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ORIGINAL

Synthesis of PVA Nanofibers to Enhance the Mechanical and Thermal Properties of PMMA Matrix for Denture Base Applications

Síntesis de nanofibras de PVA para mejorar las propiedades mecánicas y térmicas de la matriz de PMMA para aplicaciones en bases de prótesis dentales

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ABSTRACT

This study attempts to prepare PMMA nano-composite samples armed with polyvinyl alcohol electrospun nanofibers. One layer, two layers, and three layers of nanofiber reinforcement were all utilized. As well as, one drop (0,01 g) and three drops of the cardamom oil were used. Using a differential calorimeter, thermal experiments were carried out, including those for the Tg and the thermal dissociation point. Mechanical tests were conducted on the samples, including compression testing to determine the elongation characteristics, compressive strength, Young's modulus, yield strength, and toughness. All samples were also subjected to an impact strength test. Results showed that the Tg point increased due to the presence of nanofibers from 136 to 195 Celsius, and that 277 Celsius was the thermal breakdown point. According to the mechanical property data, the impact strength increased as the percentage of nanofibers rose and increased further when one drop of cardamom oil was mixed with a very tiny amount (0,01 g) of material. Additionally, it was noted that the samples did not break easily since nanofibers and a drop of cardamom oil were present. Additionally, it was demonstrated that the sample's strength increased to 195 kJ/m^2, as well as 0,02 weight % of fibers, the maximum strength value.

Key words: Nanocomposites; Electrospinning Method; Morphological Properties; Mechanical Performance; And Thermal Characterizations.

RESUMEN

En este estudio se intenta preparar muestras de nanocompuestos de PMMA armadas con nanofibras electrospun de alcohol polivinílico. Se utilizaron una capa, dos capas y tres capas de refuerzo de nanofibras. Asimismo, se utilizaron una gota (0,01 g) y tres gotas del aceite de cardamomo. Utilizando un calorímetro diferencial, se llevaron a cabo experimentos térmicos, incluidos los de la Tg y el punto de disociación térmica. Se realizaron pruebas mecánicas con las muestras, incluidas pruebas de compresión para determinar las características de alargamiento, la resistencia a la compresión, el módulo de Young, el límite elástico y la tenacidad. Todas las muestras se sometieron también a una prueba de resistencia al impacto. Los resultados mostraron que el punto Tg aumentaba debido a la presencia de nanofibras de 136 a 195 Celsius, y que 277 Celsius era el punto de ruptura térmica. Según los datos de las propiedades mecánicas, la resistencia al impacto aumentaba a medida que aumentaba el porcentaje de nanofibras y aumentaba aún más cuando se mezclaba una gota de aceite de cardamomo con una cantidad muy pequeña (0,01 g) de material. Además, se observó que las muestras no se rompían fácilmente al estar presentes las nanofibras y una gota de aceite de cardamomo.

Además, se demostró que la resistencia de la muestra aumentaba hasta 195 kJ/m2, así como el 0,02 % en peso de fibras, el valor máximo de resistencia.

Palabras clave: Nanocompuestos; Método de Electrospinning; Propiedades Morfológicas; Comportamiento Mecánico y Caracterizaciones Térmicas.

INTRODUCTION

Nanotechnology is a science relating to the structure of materials at the atomic and molecular level and to morphology at the nanoscale. One-dimensional nanostructures, such as nanowires, nanofibers, and nanotubes have been produced recently for their unique features and potential uses in electronics and nanocomposites. The advanced method for manufacturing ultrafine fibers, electrospinning, was invented by Formhals in 1934. In recent years, Reneker and co-workers have taken an interest in this technique which described electrospinning of several polymer solutions.[1] Since 1937, acrylic resins have been the most widely utilized material in the production of denture bases.[2] It is superior to other materials since it is simple to work with and repair, non-toxic, and lightweight, but it has a number of disadvantages, one of which is that it has subpar mechanical qualities.[3] A denture fracture results from two different types of forces, namely impact and flexural fatigue. The most distant intraoral denture fracture through function arises mostly due to resin stress. Outside the mouth, high impact forces due to dropping prostheses cause fractures.[4] To enhance the mechanical properties of acrylic resin, many efforts have been made. One such effort is the incorporation of some types of reinforcement into the denture base material.[5] In a report published in 2013, Hanan evaluated the effects of adding Siwak powder to PMMA in three different weight concentrations, with an average particle size of (75 μm) in each. For both the stander group A and the experimental groups, the tensile strength, elongation, transverse strength, impact strength, compressive strength, and surface roughness of heat-polymerized acrylic resin specimens were assessed. Various weight ratios of Siwak powder first ratio (3%), second ratio (5%) and third ratio (7%) were combined with acrylic resin to create these groupings. For measuring the tensile strength and elongation of acrylic resin, stainless steel samples with the following dimensions were created: 65 mm, 12,5 mm, 2,5 0,03 mm. She came to the conclusion from her research that the Siwak powder (5%) had little effect on the tensile strength and that the compressive strength was (P = 0,05). Comparing the experimental group to the control group, the addition of Siwak powder at a ratio of 3 percent had little impact on the experimental group's impact strength, but adding Siwak powder at a ratio of 7 percent revealed a significant decline in tensile strength, impact strength, and compressive strength.[1]

Fillers with nanoscale dimensions have a significant impact on the mechanical properties of denture base materials. Reem et al. (2013) presented a paper on increasing the thermal, mechanical, and corrosion resistance properties of Poly Methyl Methacrylate samples supplemented with licorice and pomegranate peel particles. Thermal analysis of the samples (PMMA+5% licorice particles and 5% pomegranate peel particles) reveals that their thermal conductivity and diffusivity are, respectively, (73 MPa, 70 MPa, 4,800 GPa, 4,300 GPa,.5600 W/m k,.5400 W/m k,.3800 mm2/sec,.2800 mm2/sec). The samples (PMMA + 1% licorice particles and 1% pomegranate peel particles) had the best mean values of elongation at break (3,600 %, 3,500 %, respectively). The outcomes demonstrated that the qualities of wear, tensile, and thermal properties were enhanced by the addition of licorice and pomegranate particles to poly methyl methacrylate resin.[2]

In 2021, Teba et al. published an paper about enhancing the mechanical properties of heat cured acrylic resin by adding the natural sisal fibers powder. 90 specimens were prepared for use in her investigation. The samples were separated into three main groups based on the presence of sisal fiber powder: the control group, which consisted of 30 heat-cured PMMA specimens without additives; the second and third experimental groups, each consisting of 60 heat-cured PMMA specimens with salinized sisal fiber powder, with weight percentages of 1 and 3 percent, respectively. The flexural strength of the specimens was measured using a three-point bending test, while the impact strength was assessed using Charpy's machine and the tensile test was carried out in accordance with ASTM (D-638). The (ANOVA) test was used for data analysis. The findings of this investigation show a highly significant difference between the control specimens and specimens reinforced with sisal fiber powder in terms of flexural strength and tensile strength. There was no discernible change in impact strength between the reinforced groups and the control group. The flexural and tensile strengths of acrylic resin are affected by the reinforcement with natural sisal fiber powder, although the impact strength is not significantly changed.[3] The aim of this work is preparation of nanocomposites material of PMMA (Poly Methyl Methacrylate) reinforced with PVA nanotextile fibers used in the manufacture of denture base.

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METHOD

Materials

Polyvinyl alcohol (PVA) was used as a reinforcing material along with heat-cured denture base acrylic resin (Meliodent, Kulzer, Germany).

Preparation of polyvinyl alcohol nanofibers

Poly vinyl alcohol and distilled water were combined using a stirrer, and the mixture underwent an electrospinning procedure to produce nanofiber textiles. The process settings were: 20 KV applied voltage, 15 cm between tip and collector, 25 °C temperature, 480 rpm rotation speed, and 1 ml/hr flow rate with a 0,38 mm needle diameter. Figure 1 depicts how the electrospinning machine is set up.

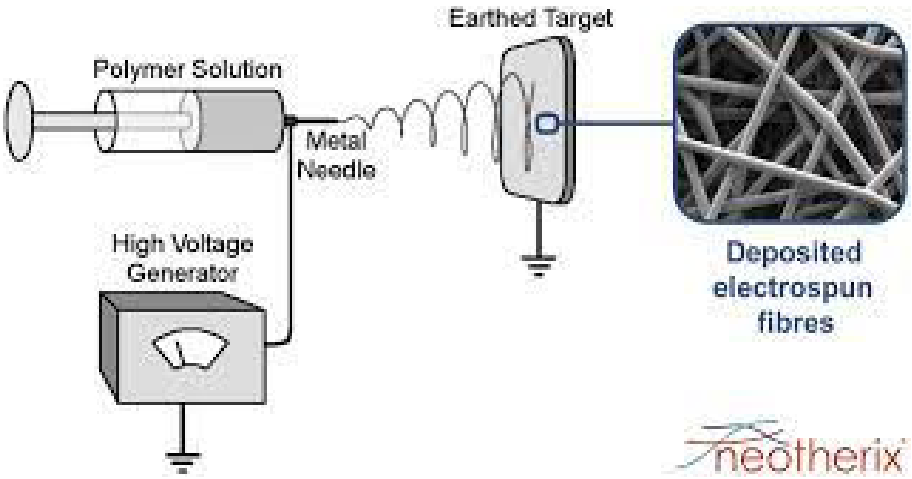

Figure 1. Electrospinning set up

Table 1. Contents of prepared nanocomposites samples

No. of sample	Contents	First group			2nd group	3rdgroup
		No. of PVA layers	Weight ratio of PVA NF in Impact samples wt% (I.S)	Weight ratio of PVA NF in compression, and hardness samples wt%	1st group + 0,01 g of Cardamom oil for (I .S)	1st group + 0,02 g of Cardamom oil for (I.S)
NC 0	PMMA	0	0	0	0	0
NC A	PMMA+ PVA NF	1	0,02	0,01	0,02 NF wt + 0,01g C.O	0,02 NF wt + 0,02g C.O
NC B	PMMA+ PVA NF	2	0,04	0,02	0,04 NF wt + 0,01g C.O	0,04 NF wt + 0,02 g C.O
NC C	PMMA+ PVA NF	3	0,06	0,03	0,06 NF wt + 0,01g C.O	0,06 NF wt + 0,02g C.O

Tests and Measurements

The viscosity of solution was tested via Brookfield DV-III Ultra Rheometer with spindle number 63, 125 rpm, and 25 o C. Surface tension test was carried out using Platinum Ring Method according to ISO: 1995 by JZYW-200B Automatic Interface Tensiometer. Using a conductivity meter (HANNA instruments - EC 214 conductivity Meter), the electrical conductivity of solution was tested. SEM (VE-8800, Keyence Co., Tokyo, Japan) was used to assess the electrospun PVA fibers' distribution, average fiber diameter, and fiber morphology on samples sputtered with palladium-plating. The solution parameters involve (concentration, electrical conductivity of solution, viscosity and surface tension) were tested and are as following (12% w/w, 7,5 µS/cm, 650cPs, and 40mN/m) respectively.

RESULTS AND DISCUSSION

Morphological Characterization

The surface morphology of prepared PVA nanofibers studied using scanning electron microscopy (FEI INSPECT F50), with accelerating voltage of (20 kV). Figure 2 shows the SEM image of prepared PVA nanofibers. Scanning electron microscopy image proved the formation of uniform and solid PVA nanofibers with random orientations. The SEM results indicated to obtain a fine and smooth PVA nanofiber with small diameters ranging from (350 to 500 nm) when using the preparation parameters (voltage = 20 KV, distance = 15 cm, temperature = 25 °C,

rotation speed = 480 rpm, and flow rate = 1 ml/hr), these small diameters of PVA nanofibers create a large surface area.[4,5]

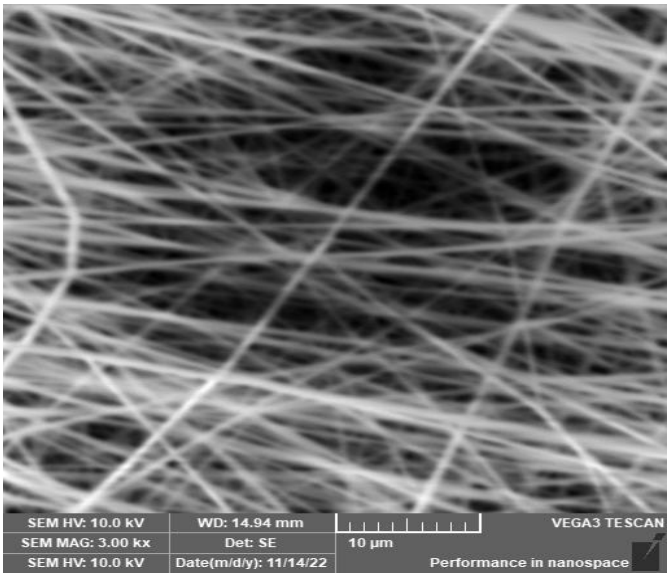

Figure 2. SEM image of prepared PVA nanofibers

From figure 2 observed that the stability of the pumping process allowed to manufacturing free beads PVA nanofibers with small diameters. The morphology of nanofibers can be affected with both the solution parameters and processing parameters, smooth nanofibers can be reached with the controlled selection of these parameters as suitable values of the high voltage of power supply, surface tension, viscosity, concentration and electrical conductivity of solution leads to stability of electrospinning process.[6]

Mechanical Characterizations

Impact Strength Exam

The impact exam used to study the toughness of the prepared materials, where the toughness is a factor of the material ability to absorb energy during the plastic deformations, whereas the ductile materials have high toughness due to the high amount of plastic deformation that can be tolerated.[7,8] As indicated in figure 3 (a), the impact specimens were prepared with dimensions according to the standard ASTM (80 mm x 10mm x 4mm) for un-notched specimens, while figure 3(b) shows the experimental impact samples that prepared in this work.

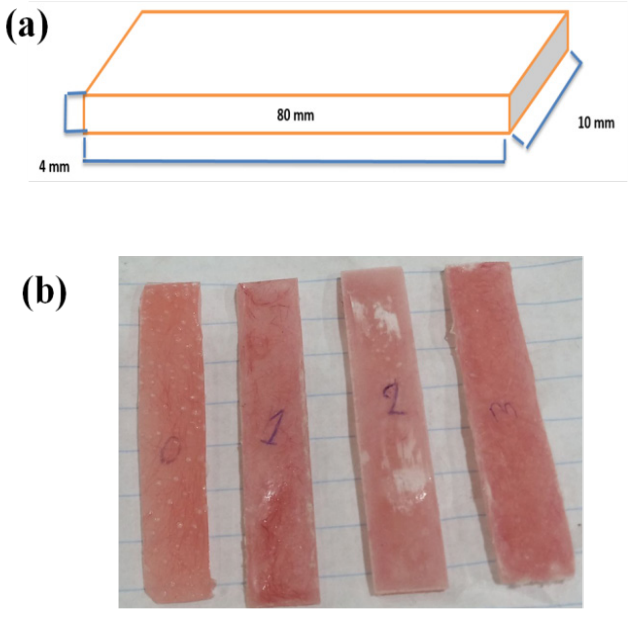

Figure 3. (a) the impact sample dimensions and (b) the prepared impact samples

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The impact strength results of prepared composite materials summarized in figure 4. The obtained results demonstrated that when the PVA nanofibers fraction (weight ratio) increased led to clearly enhance the impact strength values, where the samples with the high weight ratio of PVA nanofibers recorded the highest impact strength values (0,34, 0,4 and 0,5 KJ/cm^2), as presented in figure 4. The prepared samples were divided into three groups, as shown in table 1, observed that the impact strength values of the first group samples increased from (0,1 KJ/cm^2) for the un-enhanced sample to (0,34 KJ/cm^2) for the enhanced samples with three layers of PVA nanofibers without breakage, this attributed to increase the breaking energy after the enhancement, and may the effective stress transfer from PMMA matrix to the PVA nanofibers through the interface regions, in addition, the strong hydrogen bonding interactions between PMMA matrix and PVA nanofibers prevented the phase separation and thus enhance the impact strength of prepared samples . The large surface area of PVA nanofibers leads to reinforcement the mechanical properties.[9,10,11] The results indicated that the added PVA nanofibers led to increase the impact strength value about (240 %) comparing with the pure sample.

The impact strength results of the second group revealed that the impact strength values improved with the increasing the PVA nanofibers weight ratio without breaking the samples when a drops of cardamom oil (0,01 gm) is added. The fourth sample (with three layers of PVA nanofibers and cardamom oil) recorded high impact strength value (0,4 kJ/cm^2) due to the cross-linking effect of the cardamom oil which raises the density of linkages within the composite material and the bonding with PVA nanofibers and then increasing the required energy to reach the fracture. On the other hand, when a drops of cardamom oil (0,02 g) are added to the enhanced composites within the third group, observed that the samples break and the impact toughness increases as the weight of the PVA nanofibers increases. The fourth sample, which includes three layers of nanofibers and cardamom oil (0,02 gm) recorded the highest impact value (0,5 kJ/cm^2), this is due to the fact that adding more cardamom oil droplets serves as a cross-linking agent, considerably increasing the density of the bonds within the sample which leads to obtain brittle samples and more susceptible to breaking.[12,13,14]

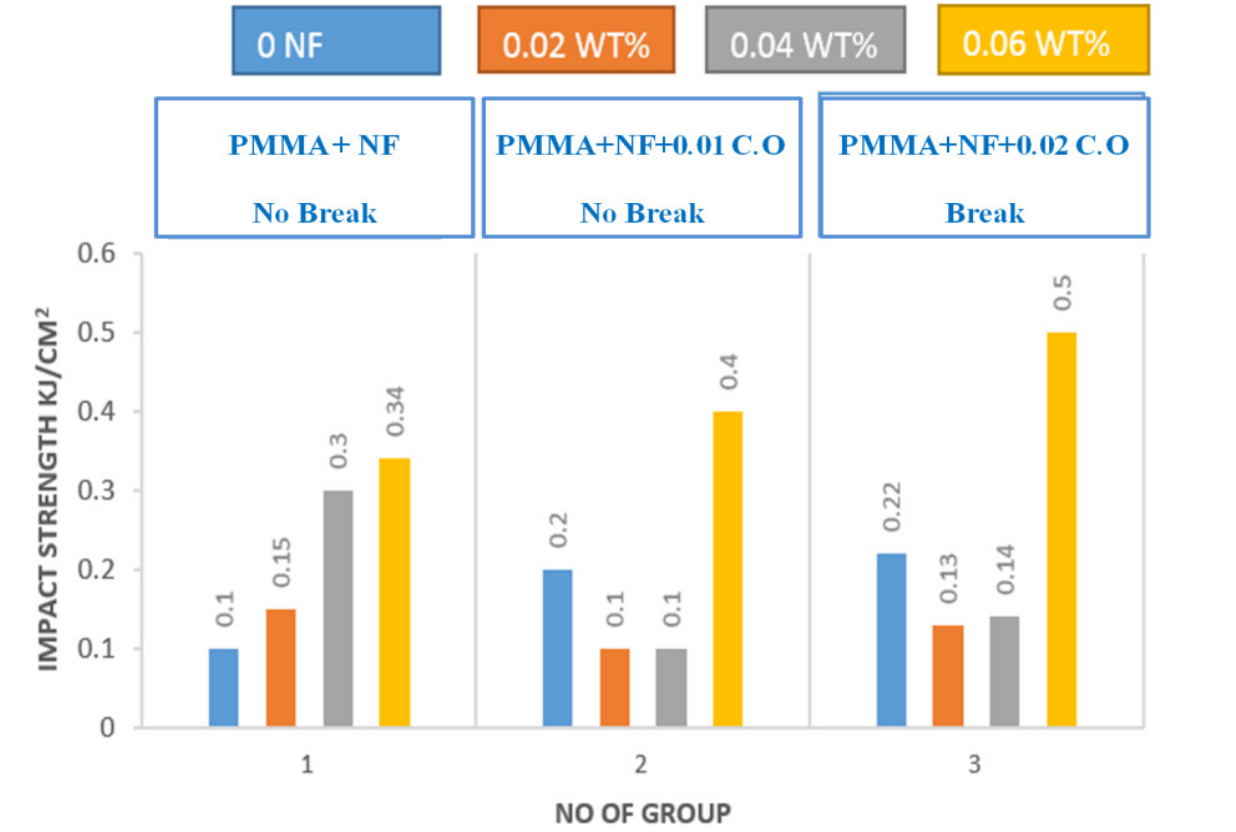

Figure 4. Impact strength of prepared composites samples.

Hardness Exam

The hardness is defined as a measure of the surface resistance to localized plastic deformation such as an indentation or scratch as a result of mechanical stress.[15,16] The hardness test was conducted for the prepared composite materials in order to study the surface characterizations. Figure 5(a, b) presents the prepared samples of hardness test and the Shore D hardness of nanocomposites as a function of PVA nanofibers content. The obtained results for the first group revealed that the hardness values decreased when the PVA nanofibers weight ratio increases, to be (71,1) for pure PMMA and (44,8) for the sample with three layers of

PVA nanofibers, which can be attributed to the PVA nanofibers leads to reduce the internal crosslinking and consequently their surface hardness. While the second group samples with the added cardamom oil (0,01 g) to the prepared composites samples shows slightly increasing in the hardness value with the PVA nanofibers weight ratio increases, the hardness value rises from (71,1) for pure PMMA to (72) for the sample enhanced with three layers of PVA nanofibers and cardamom oil, as shown in figure 5(b) due to the fact that a small amount (0,01 g) of the added cardamom oil promotes the crosslinking within the samples, and thus reinforcing the surface hardness.[17,18,19]

The hardness values for the third group samples with the higher cardamom oil (0,02 g) demonstrated that the hardness values significantly increased as a function of the PVA nanofibers weight ratio, the hardness value jumped from (71,1) for pure PMMA to (83) for the sample enhanced with three layers of PVA nanofibers and cardamom oil, as shown in figure 5(b), attributed to the added cardamom oil in a abundant amount led to higher crosslinking ratio within the sample, and then increasing the hardness (brittleness), which leads to rapid fracture of the samples. These results well agreed with the impact strength results as presented in figure 4.[20,21]

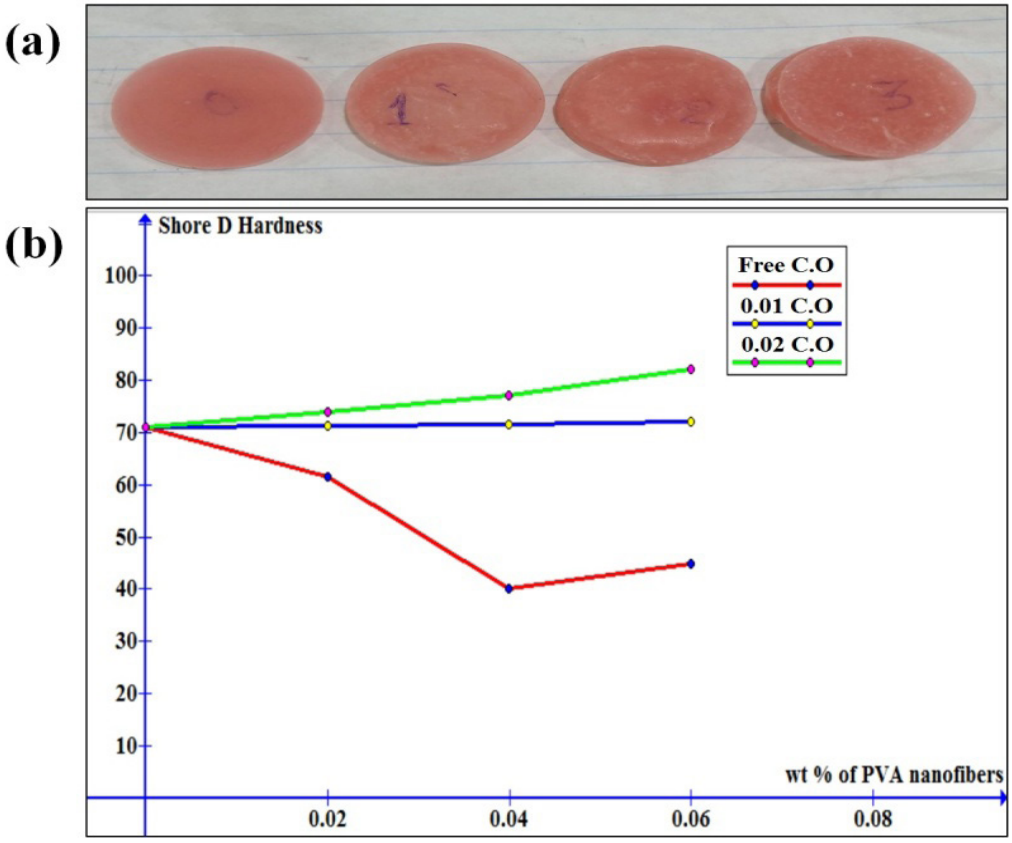

Figure 5. (a) the prepared impact samples and (b) the hardness of prepared composites samples as a function of weight ratio

Compression strength Exam

The compressive strength is defined as the capacity of the structure or material to resist the loads that tends to reduce the material size, in contrast to the tensile strength which resists the loads that tend to elongate.[22,23] Figure 6 presents the compression test results of prepared composites involving (compression strength, elastic modulus under compression, yield point, maximum elongation percentage, and toughness that refers to the area under the stress strain curve of compression) respectively of pure PMMA and PMMA enhanced with different weight ratio (1, 2 and 3 wt %) of PVA nanofibers.

The compression test results demonstrated that the composite sample consists of PMMA enhanced with (0,02 wt. %) of PVA nanofibers recorded the better compression results with the highest values of compression strength of (375 MPa), elongation percent (600%), yield strength (450 MPa), toughness (195 kJ/m^2), and mid elastic modulus value of (48 MPa), these results attributed to the presence of the PVA nanofibers at the weight ratio (0,02 wt. %) leads to ductile fracture and plastic deformation of the sample. on the other hand, the other samples that enhanced with (0, 0,01 and 0,03**)** wt of PVA nanofibers have the lowest mechanical properties under compression stress due to the brittle fracture was happened.[24,25,26]

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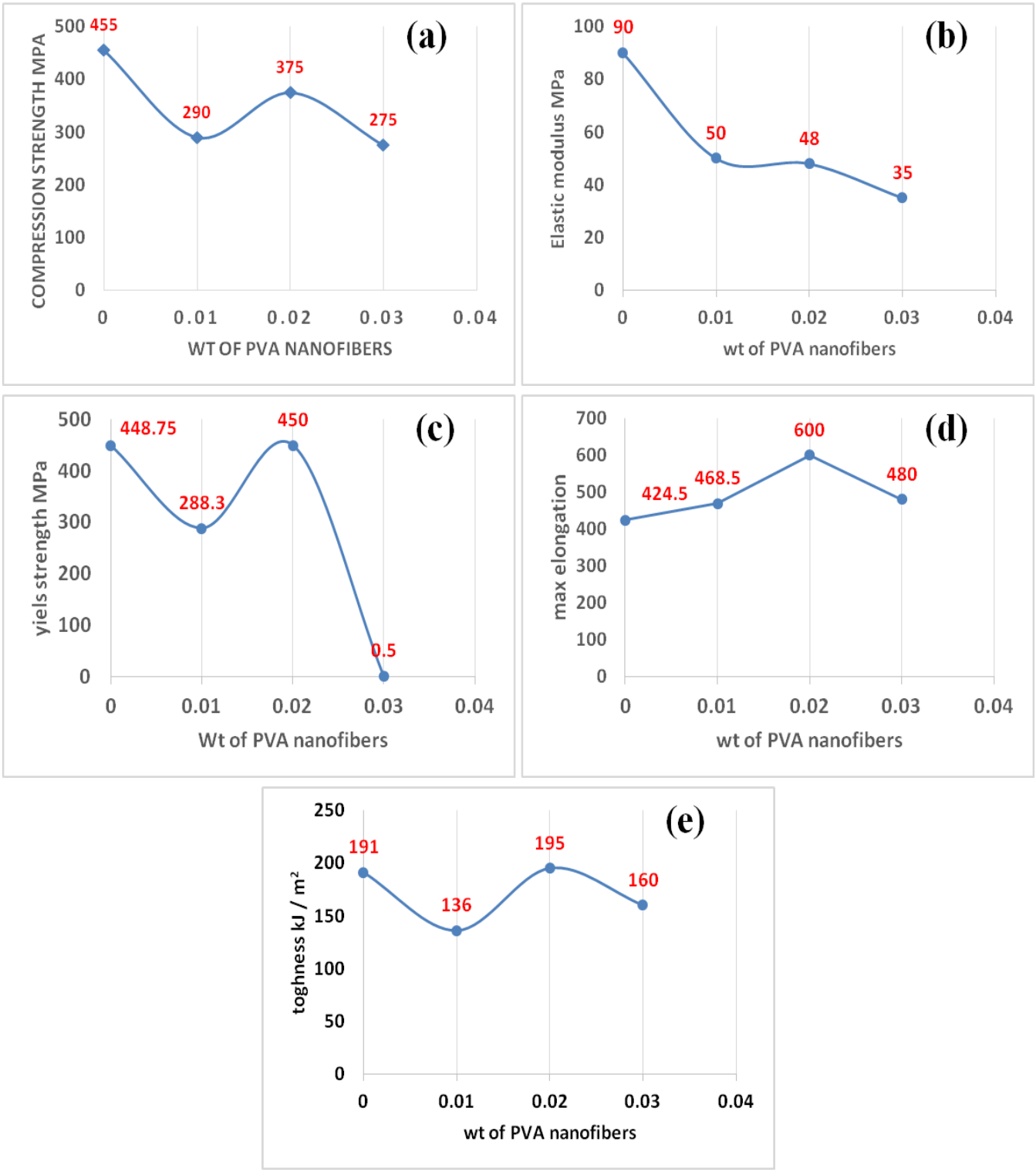

Figure 6. Compression test results (a) compression strength (b) elastic modulus (c) yield point (d) maximum elongation (e) toughness of prepared composites

Thermal Characterizations

Differential Scanning Calorimetry (DSC)

The DSC exam is a technique used to measure the temperature and heat flow associated with phase transition temperatures of materials.[27,28] The DSC test of prepared samples was conducted in order to understanding the thermal behavior of the samples. Figure 7 shown the DSC curves of pure PMMA and PMMA enhanced with (0,02) PVA nanofibers and the results of Tg and T decomposition were listed in table 2.

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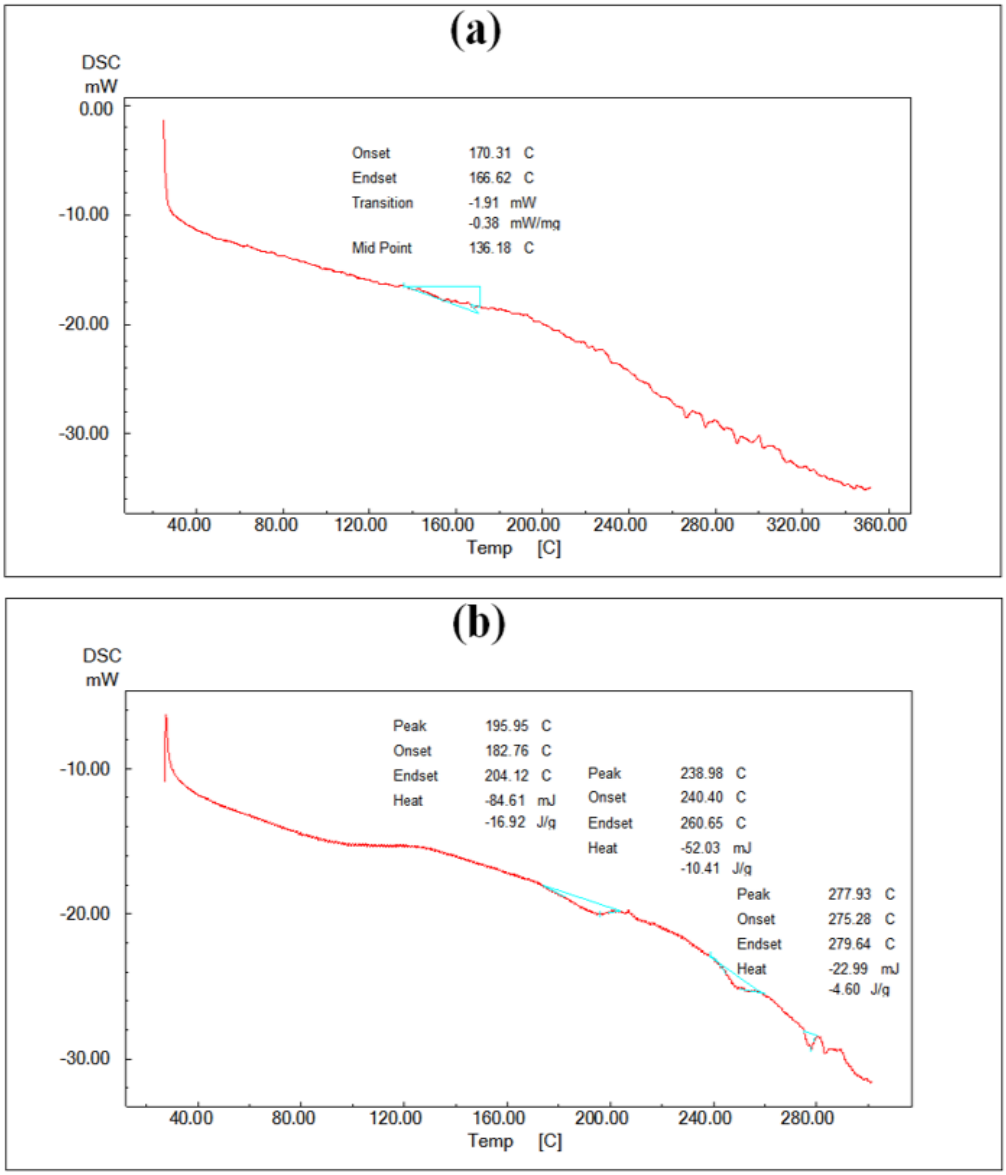

Figure 7. (a) DSC curve of pure PMMA and (b) DSC curve of PMMA + (0,02) PVA nanofibers

From figure 7(a) and table 2, observed that the Tg of pure PMMA was about (136 °C) and this temperature increased to (195 °C) after the adding of PVA nanofibers due to the PVA nanofibers leads to restricting the polymer chains movement and increase the stiffness of PMMA polymer chains. On the other hands, there are another Tg (238 °C) was appeared after the adding of PVA nanofibers as shown in figure 7(b) which is corresponding to present of PVA nanofibers. In addition that the decomposition point of nanocomposites is about (277,77 °C), as shown in figure 7(b).[29,30,31]

Table 2. DSC results of pure PMMA and PMMA enhanced with (0,02) PVA nanofibers

Sample	Contents	T_{g1} ° C	T_{g2} ° C	T_{dec} ° C
NC 0	PMMA	136	-	-
NC B	PMMA+ (0,02) PVA NF	195	238	277,77

CONCLUSION

The results of the current study lead us to draw the conclusion that treatment of PMMA with layers of nanofibers made of polyvinyl alcohol improves its mechanical properties, including impact strength. The presence of the fibers causes the fracture to change from brittle to ductile, protecting the samples from breakage because

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the energy required to cause a fracture is greater than the device's maximum value. Additionally, we found that a very small amount of cardamom oil increased the impact's durability and increased the energy needed to cause a fracture without the sample failing. The samples become weaker and brittle as the cardamom oil content rises, which makes them easier to break. Contrarily, compressive strength rises as the weight of fibers increases, and we see that the sample becomes more resilient to compressive stresses because it undergoes plastic deformation, which also enhances its strength.

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FINANCING

None.

CONFLICT OF INTEREST

None.

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