiO₂ with and without coupling agent.

Scanning electron microscope (SEM)

A comparative analysis of the structural properties of SiO₂ and PVB/SiO₂ composites with and without the coupling agent is summarized in the SEM images in Figure 3. As the SiO₂ content of the composite matrix increased, the crack development increased compared with that of the neat PVB film. This may have been due

to the improved interfacial distribution of SiO_2 in the polymer. While In the nanocomposite samples with coupling agents PVB/ SiO_2 /C, the cracking was decreased with increasing the ratio of SiO_2 at 5% and 7%.

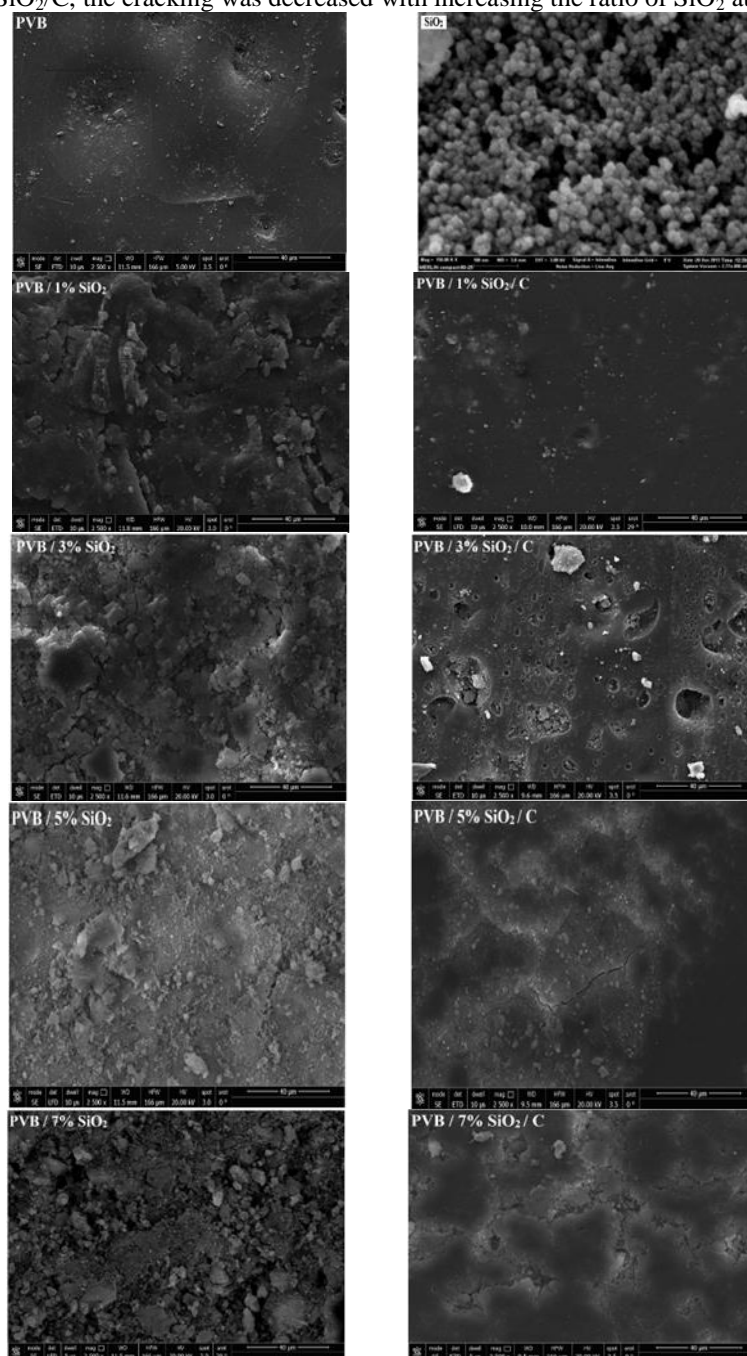


Figure 3. SEM of PVB and PVB with different ratios of SiO_2 with and without coupling agent.

X-Ray diffraction (XRD)

Figure 4 displays the X-ray diffraction (XRD) patterns of the PVB, PVB/ SiO_2 , and PVB/ SiO_2 /C nanocomposites. Owing to the amorphous structure of the polymer, pure PVB displays a distinctive diffraction peak (d_{001} plane) at $2\theta = 20.433^\circ$, with an interplanar distance of 4.34299 [1], for PVB/ SiO_2 , the peak of PVB polymer was shifted from $2\theta = 20.433^\circ$ to $2\theta = 19.832^\circ$ with an interplanar distance of 4.34299 to 4.47310 Å due to the presence of SiO_2 , which penetrates into the polymer and increases the interfacial distances between the polymeric chains. While in the case of using the coupling agent, its presence helped the SiO_2 homogeneously dispersed in the PVB matrixes, and the evidence is the shift of PVB's peak to $2\theta = 21.222^\circ$ and decrease the internal distances to 4.18325 Å in the XRD patterns.

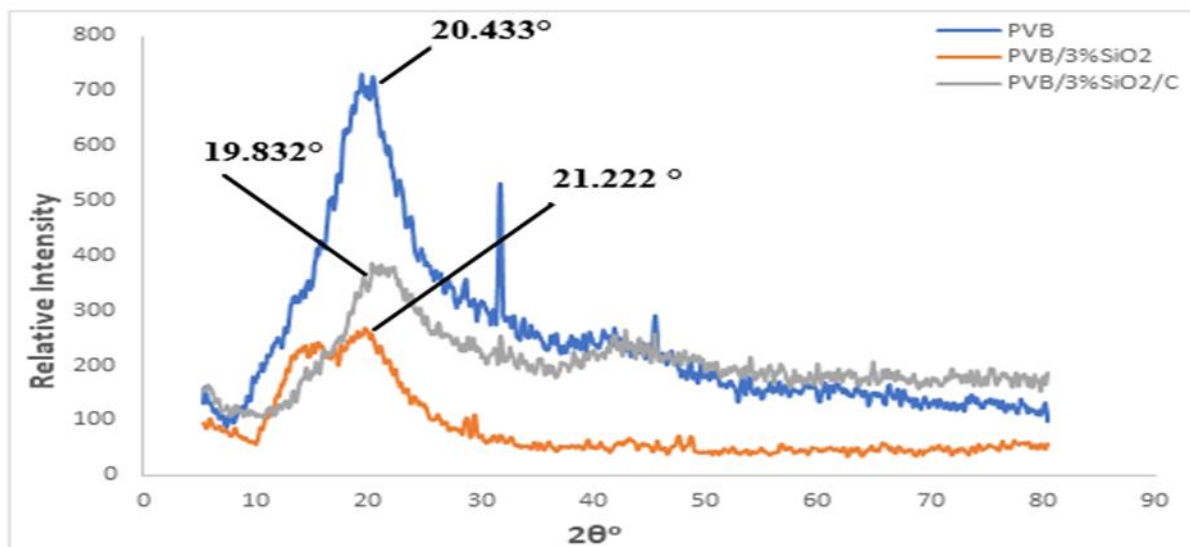


Figure 4. XRD of PVB and PVB with SiO₂ with and without coupling agent.

Thermogravimetric analysis (TGA)

To define the application areas, it is crucial to analyze the thermal properties of polymer-based nanocomposites. Nanocomposite film thermogravimetric analysis (TGA) measurements are shown in Figure 5. In the TGA curves, the weight losses were compared as a function of temperature. Accordingly, thermal degradation of the neat PVB and all samples occurred via a one-step behavior starting at 269–331 °C and nearly finishing at 477–502 °C. The most stable sample was PVB/1% SiO₂ at 613 °C with a residue of 25%, compared to pure PVB with a residue of 23.17%, as shown in Figure 5(a). Conversely, the thermal degradation was enhanced by adding a coupling agent to the prepared nanocomposites PVB/SiO₂. The degradation temperature started between 291–331 °C and ended in the range of 495–528 °C. The best ratio of SiO₂ that improved the thermal stability of PVB was 3% in the presence of a coupling agent with a weight residue of 53.35% Figure 5(b)). By applying the prepared samples to the non-woven without coupling agent Figure 6(a)), all samples did not change until 314 °C and the thermal degradation occurred in one step, onset from 314–424 °C and offset at 402–543 °C. After studying the thermal stability of the non-woven samples at 50% lost, the sample with PVB/1% SiO₂ is the most stable at 594.87 °C, followed by the sample PVB/3% SiO₂ at 465.62 °C. On the other hand, it was found that the most thermally stable sample is PVB/1% SiO₂, which is the residue still 50% until 632 °C while increasing the silica ratio 3%, 5%, and 7% gradually, the residue decreased 42.77 %, 35.03%, and 16.59%, respectively, which indicates that increasing the SiO₂ ratio did not improve the thermal stability of the non-woven. In the case of adding the coupling agent Figure 6(b)), it was found that the thermal stability of the sample of PVB/1% SiO₂ was more thermally stable, with a residual 56% compared to the non-woven matrix of 31.41% at 632 °C and non-woven without coupling agent. This indicates that the coupling agent helped the silica spread into the PVB, but it did not affect with increasing SiO₂ ratio because their bonds with each other were strong, forming aggregates within the PVB. From this study, it is clear that the ideal percentage of SiO₂ to improve the thermal stability properties of PVB is different in the case of the polymer films than when it is applied to the non-woven fabric because of the interference of the nature of the fabric as a polyester. Increasing the percentage of SiO₂ in the case of the polymer decreased the thermal stability due to the agglomerations that occur in the silicon dioxide molecules, but in the case of applying it to the fabric, the agglomerations are relatively reduced at 1% and 3% and begin to increase at the ratios of 5% and 7% of SiO₂.

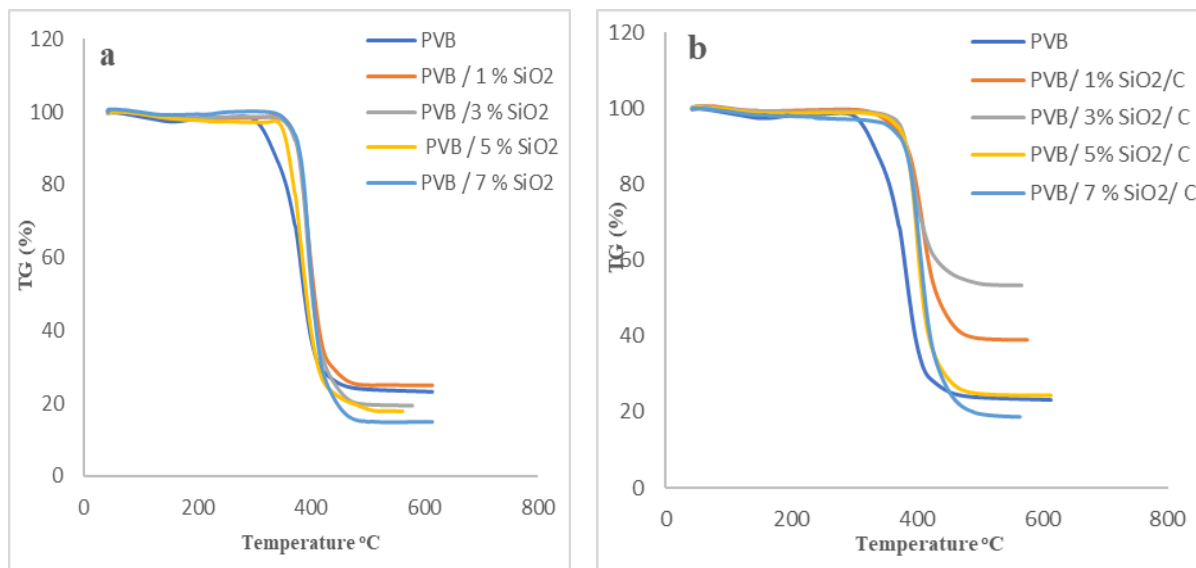


Figure 5. TGA of PVB / SiO₂ with and without coupling agent.

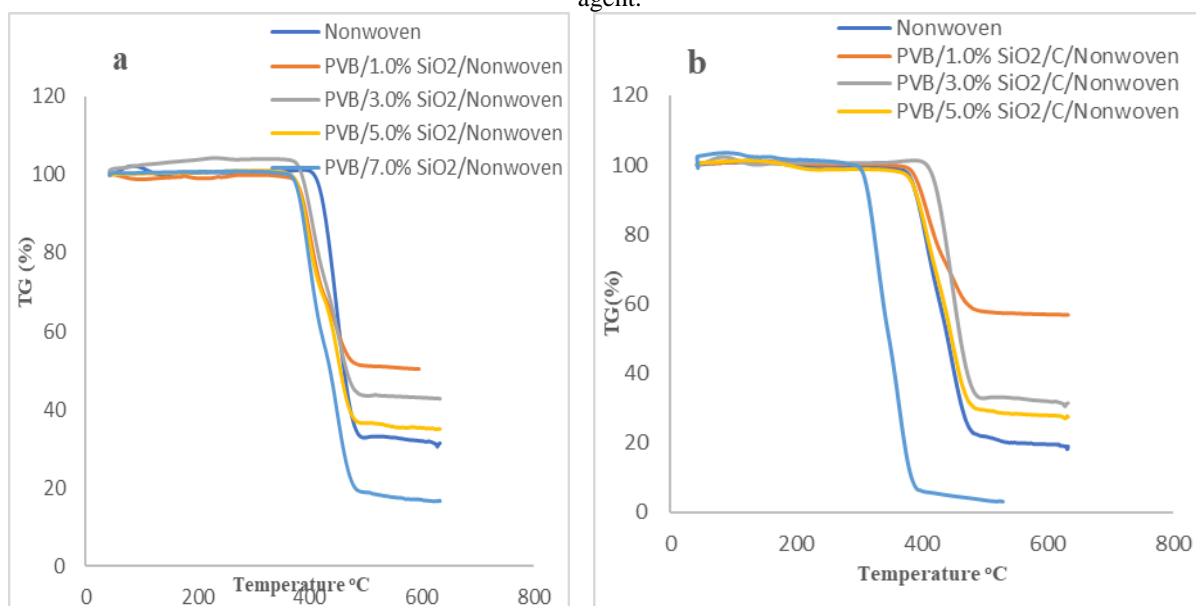


Figure 6. TGA of Non-woven/PVB / SiO₂ with and without coupling agent.

Mechanical properties

The tensile properties of the PVB/silica nanocomposite films were investigated for all samples, as shown in Figure 7, with variations in Young's modulus (YM), ultimate tensile strength (UTS), and elongation at break (EB%). The nanocomposite films showed increased base matrix stiffness with increasing 1% and 3% SiO₂ silica contents. However, the tensile reduction, as well as the cracking, increased with increasing the SiO₂ at 5% and 7%, which was validated through SEM images. Particle-particle bonding increases at high silica loading, resulting in aggregates inside the film, causing the matrix to become overly rigid. Because of this, the entangled polymer chain requires high fracture energy to break and/or stretch, as well as increased resistance to crack formation and lower crack propagation energy [13].

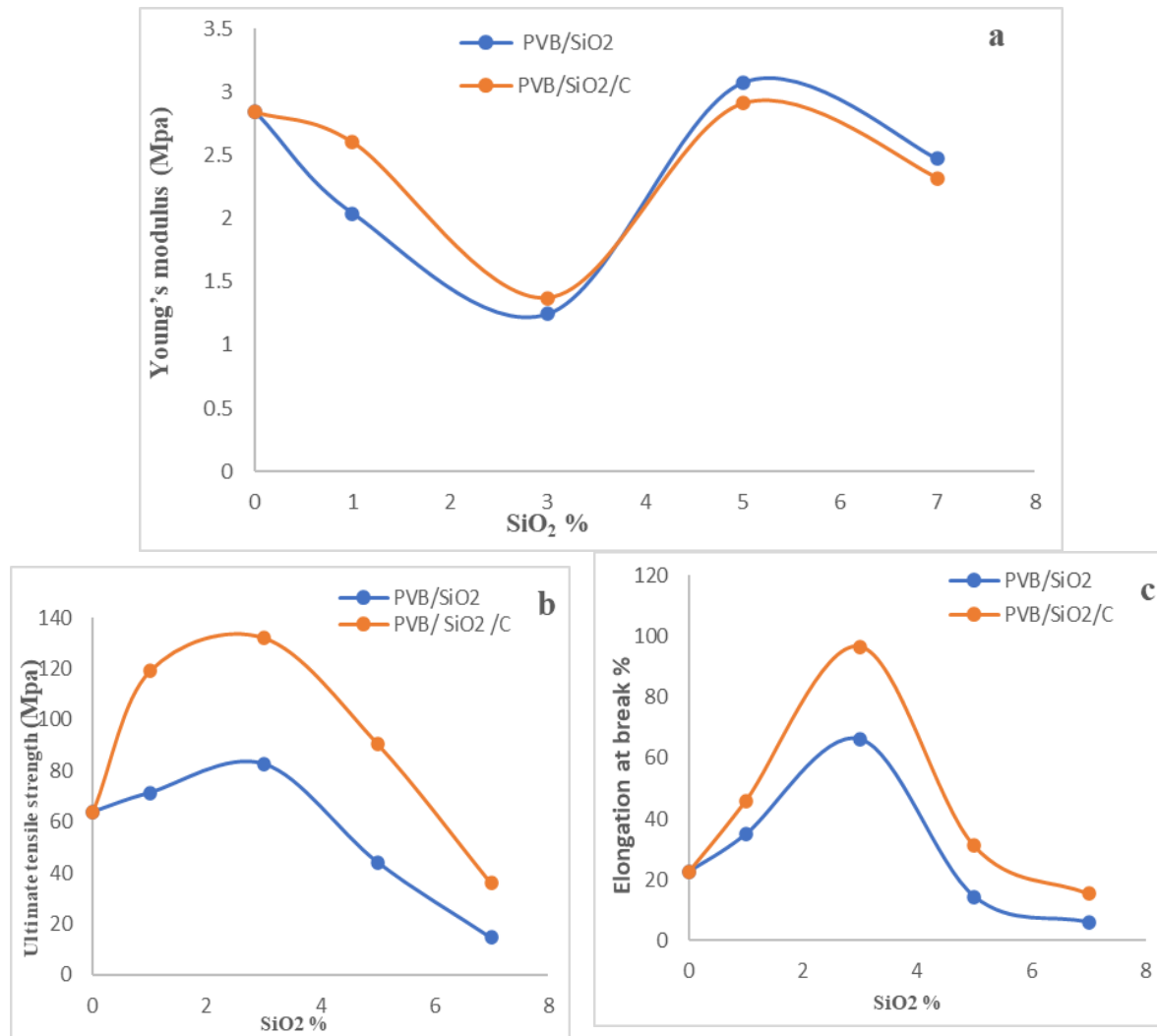


Figure 7. Tensile studies of the PVB / SiO₂ with and without coupling agent (a) Young's modulus (YM), (b) Ultimate tensile strength (UTS) and (c) Elongation at break %.

The films of the prepared samples showed that adding SiO₂ nanoreinforcement steadily enhanced the hardness. Indentation hardness was improved by 1.125% in the PVB film containing 3 wt percent SiO₂ nanoparticles compared to the clean PVB film, according to the results. While In the presence of coupling agent (C), the hardness was increased for neat PVB, yielding 1.25 % at 3% SiO₂. It was observed that the coupling agent had a significant effect at 5 % and 7% than without coupling, as presented in Figure 8.

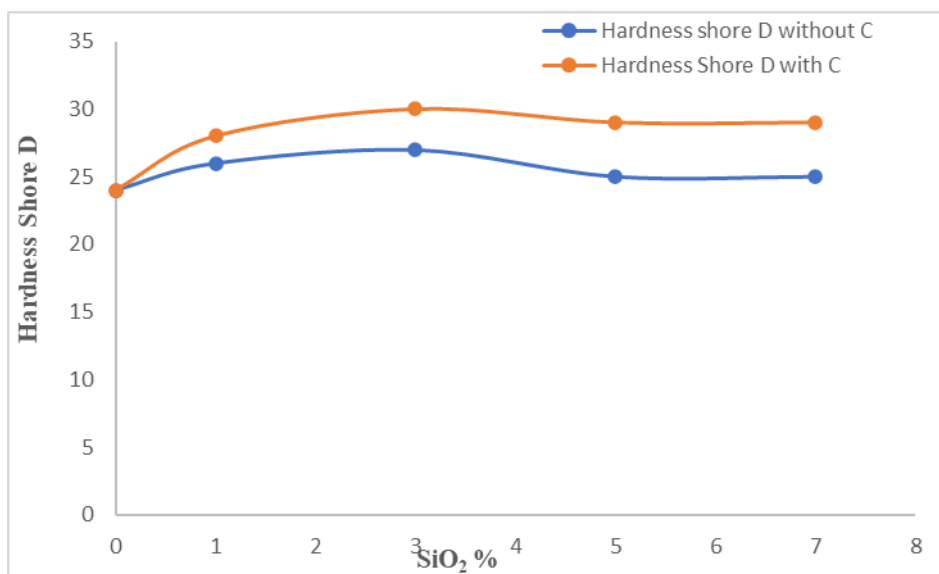


Figure 8. Hardness Shore D for PVB with different ratios of SiO₂ with and without coupling agent.

Figure 9 illustrates the ranges of Young's modulus (YM), ultimate tensile strength (UTS), and elongation at break percent (EB%) examined for all PVB / SiO₂ non-woven (NW) in the tensile properties with and without coupling. The tensile strength was attained for the composite at 1% and 3% SiO₂, with 6.65 and 7.12 MPa, respectively, whereas the lowest values were recorded for untreated NW. The highest tensile strength of NW-PVB / SiO₂ in the presence of a coupling agent was 7.75 MPa at 3%, which is adequate for non-woven applications. Modification with SiO₂ particles improved the tensile strength, which provides evidence of the improved dispersion and stabilization of SiO₂ particles within the fiber entanglement, which is an essential requirement for improving mechanical behaviors [15]. In other words, the coupling agent increased the particle stabilization of PVB / SiO₂ composites and fibers due to increased interfacial contact between them [10].

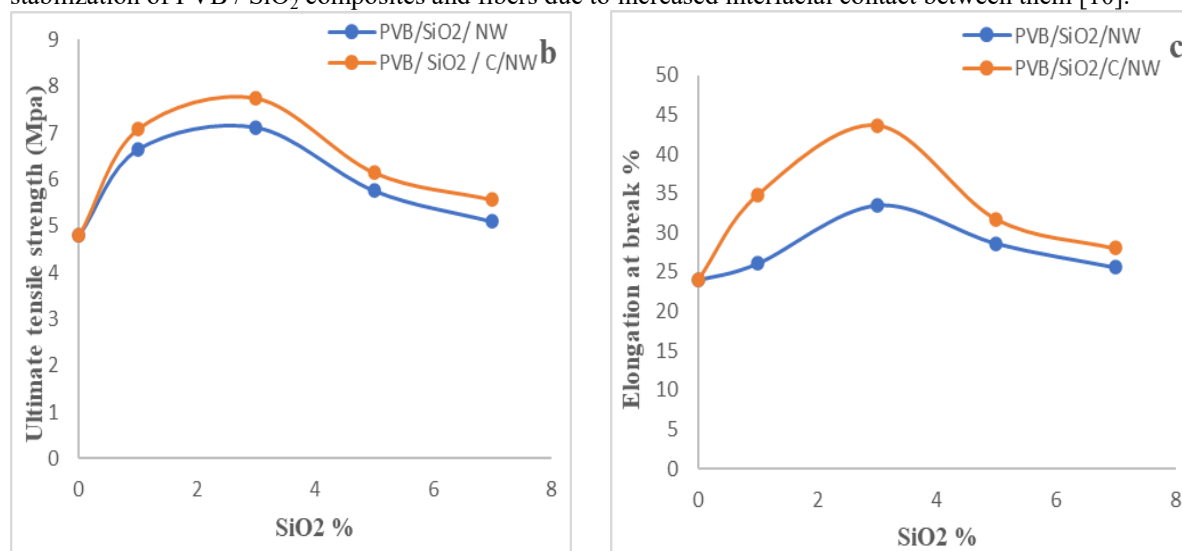


Figure 9. Tensile studies of the PVB / SiO₂ non-woven (NW) with and without coupling agent (a) Young's modulus (YM), (b) Ultimate tensile strength (UTS) and (c) Elongation at break %.

Weathering resistance simulation test

Weathering resistance simulation test

UV-simulated weathering was used to examine the effects of outdoor durability aging on non-woven samples. Differences in appearance before and after 120-hour UV exposure. After the accelerated weathering tests, all the exposed prepared samples exhibited significant weight loss relative to their initial weights but no volume change. There was no apparent color change in any of the samples and no visible traces of UV exposure. As shown in Figure 10, none of the examined samples had any discernible warpage. Figure 11 depicts the weight loss (%) of treated non-woven samples before and after weathering resistance and demonstrates how it increases

as SiO₂% increases and then decreases as SiO₂% increases. Due to UV light-induced chain scission and oxidation of the PVB polymer matrix, weight loss increases during prolonged exposure to natural weathering [14], much as when coupling agents are present because of their oxidation and degradation. The non-woven polyester's PVB/SiO₂ advantage has been particularly well studied for its long-term weather and crack resilience.

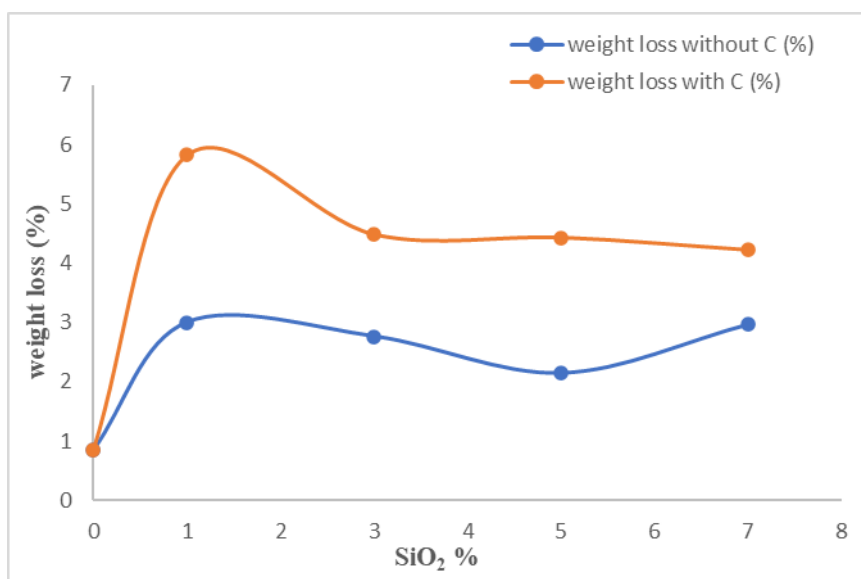


Figure 10. Weight loss (%) of treated non-woven samples before and after weathering resistance.

IV. Conclusion

Non-woven textiles, which are fibrous structures with fibers ordered in a random pattern, have important properties and characteristics that are appropriate for use in many applications.

Polyvinyl butyral /SiO₂ nanocomposites were prepared using the casting method in the presence or absence of 3-glycidioxypropyltrimethoxysilane as a coupling agent to improve the interaction of silicon oxide particles with the polymeric chains to yield films with the expected mechanical properties than the neat polymer. The prepared films were characterized using FTIR and SEM, which confirmed the structure and homogeneity of the surface. The TGA and mechanical properties of these nanocomposite films were studied, and a significant improvement in the thermal stability of the samples was predicted. The best ratio of SiO₂, which improved the thermal stability of PVB was 3% in the presence of a coupling agent with a weight residue 53.35%. The base matrix stiffness of nanocomposite films exhibited improved tensile properties with increasing silica content at 1% and 3% of SiO₂, and the hardness value increased as SiO₂ nano reinforcement was gradually added. While in the presence of coupling agent (C), the hardness was increased for neat PVB, yielding 1.25 % at 3% SiO₂.

The non-woven polyester was successfully treated with the prepared polyvinyl butyral/silica nanocomposites and characterized using FTIR. Thermogravimetric analysis (TGA) was performed for the treated non-woven polyester. The most thermally stable sample was PVB/1% SiO₂, which remained the residue still 50% until 632 °C, but in the presence of coupling agent, the thermal stability of the sample of PVB/1% SiO₂ /C was more thermally stable with a residual 56% compared to non-woven matrix 31.41% at 632 °C, and non-woven without coupling agent. The highest tensile strength of NW- PVB / SiO₂ in the presence of coupling agent was 7.75 MPa at 3%, which is adequate for non-woven applications. It was discovered that no volume change, but weight loss, occurred for all the exposed prepared samples compared to their initial weights following the accelerated weathering testing of the non-woven fabric via visual change before and after exposure to UV weather for 120 h.

Author contributions

A. A. Mousa Writing—review and editing, Methodology, Supervision, Data curation, practical work, and writing —original draft.

M. G. Mohamed Writing—review and editing, Methodology, Supervision, Data curation, and writing —original draft.

A. H. Moustafa Writing— Review and Editing, Methodology, Supervision, Data curation, and Writing—original draft.

H. A. El-Sayed Writing— Review and Editing, Methodology, Supervision, Data curation, and Writing—original draft.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have influenced the work reported in this study.

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