Exp. Theo. NANOTECHNOLOGY 10 (2026) 197-206 https://doi.org/10.56053/10.S.197

Preparation of nanocomposites fibers via PVA/natural nanomaterial

Fatima J Musaa, Hanaa J. Kadhim Alesa[*]

Department of Polymer Engineering, College of Materials Engineering, University of Babylon, Babylon, Iraq
[*]) Email: mat.hanua.jawad@uobabylon.edu.iq

Received 17/11/2025, *Received in revised form* 15/12/2025, *Accepted* 28/12/2025, *Published* 15/2/2026

The aim of this study is to manufacture a nanocomposite of mechanically ground nano red chicken eggshells and PVA polymer with 10% conc. and using five ratios of eggshell nanoparticles like (0.004, 0.005, 0.006, and 0.007) gram. There are many testes like tensile strength, scanning electron microscopy (SEM), Fourier Transform Infrared (FTIR) Spectroscopy and thermal differential scanning (DSC) tests are performed on the resulted nanocomposite threads. Use Electro spinning technique Nano fibers reinforced with nanoparticles are prepared. Results of SEM proved that the nanofibers diameter decreases with increasing the nanoparticles ratio, FTIR results showed that there are a physical reaction between the nanofibers and nanoparticles without any chemical reaction. DSC results proved that increasing the glass transition temperature with increasing the nanoparticles ratio. Tensile strength of nanocomposites fibers increases with increasing the nanoparticles ratio as well as the plastic deformation of nanofibers appeared.

Keywords: PVA nanofibers; Eggshell nanoparticles; Mechanical.

1. INTRODUCTION

The most significant technological development in modern areas is nanotechnology, which uses tiny materials with special qualities because of their large surface area [1]. Many nanomaterials have a variety of shapes, such as nanofibers [2] and spherical materials [3]. The emergence of bio-nanocomposites, a class of materials characterized by intricately designed structures, ushers in a new age in the search for revolutionary approaches to healthcare and beyond [4]. Biopolymers are combined with nanoscale reinforcements, usually in the form of nanoparticles [5], nanotubes [6], or nanofibers [7], to create bio-nanocomposites with unique advanced properties. These composites provide materials with improved mechanical, thermal and barrier properties by combining the unique properties of nanoscale additives and the biopolymer matrix [8,9]. Eggs are consumed around the world because they provide all of the essential amino acids, vitamins, and minerals. Eggshell trash is mostly composed of Ca+, magnesium carbonate (MgCO3), Calcium carbonate ($CaCO_3$), tetracalcium aluminoferrite (C_4AF) and protein. As well as, constitutes a significant portion of the solid waste generated by food preparation and production companies. Although non-harmful, this natural solid waste is typically disposed of in landfills without any processing because it is previously useless [10]. Nanoparticles represent a new class of engineering materials. These are small particulate solids with a less than 100 nm in one dimension at less. Nanoparticles show improved size-dependent features when compared to larger counterparts of the same substance. The nanoparticles' size and form determine their distinct properties. Thus, control over size and form allows for the construction of nanoparticles with specific qualities sought in their applications.[11] Nano particles have high surface area to volume ratio with quantum effect , it have high mechanical properties due to their crystalline structure ,high bioactivity , less porosity and less grain boundaries [12]. Mechanical grinding is a top-down method for synthesis of nanomaterials; it is simple and inexpensive, as well as, it may yield large amounts of nanoparticles [13]. Electrospinning is a flexible technology for generating polymer fibers with fiber diameters ranging from a few micrometers to nanometers employing polymer solution. The procedure is easy and affordable when compared to other conventional methods [14,15]. Electrospinning allows for the fabrication of more complicated fiber morphologies such as smooth fibers, fibers with differed diameters, coiled and straight nano-fibers. Micro and nano fibers have a broader range of uses such as biosensing, water treatment, catalysis, energy harvesting, tissue engineering, and threading [16-18].

The current study aims to fabricate the polyvinyl alcohol nanofibers reinforced with eggshell nanoparticles, which are previously prepared via mechanical grinding approach, utilizing the electrospinning method of nanofibers for use in bone sutures.

2. EXPERIMENTAL

2.1 Materials and methods

Polyvinyl alcohol PVA (Molecular weight: approx. 115,000, CAS Registration No: 9002-89-5 ADR/PG), Red eggshell are used in this study. Distilled water is utilized throughout the whole research work for solution/sample preparation. Different equipment such as dumbbell sample making machine (model: HS-5010, Jinan Hensgrand Instrument Co), electronic balance (type: ABS120-4, AWB1000176), Ball Miller (No:030580), 8411 electric sieve shaker (type : 8411), electric blast dry box (model:wg43,Tianjin Taisite Instrument Co), and ultrasonic cell crusher (sjia-1200w,MTI co) are utilized for various measurement.

2.2 Preparation of PVA solution

The solution of pva is prepared by dissolving PVA granules with distilled water in four different proportions (8%, 10%, 12%, 14%, and 16%) wt. Then, fibers are formed from this solution using an electro spinning device to conduct an initial tensile test using (microcomputer controlled electronic universal testing machine –model wdw, 5e-class-1) to know which concentrations have the greatest tensile strength to adopt in forming the composite material.

2.3 Preparation of nano composite solution

After conducting the necessary tests on the PVA solutions and choosing 10% as the best percentage, the composite material is manufactured using the following steps:

1. Four weights of nano eggshells (0.004, 0.005, 0.006, and 0.007) grams are selected.

2. These weights are dispersed with 100 ml of distilled water by ultrasonic device for 30 minutes for each weight.
3. After completing the dispersion process, these nano-solutions are transferred to the electric mixer and the previously prepared 10% PVA solution is gradually added to them, increasing the mixing speed to 550 rpm. Continue mixing for one hour. We noticed that the color of the substance changed from transparent to milky white.
4. The resulting material is placed in the oven to remove air bubbles from it. This continued for two hours until the material became completely free of bubbles.
5. The material is transferred to the electro-spinning machine to manufacture the composite material fibers.

These steps are repeated for four ratios of nanomaterial mixed with PVA solution:
10%PVA=PVAE1
10%PVA+0.004Eggshell=PVAE2
10%PVA+0.005Eggshell=PVAE3
10%PVA+0.006Eggshell=PVAE4
10%PVA+0.007Eggshell=PVAE5

3. RESULTS AND DISCUSSION

Figure 1 and table 1 show the FTIR analysis of PVA nanofibers reinforced with eggshell nanoparticles.

Table 1 Peaks and bonds of PVA nanocomposite fibers.

Peaks cm^{-1}	Bonds
1425 **1370** **1125**	$CaCO_3$
3350 **3200**	-OH group in the PVA nanofibers
2915 **2860**	CH Stretching In the PVA nanofibers

As notice, the increasing of eggshell nanoparticles ratios leads to increasing the peaks intensity at (1125 – 1425 cm^{-1}) due to increasing the $CaCO_3$ contents. In addition, FTIR data indicated the interaction of eggshells and polymer (PVA) nanocomposites fibers due to functional groups, such as -OH and C=O groups, this is agreement with Taşaltın, et al 2022 [19].

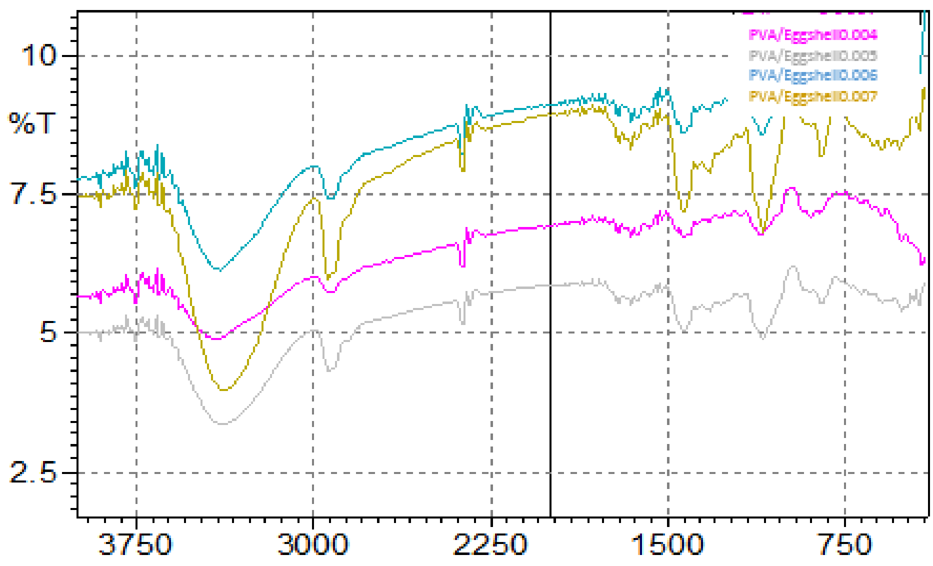


Figure 1 FTIR analysis of PVA/eggshell nanocomposite fibers.

Figure 2 show SEM images of eggshell that used in this study. As proved in the image, the nanoparticles utilized, which are created using a nanofilter with pores smaller than 100 nanometers, ranged in size from 30 to 40 nanometers. The particle size of nanoparticles created through the mechanical grinding process is affected by several factors, including grinding time, grinding substrate mass, ball size, and vibration power. Increasing the grinding time reduce particle size, and utilizing tiny sieve pores reduces particle size to the appropriate size for the resulting nanoparticles. It is also possible to enhance the effective particle size using ultrasonic dispersion, the use of dispersion technology leads to the separation and dispersion of nanoparticles, thus preventing the clumping that occurs in the material, making the determination of the particle diameter more accurate [20].

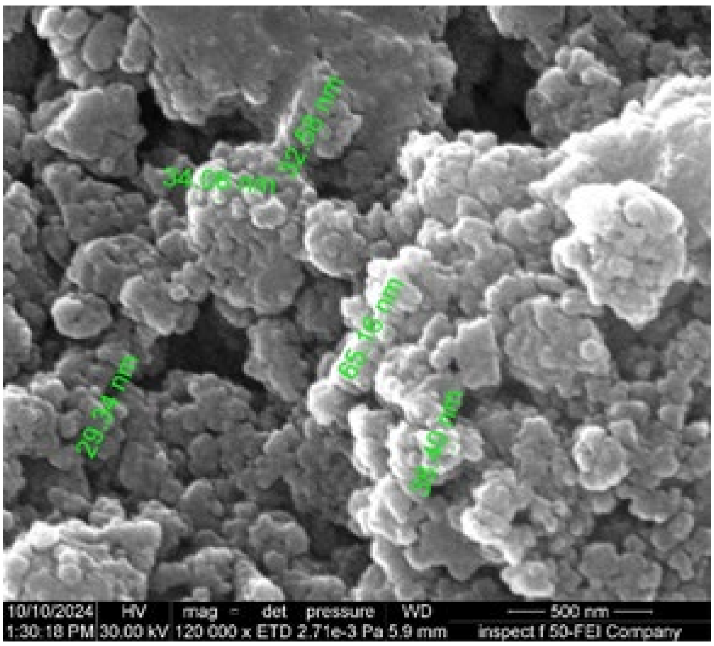
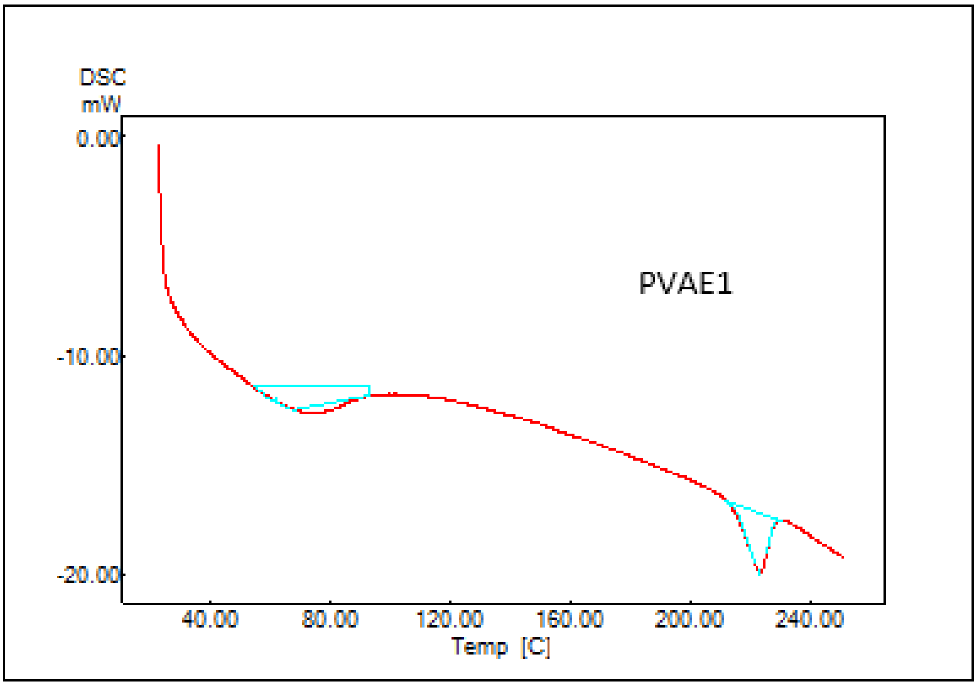


Figure 2 SEM images of eggshell nano particles after 20 hr time of grinding and sieve with 100 nm pores filter (about 30-40 nm average diameter).

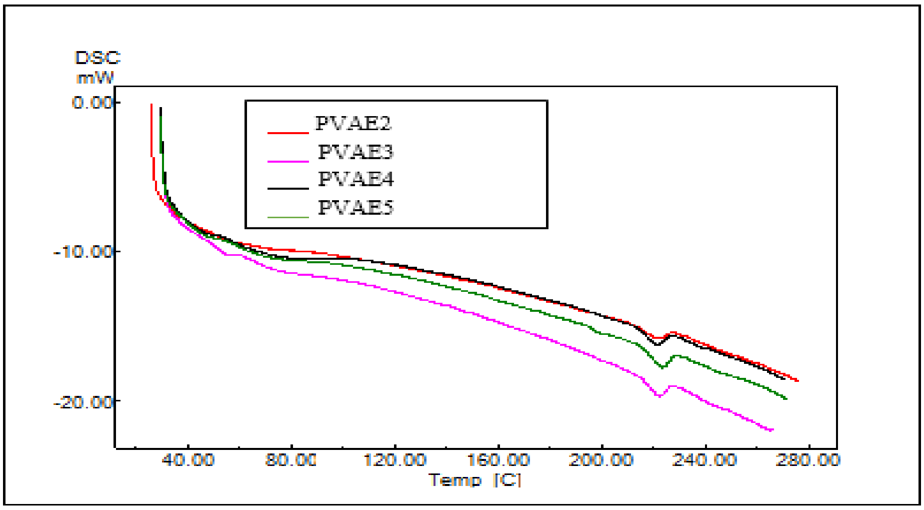
Figure 3a-b and Table 2 shows the thermal analysis of PVA nanofibers and their nanocomposites with eggshell nanoparticles. Table 2 and Figure 3 show that the glass transition temperature increases as the weight fraction of eggshell nanoparticles increases. The temperature improves from (62.02) degrees Celsius to (64.05) degrees Celsius as the weight percentage increase from (0.005) to (0.007). This is because the presence of nanoparticles hinders the movement of polymer chains, and increasing the glass transition temperature value [21]. On the other hands , we observe that increasing the proportion of nano-eggshells improves the melting temperature of the nanofibers [22], improving the thermal stability of the nanofibers reinforced with nano-eggshell particles[23].

Table 2 Thermal analysis of nanofibers via DSC.

Sample	Tg 0c	Heat mW	Tm 0c	ΔHm J \ g Melt enthalpy
PVAE1	62.02	-0.43	222.91	**-14.30**
PVAE2	62.91	-0.57	222.96	**-3.50**
PVAE3	67.35	-1.25	222.98	**-4.69**
PVAE4	68.34	-0.76	223.75	**-4.30**
PVAE5	64.05	-1.03	223.55	**-4.47**



(a)



(b)

Figure 3 (a) DSC analysis of PVAE1 (pure PVA nanofibers). **(b**) Differential scanning calorimetric for four sample of PVA/eggshell nanocomposites fibers with different concentration of eggshell nanoparticles.

Figure 4 and Table 3 show the main parameters of the tensile test of the PVA/Eggshell fibers. The polymeric material experiences elastic deformation in this area because of the polymer chains' elongation and stretching without any bond breaking. As cracks start to appear inside the polymer, this curve eventually stops being linear [24]. As the applied stress increases, these cracks widen and unite to produce larger cracks that keep getting bigger until the sample fractures.

Note: Note: It is clear that PVAE3 composite fibers achieved the highest tensile strength among the other concentrations. The addition of small amounts of eggshell nanoparticles increased the tensile strength by acting as fracture barriers and by subjecting the nanofibers to plastic deformation. This is explained by the improvement of mechanical properties with a somewhat increased nanoparticle content. The addition of the nanoparticles in small amounts and their homogeneous distribution within the nanofibers through ultrasonic dispersion leads to a homogeneous fusion of the fibers with the nanoparticles during the pumping process, resulting in a homogeneous fiber diameter in which all polymer chains orient toward the fiber axis, resulting in increased fiber tensile strength and other mechanical properties. In addition to increasing the amount of energy consumed for fracture and thus increasing the strength of the composite nanofibers, because the nanoparticles act as crack growth inhibitors, they increase the energy required for failure, which in turn improves the fiber's mechanical properties. Furthermore, fibers without beads have better properties because the beads act as defects that weaken the fiber's mechanical properties. Adding high percentages of nanoparticles leads to the formation of clumps of the nanomaterial, its inhomogeneous distribution, and irregularity of the fibers, which weakens its mechanical properties and leads to failure at low loads [24,25].

Table 3 Mechanical properties of PVA nanofibers and their nanocomposites with eggshell nanoparticles

Sample	Tensile Strength	Yield point	Young's modulus
PVAE2	11.30	2.260	0.8
PVAE3	66.0	1.320	0.25
PVAE4	3.454	0.0840	0.2
PVAE5	7.06	1.411	0.03

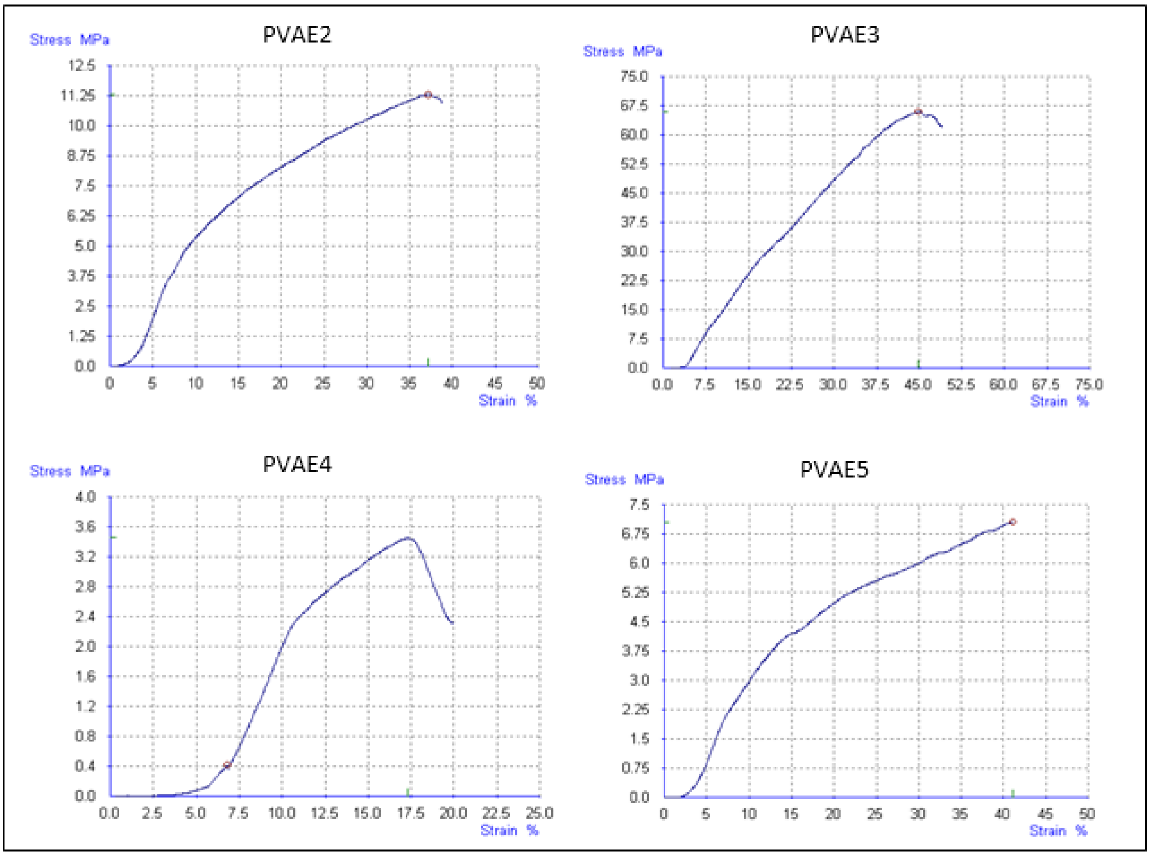


Figure 4 Tensile test of PVA/eggshell fibers.

Figure 5 A-E shows the SEM images of PVA nanofibers and their nanocomposites with eggshell. The image shows that the fibers produced by the electrospinning technique are free of beads and have a homogenous morphology, which indicates the stability of the pumping mechanism. Variations in the weight ratio of the added nanoparticles cause variances in the final nanoparticle sizes. The additional particles improve the electrical conductivity of the solution while decreasing the diameter of the resultant fibers. This is because the additional nanoparticles increase the number of free ions in the solution, which increases its electrical conductivity. Adding more nanoparticles causes particle agglomeration, a decrease in solution conductivity, and an increase in pumping resistance, resulting in an increase in fiber diameter. This is due to the decrease in the number of free ions in the solution, which leads to an increase in the diameter of the fiber and the obstruction of the pumping process, making it unstable and irregular [19].

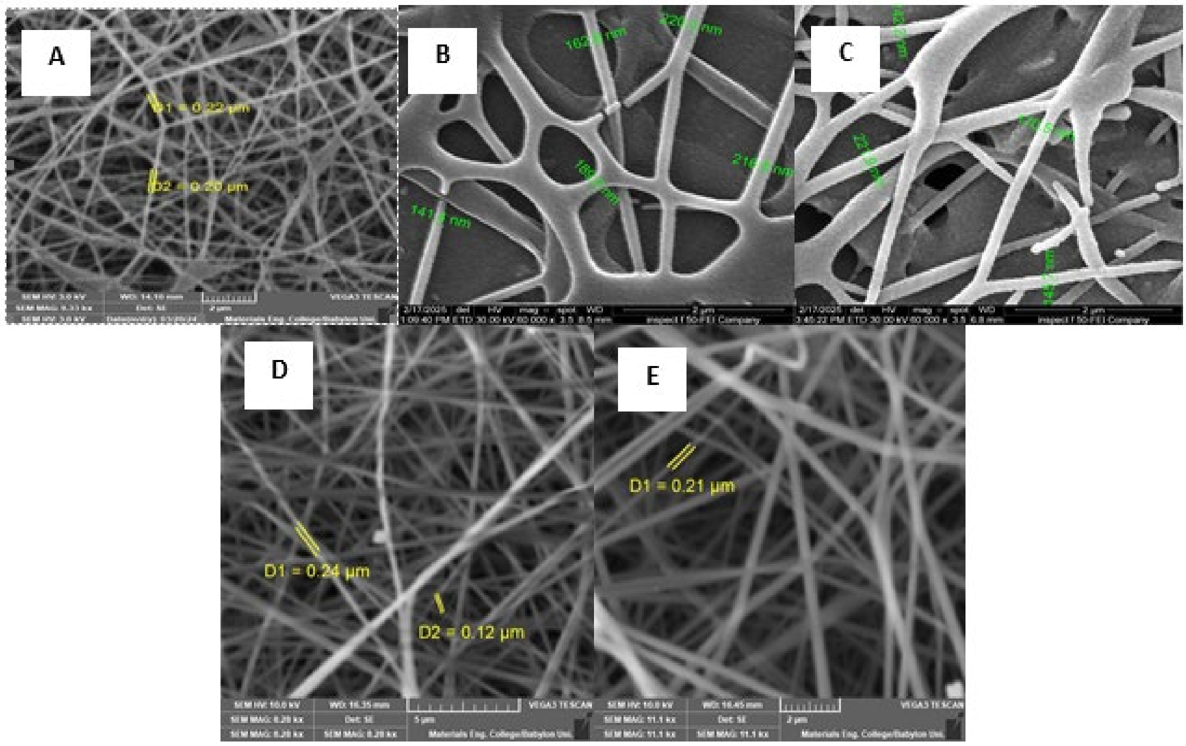


Figure 5 Scanning electron microscope PVA/eggshell fiber: A) PVAE1, B) PVAE2, C) PVAE3, D) PVAE4, E) PVAE5.

4. CONCLUSIONS

From this work, ready-to-use polymer nano fibers were obtained for bone sutures, and many tests were conducted to determine their properties. From the FTIR results, it is clear that there is an interaction between the eggshell and the polymer nano fibers due to the apparent functional groups such as OH and c=o. From the DSC results, it is clear that Tg and Tm increases with the increase in the weight of the added nanomaterial. The tensile test shows that the polymeric material undergoes elastic deformation as a result of the elongation and stretching of the polymer chains without any bond breakage. We note that PVAE3 polymer fibers achieved the highest tensile strength among other fibers. The addition of small amounts of nanoparticles increases the tensile strength because they act as fracture barriers and subject the nanofibers to plastic deformation. SEM images show that the electrospun fibers are free of beads and have a homogeneous morphology.

Abbreviation

PVA: poly vinyl alcohol, FTIR: Fourier transform infrared spectroscopy, SEM: scanning electron microscopy.

Conflict of interest

The authors declare that there is no conflict of interest.

Consent for publications

All authors confirm consent to publication.

Availability of data and material

The authors declare that they embedded all data in the manuscript.

Authors' contributions

All of the authors—F. J. and H. J.—participated in the sample and material preparation. They participated in testing and discussing the samples as well. Additionally, they assisted in arranging the research to ensure that it ready for publication.

Funding

There is no supporting organization or company; the authors covered all research expenses.

Acknowledgement

The researchers would like to express thanks to the staff at the University of Babylon's College of Materials Engineering laboratories for performing the testing for this study.

References

[1] R.M. Alradha, J. Hanaa, A.A. Al-Zubiedy, Egypt. J. Chem. 66 (2023) 141 https://doi.org/10.21608/ejchem.2022.133004.5876
[2] F.J. Musaa, H.J. Kadhim, Iraqi J. Appl. Phys. 21 (2025) 380 https://doi.org/10.2025/e9kndy89
[3] N. Ben Azaza et al., Opt. Mater. 96 (2019) 109328 https://doi.org/10.1016/j.optmat.2019.109328
[4] R. Mohamed, N. Adnan, H. Jawad, B. Mohammed, R. Mahdi, Baghdad Sci. J. 22 (2025) 955 https://doi.org/10.1016/j.rico.2025.100542
[5] M. Rasheed, SuhaShihab, O. Alabdali, H. H. Hassan, J. Phys. Conf. Ser. 1879 (2021) 032113 https://doi.org/10.1088/1742-6596/1879/3/032113
[6] A.A. Flayeh, H.J. Kadhim, Fullerenes Nanotub. Carbon Nanostruct. 30 (2022) 1090 https://doi.org/10.28978/nesciences.1479785
[7] H.J. Kadhim, R.M. Salih, IOP Conf. Ser. Mater. Sci. Eng. 757 (2020) 012001 https://doi.org/10.1088/1757-899X/757/1/012001
[8] I. Rezaei, A. Sadeghi, Arab. J. Sci. Eng. 46 (2021) 12479 https://doi.org/10.1007/s13369-021-06016-5
[9] A.J. Hussein, M.N. Al-Darraji, M. Rasheed, M.A. Sarhan, IOP Conf. Ser.: Earth Environ. Sci. 1262 (2023) 022005 https://doi.org/10.1088/1755-1315/1262/2/022005
[10] E.M. Shcherban' et al., Appl. Sci. 12 (2022) 6606 https://doi.org/10.3390/app122211744

Exp. Theo. NANOTECHNOLOGY 10 (2026) 197-206
[11] D. MEKAM, D. MESRI , H. Rozale. Experimental and Theoretical NANOTECHNOLOGY 3 (2019) 281 https://doi.org/10.56053/3.3.281
[12] T. Duńvki, V. Saryviak, Experimental and Theoretical NANOTECHNOLOGY 3 (2019) 269 https://doi.org/10.56053/3.3.269
[13] D.B. Devedov, R.J. Asherov, G.H. Glenko, N.O. Ferstaloya, Experimental and Theoretical NANOTECHNOLOGY 3 (2019) 253 https://doi.org/10.56053/3.3.253
[14] T. Saini, J. Meena, V. Verma, S. Saini, R. Malik, Polym. Technol. Mater. 23 (2025) 1 https://doi.org/10.1080/25740881.2024.2438046
[15] B.A. Abdulkadir, H.D. Setiabudi, Polym. Technol. Mater. 33 (2025) 69 https://doi.org/10.1080/00986445.2024.2386304
[16] H. Dong, S. Wang, K. Yang, Y. Chen, J. Coat. Technol. Res. 21 (2024) 1173 https://doi.org/10.1007/s11998-024-00911-w
[17] D.A. Kadham, A.J. Braihi, H.J. Kadhim, Fullerenes Nanotub. Carbon Nanostruct. 31 (2023) 388 https://doi.org/10.1080/1536383X.2023.2168266
[18] R.M. Salih, H.J. Kadhim, Kuwait J. Sci. 50 (2023) 262 https://doi.org/10.1016/j.kjs.2023.01.008
[19] N. Taşaltın et al., J. Mater. Sci. Mater. Electron. 33 (2022) 6496 https://doi.org/10.1007/s10854-022-07823-8
[20] M. Zhu et al., Materials 15 (2022) 6033 https://doi.org/10.3390/ma15176033
[21] A. Sowińska-Baranowska, M. Maciejewska, Materials 16 (2023) 2988 https://doi.org/10.3390/ma16082988
[22] R. Barbaz-Isfahani et al., Ind. Crops Prod. 205 (2023) 117498 https://doi.org/10.1016/j.indcrop.2023.117498
[23] N. Rathinavel et al., Adv. Civ. Eng. 33 (2023) 6655921 https://doi.org/10.1155/2023/6655921
[24] P. Nagaraaj, Food Packag. Shelf Life 41 (2024) 101239 https://doi.org/10.1016/j.fpsl.2024.101239
[25] M. Deng, Z. Dong, C. Zhang, Constr. Build. Mater. 235 (2020) 117801 https://doi.org/10.1016/j.conbuildmat.2019.117801