Research Article

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Mechanical properties of polymeric biomaterials: Modified ePTFE using gamma irradiation

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Abstract: Evaluating the mechanical properties of expanded polytetrafluoroethylene (ePTFE) is essential to measure its resistance to permanent deformation from an applied force. These mechanical ePTFE properties must be comparable to the properties of real tissue. Various hydrophilic comonomers 2-hydroxyethyl methacrylate (HEMA), *N*-isopropylacrylamide (NIPAAm), and *N*-vinylcaprolactam were used individually for copolymerization with acrylic acid (AA) to be grafted onto ePTFE using the gamma irradiation-induced grafting method. After surface modification, the hydrophobic and mechanical properties of ePTFE were altered. The water uptake and contact angle measurement showed that the modified ePTFE was less hydrophobic (~500%, $\theta < 90^\circ$) than the unmodified ePTFE (0%, $\theta = 140^\circ$). Moreover, the mechanical properties of ePTFE changed after the modification process due to the polymer grafted onto the ePTFE surface. The data from mechanical tests, such as Young's modulus (74–121 MPa), ultimate tensile strength (5–9 MPa), and elongation at break (56–121%), obtained for the sample AA-co-HEMA and AA-co-NIPAAm remain within the ranges and are considered desirable for use as a biomaterial. The mechanical strength correlates well with the percentage of the grafting yield after the modification process and is dependent on the parameters used, such as irradiation dose and type of comonomer.

Keywords: grafting, gamma, mechanical test, ePTFE

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Graphical abstract

1 Introduction

Polymeric biomaterials have a wide variety of applications for implantation as they are resilient to changes in the body environment after implantation as well as being easily fabricated in various desired properties for specific medical applications [1,2]. Nevertheless, producing polymeric biomaterials that can mimic actual tissue in soft tissue replacement is still a great challenge because no known surgical implant materials ever been shown to be completely free of adverse reactions in the human body [3].

Polymeric biomaterials that are commonly used are polyamides, polyethylene, polyacrylates, and fluoropolymers. One of the synthetic fluoropolymers that are considered to have “gold” properties is expanded polytetrafluoroethylene (ePTFE). This expanded version of PTFE has a multiple micropore structure that allows tissue at the soft tissue replacement site to grow inside the pores, thus promoting a faster healing process and proper integration with the tissue [4]. ePTFE was first used as a vascular graft in cardiovascular surgery and is currently used in the same application [5–7]. ePTFE is also used in other medical applications, such as guided bone generation [8], stent grafts, and surgical meshes [9].

Despite its wide usage in medical applications due to its inertness and nontoxic properties, ePTFE is extremely hydrophobic, which reduces its biocompatibility. As a characteristic of biomaterials, “biocompatibility” refers to materials showing good mechanical, chemical, pharmacological, and surface properties as well as beneficial interactions among cells, tissues, or muscles [1]. The standard specifications for implantable PTFE that may be used for implant products are provided by ASTM F754 [3].

Much research has been conducted on modifying ePTFE to enhance its biocompatibility. Functional groups are introduced to the ePTFE surfaces to reduce the extreme hydrophobicity without changing the bulk polymer's properties. Surface modification using radiation-induced grafting is a one-port synthesis grafting method compared to other methods such as chemical treatment, which requires multiple chemicals, an initiator, and heat to initiate the radical formation. Fluoropolymers are particularly susceptible to postradiation degradation in which radicals produced during the scission processes can be used to tailor the molecular weight in the fabricating polymer with desired characteristics [10,11]. Grafting through the irradiation method in the presence of monomers with desired functional groups is a simple method to improve ePTFE surface properties [12]. After the modification process, the extreme hydrophobicity of ePTFE can be reduced. This outcome can be seen in previous research using the same method with a different monomer, such as acrylic acid (AA) [13] or *N*-isopropylacrylamide (NIPAAM) and 2-hydroxyethyl methacrylate (HEMA) [4].

After the modification process, any polymeric materials will experience changes in physical or chemical properties. The polymeric biomaterials used for biomaterials applications should have the desired balance of biological and mechanical properties to perform as expected in a specific function. The specific optimization of properties, such as elasticity, yield stress, and ultimate strength, depends on the type and functionality of the specific implant. Although there will be some tissue reaction due to the introduction of a foreign substance, any resulting changes in mechanical, physical, and chemical properties within the localized environment should not lead to deleterious local changes and harmful systemic effects.

Mechanical performance is one of the most important biomaterial criteria to be analyzed in modified ePTFE. This property is crucial when handling mechanical stress on the healing of the soft tissue. Increasing mechanical stress potentially causes an increase in the cellular activity, which varies with the cells under study and the methodology used. As an example, soft tissue such as tendons

and ligaments displays tissue homeostasis dependent on mechanical stress and motion.

The mechanical properties of ePTFE investigated in studies by Catanese *et al.* (1999) highlight the linear density of manufactured ePTFE. These studies offer a general view on medical-grade ePTFE used in real implant applications [5]. Additionally, Zhu *et al.* reported the mechanical properties of modified ePTFE using a thermal treatment [14]. Studies on single monomers using the gamma irradiation-induced grafting method have also been reported [4,13]. Until now, a lack of research has been reported on the mechanical properties of ePTFE modified with a grafting method using dual monomers. Therefore, this article aims to promote an understanding of the mechanical properties of ePTFE after modification using a gamma irradiation-induced grafting method with dual monomers. The monomers used in this research are hydrophilic monomers, namely HEMA, NIPAAM, and *N*-vinylcaprolactam (NVCL), which were copolymerized with AA and grafted onto ePTFE. The physical and mechanical properties of the upgraded grafted comonomers were evaluated through percentage of water uptake, contact angle measurement, and a tensile test.

2 Materials and methods

2.1 Materials

AA, HEMA, NIPAAM, and NVCL were all obtained from Sigma-Aldrich. The comonomers used were a mixture of comonomer AA and HEMA, AA and NIPAAM, and AA and NVCL, and the samples were labeled as AH, AN, and AV, respectively. In this study, the ePTFE used is from the Pall Corporation and has a thickness of 0.3 mm, different from the 0.22 mm-thick ePTFE used in our previous study [15]. The total concentration of comonomer used is 1 M.

2.2 Grafting method

For tensile analysis, the ePTFE was cut into dumbbell-shaped pieces of 14 mm length and 2 mm width using a cutter according to ISO-37. The cut sample was first washed with hot methanol at 40°C to remove any contaminant inside the pores and then dried in a desiccator jar. The sample was immersed in a 1 M total concentration comonomer solution and degassed with nitrogen [15]. Sample AH was irradiated at an irradiation dose of

0.5 kGy, whereas 2 kGy for sample AN and AV with gamma cell (0.78 kGy/h; MDS Nordion). ePTFE samples were also irradiated at 2 kGy with no comonomer present, and the sample was labeled as ePTFE(2). After irradiation, the grafted samples were washed with hot methanol for 1 h to remove any ungrafted homopolymer attached to the ePTFE surface. Then, the samples were washed with deionized water and left immersed for 24 h and dried. The additional weight difference after the grafting procedure was measured as the grafting yield percentage.

2.3 Characterization

The amount of water uptake by the modified sample was measured by the increase of ePTFE-*g*-AAHEMA/AANIP-AAM/AANVCL sample weight, which was soaked in deionized water for 24 h. The percentage of water uptake was calculated using the following formula [15]:

$$\text{Water uptake (\%)} = \left[\frac{(W_s - W_d)}{W_p} \right] \times 100\%,$$

where W_s is the weight of the swollen sample (ePTFE/modified ePTFE immersed in deionized water), W_d is the weight of the dry sample (ePTFE/modified ePTFE), and W_p is the mass of p(AAHEMA/AANIPAAM/AANVCL).

The contact angle measurement was performed using the sessile drop method at 25°C (Surface Analyser LSA100, LAUDA Scientific). A total of 3 μ L deionized water was dropped by syringe on the samples, and the contact angle was measured. Five measurements were taken for each sample and the standard deviation was recorded.

Field-emission scanning electron microscopy (Merlin Compact, ZEISS) was performed on the samples under a high vacuum with a standard mode voltage at 3 kV.

To make the material conductive, the samples were coated with platinum for 5 min until an 8 nm-thick layer had formed before the analysis. A magnification of 5k $\times$ was used for the morphology analysis of the samples [16].

For the tensile test, samples were prepared in dry and wet conditions, and the test was carried out at room temperature. The measurement was conducted using an Instron testing machine (Universal Testing Machine 5564, Instron) at a constant crosshead speed of 10 mm/min under a 50 N load [17]. The wet sample was immersed in deionized water for 24 h before testing. The Young's modulus, E (MPa), elongation at break, ϵ (percentage of tensile strain), and ultimate tensile strength (UTS; maximum tensile strength in MPa) were evaluated, and five replicates were taken for all readings. Statistical analyses were performed using GraphPad PRISM software (GraphPad Software, Inc). Statistical analysis was conducted using the one-way analysis of variance method. The significant difference was defined as $p < 0.05$, $p < 0.01$, $p < 0.001$, and $p < 0.0001$ and noted as *, **, ***, and **** in the following results, respectively. If no asterisks are used, $p > 0.05$ shows no significant difference.

3 Results and discussion

3.1 Physical properties of modified ePTFE

The physical changes of the modified samples (AH, AN, and AV) can be observed in Figure 1 either in dry or in wet conditions. After 24 h immersed in deionized water, the modified sample AH, AN, and AV shows that the initial white color of the samples turns colorless. Initially, all sample length was 3.1 mm. After modification, all samples

Figure 1: Physical appearance and lengths of samples: (a) unmodified ePTFE, (b) ePTFE(2), (c) sample AH (dry), (d) sample AH (wet), (e) sample AN (dry), (f) sample AN (wet), (g) sample AV (dry), and (h) sample AV (wet).

Table 1: Length, grafting yield, and water uptake of unmodified and modified ePTFE

Sample	Length (± 1 mm)		Grafting yield (%)	Water uptake (%)
	Dry	Wet		
ePTFE	30.1	—	—	0
ePTFE(2)	3.1	—	—	0
AH	20.9	3.1	35 ± 3	540 ± 40
AN	20.9	3.1	36 ± 3	552 ± 61
AV	30	3.1	16 ± 3	378 ± 68

(dry condition) of AH, AN, and AV were reduced in dimension in the range of 1–2 mm after the grafting process (Table 1). An approximately 6% reduction was found for samples AH and AN, whereas sample AV was 3% compared to the original dimension. However, in a wet condition, the modified samples extended their lengths to the original dimensions before the modification process.

The grafting yield (measured by percent of weight increase) and the water uptake of the modified samples are shown in Table 1. Results show that the grafting yield and water uptake of the modified samples were seen to correlate because a higher grafting yield is accompanied by a higher water uptake. Samples AH and AN, with an almost similar grafting yield, were shown to have a higher water uptake compared to the AV sample, which had a smaller grafting yield.

A previous study by Hidzir *et al.* reported that exposing ePTFE to gamma irradiation at 10 kGy – although without the presence of a monomer – reduced the length of the irradiated ePTFE approximately 9% compared to its original condition due to a scission process that lowered the stress of the polymer chain [13]. In contrast, the observation in this

study on a sample ePTFE(2) irradiated without the presence of any monomers at 2 kGy does not show any changes in length. This finding indicates that the scission occurring at a lower irradiation dose, that is, 2 kGy, is probably not excessive, thus resulting in a similar length of the sample ePTFE (2) with unirradiated ePTFE. Grafting the hydrophilic comonomer onto the ePTFE surfaces allows the water to be absorbed on the surface, thus extending its length. The hydrophilic functional groups of the comonomer allow for the formation of H-bonds with water molecules.

The degree of grafting is affected by parameters such as irradiation doses and the type of comonomer used [18]. The parameters used in this study to obtain the optimum grafting include irradiation doses of 0.5 kGy for the sample AH and 2 kGy for samples AN and AV. Above that desired irradiation dose, a homopolymer formed, thus reducing the grafting yield (data not shown).

3.2 Wettability and contact angle

The contact angle analysis (Figure 2(a) and (b)) demonstrated that after the modification process, the hydrophobicity of the ePTFE was altered. In Figure 2(a), the contact angle of all samples was observed for 6 min to determine its wettability because each comonomer grafted onto ePTFE (AH, AN, and AV) had different wettabilities. At $t = 0$ min, sample AH showed the highest wettability ($\leq 90^\circ$) spontaneously ($88 \pm 12^\circ$) after the liquid interacted with the modified surface. This result is followed by both samples AN and AV, showing low wettability ($\geq 90^\circ$) with contact angle values of $127 \pm 4^\circ$ and $126 \pm 3^\circ$, respectively. Sample AN started to display its high wettability properties ($90 \pm 10^\circ$) at

Figure 2: (a) Graph of contact angle for unmodified and modified ePTFE and (b) image of contact angle for unmodified and modified ePTFE.

$t = 2$ min. Meanwhile, for sample AV, the contact angle lessens at a slower rate compared to samples AH and AV as the contact angle value was $109 \pm 13^\circ$ after 6 min.

The contact angle value correlates well with the grafting yield of the modified sample. Sample AH shows a lower contact angle value compared to sample AN although the grafting value was comparable and higher than the sample AV. The contact angle depends on the wettability of the polymeric materials and is time dependent. The wettability of HEMA is faster than NIPAAM, consequently resulting in a shorter measured time, and the contact angle of HEMA was smaller than AN. The contact angle for unmodified ePTFE and ePTFE(2) shows that without the presence of monomers, the hydrophobic properties of ePTFE are changeless. Therefore, no water uptake was observed for both samples: ePTFE and ePTFE (2). Likewise, the water uptake (%) of both AH and AN was comparable after 24 h immersed in deionized water, which resulted in $540 \pm 40\%$ and $552 \pm 6\%$, respectively. The value of the water uptake also agrees with the percentage of the grafting yield as the sample AV shows the lowest water uptake, with only $378 \pm 68\%$.

3.3 Field-emission scanning electron microscope morphology of modified ePTFE

Figure 3 shows FESEM images of ePTFE surface samples. The microstructure of the unmodified ePTFE is characterized

by many pores and island-like fractions called nodes that are interconnected by fibrils (Figure 3a). Initially, the fibrils were approximately $2\text{--}4\text{ }\mu\text{m}$, and the nodes were about $1\text{ }\mu\text{m}$. Each node connects from three to five fibrils. After modification, the node structure was seen to form a larger area than the fibrils, and the fibrils were seen to shorten for samples AH, AN, and AV (Figure 3), which was an indicator of successful grafting.

The node structures appear interconnected, forming groups of nodes due to the comonomer grafted onto the ePTFE, therefore creating extended structures that concurrently cover most fibrils [19,20]. More nodes were observed depending on the degree of grafting, thus explaining why fewer islands were observed for the AV sample compared to samples AH and AN.

The morphology of sample AH in dry conditions, which consists of more nodes (Figure 3b), tends to shrink more in length compared to samples AN and AV, which consist of fewer nodes (Figure 1 and Table 1).

3.4 Mechanical properties of ePTFE and modified ePTFE

The mechanical test analysis shown in Figure 4 was performed in dry and wet conditions, and three parameters were observed: Young's modulus (E), UTS, and elongation at break (ϵ). Statistical analysis was conducted to observe the differences between the unmodified and

Figure 3: Morphology of (a) unmodified ePTFE, (b) sample AH, (c) sample AN, and (d) sample AV.

modified samples. ePTFE is a flexible polymer, and it is commonly susceptible to radiation-induced damage (scission). These properties do affect the mechanical properties and decreased with an increase in irradiation dose [10]. The effect of grafting, where slight crosslinking may also occur during the irradiation process, increased ePTFE rigidity at the same time reducing ductility. Further increases in the irradiation dose in grafting usually will lead to a brittle sample, which is one of the drawbacks of the grafting process [21]. This effect does not just happen to polymer ePTFE. For example, a study conducted by Jiang and Hui examining grafting isoprene onto polyhydroxy butyrate also experienced a similar characteristic after the modification process [22]. Nevertheless, the selection of an optimum irradiation dose and concentration of monomers as well as the introduction of Mohr's salts to reduce the homopolymer formation in this study can prevent the sample becoming brittle.

The E value (Figure 4(a)) for the ePTFE(2) is 10.8 ± 1.7 MPa without a monomer shows no significant changes ($p = 0.8$) compared to the unmodified ePTFE (12.8 ± 3.1 MPa). The E of the modified samples grafted with comonomers AH, AN, and AV shows a higher value in dry conditions than wet conditions. Sample AH (130.8 ± 19.5 MPa) shows the highest E value followed by sample AN (74.0 ± 15.6 MPa) with significance values of $p < 0.00001$ and $p < 0.00001$ in

dry conditions compared to wet conditions, which represents the stiffness of the sample. The result shows a substantial increase in E value for dry samples (i.e., AH and AN) with 10- and 6-fold increases compared to unmodified ePTFE, respectively. In contrast, the E value for the sample AV (17.8 ± 2.6 MPa) in dry conditions was observed to slightly increase ($p = 0.4$) compared to unmodified ePTFE. For wet samples, the value of E for all modified samples AH, AN, and AV did not show any significant differences, with values of 14.4 ± 1.2 , 12.9 ± 2.2 , and 11.6 ± 1.2 MPa, respectively.

For the UTS value (Figure 4(b)), a significant difference was observed for all dry samples, namely AH ($p = 0.0004$), AN ($p = 0.0018$), AV ($p = 0.0004$), and ePTFE(2) ($p = 0.0002$), respectively, compared to the unmodified ePTFE. The UTS for ePTFE(2), AN, and AV shows a lower value with 4.2 ± 0.4 , 5.2 ± 1.4 , and 4.4 ± 0.4 MPa, respectively, compared to unmodified ePTFE (6.6 ± 0.7 MPa). In contrast, the UTS values for wet samples show no significant differences ($p > 0.05$) compared to the unmodified ePTFE. Sample AH shows the highest UTS value 8.8 ± 0.9 MPa.

The percentage of ϵ (Figure 4(c)) shows a significant decrease for all modified samples in both dry and wet conditions, as well as in sample ePTFE(2) ($p < 0.00001$) compared to the unmodified ePTFE ($\epsilon = 179.8 \pm 15.7\%$). Sample AN in dry conditions shows the least value of ϵ ,

Figure 4: Statistical analysis and mechanical properties ePTFE and modified ePTFE for (a) Young's modulus (E), (b) UTS, and (c) elongation at break (ϵ).

Table 2: Comparison of modified ePTFE in this study with previous study and ePTFE available in the market

Study	Modification method/application	Brand	<i>E</i> (MPa)	UTS (MPa)	ϵ (%)	Ref.
Modified ePTFE in this study compared with other studies						
This study	Radiation	Pall Corporation	12–131	4–9	56–121	—
Yan et al. (2021)	Chemical	Daikin	—	2–3	~40–150	[8]
Zhu et al. (2015)	Thermal	Gore Tex®	43–59	—	~60–110	[14]
ePTFE biomaterials available in the market						
Park et al. (2020)	Vascular graft	Advanta™ VXT	—	~17	—	[23]
Zhu et al. (2015)	Pericardial membrane	Gore Tex®	34	—	~110	[14]
Mun et al. (2012)	Vascular graft	Gore Tex®	32	14	28	[24]

followed by sample AH and ePTFE(2) with a value of $56.2 \pm 28.1\%$, $70.2 \pm 5.1\%$, and $103.6 \pm 16.5\%$, respectively. The elongation for modified samples AH and AN in wet conditions show a higher ϵ value compared to dry conditions with ϵ of $121.6 \pm 16.0\%$ and $86.4 \pm 7.2\%$, respectively. Finally, the ϵ values for sample AV in dry or wet conditions were almost similar, at 97.2 ± 8.9 and $98.4 \pm 10.0\%$, respectively.

The *E* values in this study show analogous patterns to a study conducted by Yan et al., where the value of ePTFE modified with silk fibroin and salicin also showed an increasing value, however, insignificant compared to the unmodified sample tested in dry condition [8].

The decrease in ϵ value for sample ePTFE(2) shows that the chain scission of the polymer due to gamma irradiation resulted in exposure damage in the trunk polymer [11]. This result (sample ePTFE(2)) was found to have a similar pattern to that reported by Hidzir et al., where the ePTFE irradiated at a higher dose (10 kGy) was used [13]. The tensile test performed on the ePTFE(2) sample shows that the irradiation process does not affect physical but mechanical properties, such as UTS, and the ϵ value shows a significant difference. The ϵ value for modified ePTFE (samples AH and AN) in the wet condition was higher than that of samples in the dry condition. Conversely, the ϵ value for AV in both dry and wet conditions showed no significant difference and correlated well with the lower grafting yield of AV obtained in this study ($16 \pm 3\%$). Previous research by Zhu et al. for ePTFE modification using thermal treatment also shows a decrease in value [14].

A comparison between the mechanical properties of the modified ePTFE with previous research and application of ePTFE as biomaterials is summarized in Table 2. The mechanical properties obtained in the current study are within acceptable ranges for *E*, UTS, and ϵ compared to the previous studies and are comparable with ePTFE biomaterials available in the market. Therefore, these results indicate the modified ePTFE in current study remains ideal for biomechanical characteristics.

The grafting yield is an indicator of the measured amount of comonomer grafted onto ePTFE, and the hydrophilicity usually correlates proportional to the grafting yield value. Less grafting yields for the AV sample resulted in the less-functional group attaching to the hydrophobic ePTFE. Therefore, similar properties for wet and dry sample conditions were observed for this sample. In contrast, the higher grafting yields obtained for samples AH and AN ($35 \pm 3\%$ and $36 \pm 3\%$) resulted in more nodes in the samples, thus affecting their elasticity (with more rigidity observed) compared to the unmodified ePTFE.

4 Conclusion

In sum, the mechanical behavior of ePTFE after modification with various comonomer by gamma radiation showed changes in the tensile performance (e.g., young's modulus, UTS, and elongation at break). The morphological changes after modification of ePTFE as well as the percentage of the grafting yield play a role in the tensile performance obtained. The mechanical data also proved that the irradiation dose can affect the mechanical performance of the polymeric material without the presence of monomers. The data from mechanical tests in this study, however, remain within acceptable ranges. Thus, ePTFE is considered desirable for use as a biomaterial. More studies are needed for further understanding of the mechanical properties of biomaterials.

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